Structural Characterization Techniques of Materials

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Abstract. After twentieth century there was a lot of study 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 are number of techniques to characterize samples such as x-ray diffraction, atomic absorption, thermal analysis, electron microscopy .This article presents scientific processes to characterize materials using modern technologies like such as: (I) thermogravimetry (TG/DTG), (ii) differential scanning calorimetry (DSC), (iii) differential thermal analysis (DTA), (iv) termomechanical analysis (TMA), (v)Non Destructive Techniques(NDT) and So on.

INTRODUCTION

The evolution of the seventeenth century scientific and technological evolution of industry in the late nineteenth century not reflected changes in understanding the structure of materials. Experts must be able to analyze and distinguish all materials or combinations of materials in use today—whether they may be metals, ceramics, polymers, semiconductors, or composites. To understand a material's structure, how that structure determines its properties, and how that material will subsequently work in technological applications, researchers apply basic principles of chemistry, physics, and biology to address its scientific fundamentals, as well as how it is processed and engineered for use. The characterization of materials is important for understanding their properties and applications. There are some techniques like X-ray diffraction allowed the determination of the crystalline structure of various materials and other indirect techniques such as thermal analysis. The techniques available for characterization of materials can be categorized in two broad classes, destructive & Non-Destructive Techniques. The destructive techniques cause certain variation in physical shape of and material properties, in some cases it damages the materials. Thus there will be degradation of the strength of the material is being evaluated. In Non-Destructive evaluation technique there is no physical damage while characterization of the material. In this paper we are discussing some characterization techniques: (i) Thermogravimetry (TG/DTG) (ii) Differential scanning calorimetry (DSC) (iii) Differential thermal analysis (DTA) (iv) Mechanical dynamical analysis (DMA) (iv) Termomechanical analysis (TMA) (v) X-ray fluorescence (FRX) (vi)Non Destructive Techniques(NDT) and So on.

VARIOUS TECHNIQUES OF CHARECTERIZATION

Thermal Analysis

Thermal analysis consists of group of techniques in which property of the sample is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed. There are variety of techniques available to meet the needs of materials characterization and analysis. The increasing use of thermal analysis both in academic and industrial applications has promoted in different areas: organic, inorganic,

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petrochemical, pharmaceutical, natural products, construction materials, coatings, catalysis, glass, ceramic, food, grease, surfactants, and composite polymers.

a) Thermogravimetry (TGA/DTG)

Thermogravimetry (TGA) 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 thermals 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].

(TGA) 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 looses 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 has been circumvented with new thermobalances [2].

b) Differential Scanning Calorimetry (DSC)

The differential scanning calorimeter (DSC) is a fundamental tool in thermal analysis. It can be used in many industries – from pharmaceuticals and polymers, to nanomaterials and food products. The information these instruments generate is used to understand amorphous and crystalline behavior, polymorph and eutectic transitions, curing and degree of cure, and many other material properties used to design, manufacture, and test products [3]. Differential Scanning Calorimetry (DSC) is a thermal analysis technique that looks at how a material's heat capacity (Cp) is changed by temperature. A sample of known mass is heated or cooled and the changes in its heat capacity are tracked as changes in the heat flow. This allows the detection of transitions such as melts, glass transitions, phase changes, and curing. Because of this flexibility, since most materials exhibit some sort of transitions, DSC is used in many industries, including pharmaceuticals, polymers, food, paper, printing, manufacturing, agriculture, semiconductors, and electronics [3].

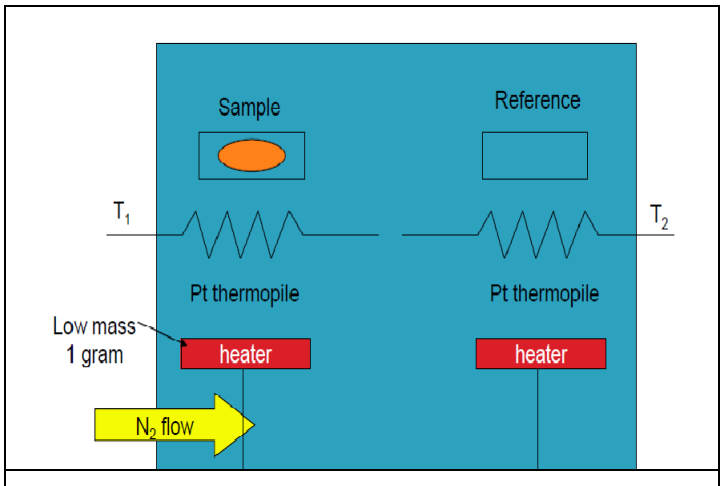

FIGURE 1. DSC Instrument Analogy

The biggest advantage of DSC is the speed with which it can be used to see transitions in materials. If you work with polymeric materials of any type, the glass transition is important to understanding material. In liquid crystals, metals, pharmaceuticals, and pure organics, phase changes or polymorphs and study the degree of purity in materials. In distilling materials, knowledge of a material's heat capacity and heat content change (called enthalpy) can be used to estimate how efficiently your process is operating. For these reasons, DSC is the most common thermal analysis technique and is found in many analytical, process control, quality assurance, and R&D laboratories. For better understanding we will consider an example of polymerization process of materials on the basis of glass transition Tg.

The glass transition (Tg) has been called the "melting of amorphous material" and as unscientific as that is, it's an adequate description. Amorphous material such as glass has random structure in solid state. This gives it the transparency that glass has, among other properties. As it warm up, its heat capacity increases. At some point you have enough energy in the material that it can be mobile. This requires a fair amount of energy compared to the baseline increase, although much less energy than the melting point does. This energy normally appears as a step change in the instrument baseline – pointing up in heat flow instruments and down in heat flux. [3]

In non-crystalline and semi-crystalline polymer of any type – synthetic high polymers such as polypropylene and polystyrene, natural polymers like rubber, or biological polymers such as proteins – the glass transition is the best indicator of material properties. As the glass transition changes due to either different degrees of polymerization or modification by additives, the physical properties of the material change. The relationship of Tg to the degree of polymerization shown in Figure 2 changes with these alterations.

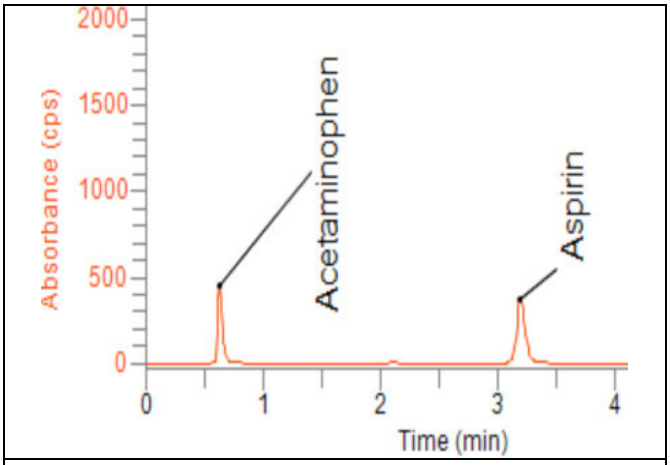

FIGURE 2. The relationship of Tg with degree of polymerization showing the Tg critical where the material develops polymeric properties

DSC can detect any change that alters the heat flow in and out of a sample. This includes more than just glass transitions and melting. We can see solid state transitions such as eutectic points, melting and conversions of different crystalline phases like polymorphic forms, dissolution and precipitation from solutions, crystallization and recrystallizations, curing exotherms, degradation, loss of solvents, and chemical reactions.

C) Differential Thermal Analysis (DTA)

In most cases, heating a system (an element, a compound, a mixture) causes physical and chemical changes. In a Differential Thermal Analysis experiment (DTA experiment), the temperature difference between the sample under investigation and an inert reference material is measured as a function of temperature. Both samples are treated with the same temperature program and the same heating and cooling rates[4].

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 owning to heat absorption or emission by the sample [1].

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

d) Termomechanical Analysis (TMA)

Whenever a sample of material is to be studied, one of the easiest tests to perform is to heat it. The observation of the behavior of the sample and the quantitative measurement of the changes in temperature can yield a lot of useful information. The study of the relationship between a sample property and its temperature as the sample is heated or cooled in a controlled manner is commonly referred to Thermal analysis (TA). Thermomechanical Analysis (TMA) is one of the method, The method is defined by the International Confederation for Thermal Analysis and Calorimetry (ICTAC) [6] as a technique where the deformation of the sample is measured under constant load. This is realized by measuring dimensional changes of solids, liquids or pasty materials as a function of temperature and time under defined mechanical forces. The result of TMA measurements is a curve showing the change of the sample length versus temperature and time. It is powerful tool used in the analytical laboratories. It can hereby provide valuable insight into the composition, phase changes, structure, sintering steps or softening which can occur in addition to thermal expansion, production conditions or application possibilities for various materials. Typical domains include plastics and elastomers, paints and dyes, composite materials, adhesives, films and fibers, ceramics, glass, metals, and composite materials. The measurements can be carried out by the number of different probe configurations. Depending on the applied load experiments may be done in compression, tension, shear, torsion, penetration or some bending mode as shown in Figure 3. The choice of the measurements mode depends on the studied properties, shape of the sample and/or its application.

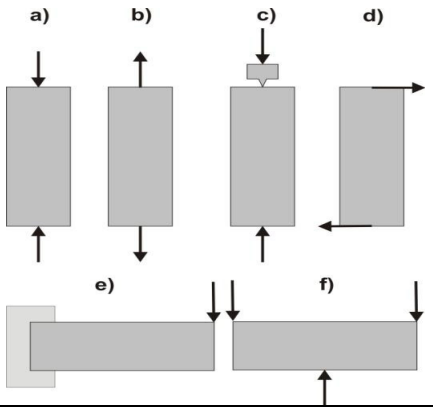

FIGURE. 3 Common mechanical deformation modes in TMA: a) compression, b) tension, c) penetration, d) shear, e) bending, f) three point bending.

A schematic diagram of the typical instrument is shown in Figure 4. The sample is placed in the chamber where the temperature is controlled by the thermocouple placed close to the sample. The measurements are performed in the protective atmosphere of inert gases like: nitrogen, helium or argon but also the other gases could be used i.e. air, carbon monoxide, hydrogen. Because of the relatively large mass of the sample the applied heating and cooling rates are usually slow. The rate of 5°C/ min is usually the maximum recommended value for good temperature equilibration across the specimen.

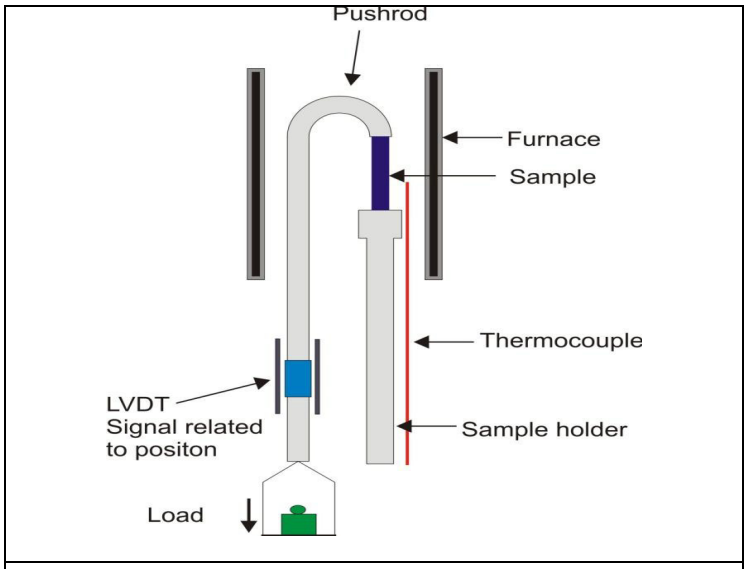

FIGURE 4. Schematic diagram of a Thermomechanical analyzer

TMA applications are in many ways the simplest of the thermal techniques. We are just measuring the change in the size or position of a sample. However, they are also incredibility important in supplying information need to design and process everything from chips to food products to engines. Because of the sensitivity of modern TMA, it is often used to measure T_{gs} that are difficult to obtain by DSC, for example those of highly cross-linked thermo sets.

Non Destructive Techniques

Non-destructive techniques have been used almost exclusively for detection of macroscopic defects in existing structures [9]. It is practical and cost effective to expand the role of non-destructive evaluation and can be included in all phases of material production. Ultrasonic NDT is the choice for inspecting the interior of material because ultrasound can penetrates deeply and can provide the characteristics in depth . For the purpose of detecting defects inside metals/materials, it is most practical alternative to heavy doses of x-rays or gamma ray.

An understanding of the behavior of the material under normal loading condition is vital, as this provides information that can be used to prevent failure. The material behavior can be used to identify samples with defects or inhomogenieties. Therefore ultrasonic inspection is used for quality control and material inspection in all major industries [11].

a) X-Ray Fluorescence (XRF)

X-ray fluorescence is a method to understand the chemical and elemental constituency of the artifacts. X-rays have been used for commercial elemental analysis since the 1950s, X-ray spectroscopy is much older Charles G. Barkla found a connection between X-rays radiating from a sample and the atomic weight of the sample. Originally, X-ray spectroscopy used electrons as an excitation source, but the requirements of high vacuum, electrically conducting specimens, and the problem of sample volatility posed major roadblocks. To overcome these problems, an X-ray source with a metal target was used to induce the fluorescent emission of secondary X-rays in the sample. Excitation of the sample by this method introduced some problems by lowering the efficiency of photon excitation and requiring instrumentation with complex detection components. Despite these disadvantages, the fluorescent emission of X-rays would provide the most widely used tool for the analyst using commercial instruments. Many samples can be examined with little or no pre-treatment. Many of the alternative techniques require dissolution procedures that are both time-consuming and costly in terms of the acids or other reagents required, vigorous cleaning is not necessary [7,8]. This is mainly due to the penetration of X-rays in the mid-Z X-ray region beyond the surface. This technique is a very fast and easy to use it enables chemical compositions to be determined in seconds.

Visual Techniques

Visual techniques is the most common and simple Non-Destructive Testing method. The test involves a close visual inspection of material surface. The material may be illuminated or the operator may use a magnifying glass, mirror or other optical aid in order to gain accurate results. The main drawback of these techniques is that the internal defects in the material are undetected.

a) Magnetic Particle Inspection

Magnetic particle inspection is a Non-Destructive Testing method used for defect detection. This method is fast & relatively easy to apply and part surface preparation is not critical. This method uses magnetic fields and small magnetic particles, such as iron fillings to detect flaws in components. The only requirement from inspect ability point of view is that the component to be inspected should be ferromagnetic materials. Magnetic particle inspection is particularly sensitive to surface or near- surface defects. The disadvantage of this technique is that it can be applied to ferromagnetic materials & will not detect deep internal flaws.

b) Electromagnetic or Eddy Current Testing

This method is an electrical measuring method, whereby eddy currents are induced in surface of material. Variation in material such as defects and electrical conductivity will affect phase and amplitude of eddy currents. This information can be displayed as meter readings or as oscilloscope display. This method is used to detect cracks, material thickness, coating thickness & conductivity measurement for material identification. The disadvantages of this method is that only conductive material can be inspected and skill & training is required than other techniques.

c) Liquid Penetrating Testing

Liquid penetrating methods are simple and are commonly used for the detection of surface breaking discontinuities, especially cracks. Color contrast or fluorescent penetrating liquid dye applied to surface, penetrates crack by capillary action and remains trapped when surface excess liquid removed. A white powder, called developer, is then sprayed or dusted over the part. The developer lifts the penetrate out of the defect. Then by visual inspection under white or ultraviolet light, the visible or florescent dye indications are located, thereby defining the defect. This method has high sensitivity to small surface discontinuities. Large areas & large volumes of materials can be inspected rapidly and at low cost by this method. Disadvantage of this method is that only surface breaking defects can be detected & only materials with a relative nonporous surface can be inspected.

d) Ultrasound techniques

It is the Non-Destructive Techniques (NDT) which is more accurate in assessing the characterization of materials than the conventional practice of visual inspection. The velocity of ultrasonic waves traveling through a material is a simple function of the material's modulus and density and many more other physical properties and thus ultrasonic methods are uniquely suited to materials characterization studies. In addition, ultrasonic waves are strongly reflected at boundaries where materials properties change, and thus are often used for thickness measurement and crack detection. The main principle of ultrasonic techniques is that high frequency vibrations in the form of a longitudinal or angled beam are included in the materials by a piezo-electric crystal, internal defects will reflect beam back to crystal which converts energy to an oscilloscope. Beam can be transmitted and received by the same crystal or through transmitting methods can be used where the beam is transmitted and received by separate crystals.
The two major methods of ultrasonic inspection are the transmission method and pulse-echo method. The primary difference between these two methods is that the transmission methods involves only the measurement of signal attenuation , while the pulse-echo method can be used to measure both transit time & signal attenuation.

i) Pulse –echo Method

The pulse-echo method, which is the most widely used ultrasonic method, involves the detection of echoes produced when an ultrasonic pulse is reflected from a discontinuity. When ultrasonic pulse interacts with defect, there will be almost total reflection. This enables the relative amplitudes and time of flight of ultrasonic pulses to be use as a measure of integrity of the material under test. The pulse echo mode, as shown in figure 5, requires accesses to only one side of a test piece and a single transducers act as transmitter and receiver.

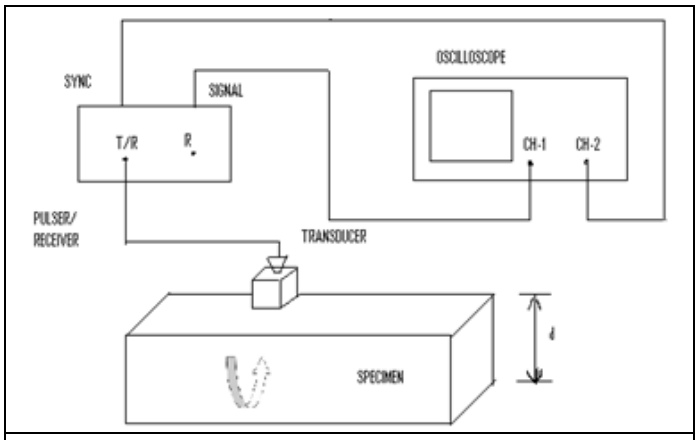

FIGURE 5: Schematic diagram of Ultrasonic Pulse-echo method

Because of large impedance mismatch between air and solid materials it is difficult to propagate waves from a transducer through air into the structure. Therefore coupling media between the two is generally employed. The acoustic impedance for a longitudinal wave (z) is defined as the product of material density ρ and longitudinal wave velocity v is given by

$$z = \rho v$$

The frequency and the wave length of ultrasound are related by $\lambda = c/f$ where λ is the wave length. Hence if the wave velocity is constant the wavelength will become smaller with increasing frequency. The detectable defect size is related to the wavelength, the higher the frequency, the smaller the detectable defect. However, in real materials the wave amplitude decrease or attenuate during propagation. Attenuation is a function of frequency: therefore as the frequency of the wave is increased its penetrating power will be reduced. The sensitivity of the ultrasonic method is dependent on the properties of the transducers used.

ii) Transmission Method

Transmission ultrasonic testing is done with direct beam or reflected beam, flaws are detected by comparing the intensity of ultrasound transmitted through a reference standard made of same material. Transmission testing requires two search unit one to receive them. Immersion techniques are most effective because they provide efficient and relatively uniform coupling between the search units and the test piece. This is more commonly used method measures the transit time as well as the intensity of ultrasound.

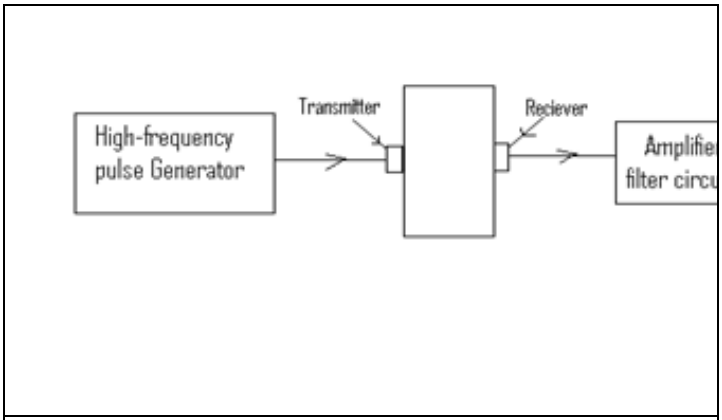

FIGURE 6. Through transmission method

Display of transmission test data can be either oscilloscope trace or meter recordings. Small differences in dimensions or sound transmission properties between two test pieces of same design can be results in large differences in the measured sound beam intensity because of a shift in the spatial distribution of standing waves. The main application of transmission methods is the inspection plate for cracks and laminations that have relatively large dimensions compared to the size of the search units.

CONCLUSION

The present review showed the most common analytics techniques applied in the materials characterization. It consists of several Thermal Analytical and Non-Destructive Techniques. Thermo Gravimetry (TG) examines the mass change of a sample as a function of temperature, DSC is a thermal analysis technique that looks at how a material's heat capacity (Cp) is changed by temperature as well as the temperature difference between the sample under investigation and an inert reference material is measured as a function of temperature. NDT that are used for the evaluation of material properties and structure. Moreover, it has been shown that Non-destructive characterization affords a most promising opportunity during production of materials, devices and structure out of several NDT test. Ultrasonic testing are most advantageous because of superior penetrating power, high sensitivity , greater accuracy , electronic operation, volumetric scanning ability, Non hazardous, portability & provides an output that can be processed digitally by a computer to characterize defects and to determine material properties. We believe that both these techniques are most significant development for characterization and analysis of material properties.

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