

Basics of Thermal and Mechanical Characterization Techniques

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Material technologies are crucial for the advancement of modern technologies. Characterization of mechanical and thermal properties of different materials are essential to comprehend intrinsic properties of the materials where advanced and innovative materials could be produced. Thin films and nanomaterials are important and were associated with nanotechnology and nanomaterials. Such materials can be applied to electronic devices, solar cells, sensors, solar harvesting devices, detector technologies, drug delivery applications, pharmaceuticals, avionic industries, medical applications, military applications, automotive industries, etc. Mechanical characterisation techniques include microhardness, fatigue, and fracture analysis, nanoindentation, etc. Besides thermal characterization techniques can also be used for the characterizations which are thermal gravimetric analysis, differential scanning calorimetry, etc. Such techniques can be used to characterize nanomaterials such as carbon nanotubes, graphene, metal nanoparticles thin films etc. In this report, we briefly discussed mechanical and thermal characterization methods used for materials applications such as thermal gravimetric analysis, differential scanning calorimetry, thermal characterization; microhardness, fatigue, and fracture analysis, nanoindentation.

Keywords: *Thermal gravimetric analysis; Differential scanning calorimetry; Thermal characterization; Microhardness; Fatigue, and fracture analysis; Nanoindentation*

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1. Introduction

Materials and material applications are essential for the advancement of modern technologies. Various applications need materials with different physical, chemical, and mechanical properties. Fundamental material characterization techniques are required to understand the intrinsic material properties [1]–[3]. The mechanical and thermal resistance characteristics of materials are crucial for particularly in heavy industries such as automotive industries, defence industries, military technologies, aerospace industries, construction technologies, metal technologies, ceramic technologies, and plastic industries [4]–[6]. Therefore, mechanical, and thermal characterization methods have significant importance [7], [8].

Thermal characterization methods encompass a range of techniques developed to understand the behaviour of materials under and against temperature. These methods determine properties such as the material expansion coefficient, thermal resistance, thermal conductivity, and heat capacity [4], [7]–[9]. They can also be used for the determination of transition characteristics and the assessment of the material's decomposition, investigation of the alteration in its chemical structure under specific temperatures. Understanding such behaviours enables researchers to develop materials with heat high resistance for the industry, optimize the thermal behaviour of materials, and increase the energy efficiency [4], [7]–[9]. Mechanical characterization techniques are employed to understand the elasticity, toughness, fracture behaviour, elastic modulus, deformation behaviour, fracture, and flexibility strengths of materials [4], [9]–[11]. Various test

and analysis methods can be utilized for this purpose. These techniques are used to enhance the strength and performance of materials under a force or a pressure. Such a case leads to the development of more efficient and resilient products in fields such as automotive, aerospace and space, military technologies, and weapon technologies[4], [7]–[11].

In conclusion, thermal and mechanical characterization methods play a critical role in understanding and optimizing the properties of materials in scientific research. The use of such methods allows materials to be employed in engineering and scientific fields where more reliable, durable, and efficient materials can be fabricated. Our report focuses on thermal gravimetric analysis, differential scanning calorimetry, and thermal characterization techniques. Additionally, it includes an examination of mechanical tests such as nanoindentation, microhardness, fatigue, and fracture analysis. It is anticipated that our report can provide basic information about mechanical and thermal tests.

2. Mechanic Tests

Mechanical tests are important characterization methods used to evaluate the mechanical properties of materials. Nanoindentation and microhardness testing are significant techniques among these tests.

2.1. Nano Indentation

The technique is used to evaluate the mechanical properties of a material at a microscopic scale where measurement was conducted on the surface of the material. In this technique, a small force (usually at the nanonewton level) is applied to the material surface, measuring the deformation on the surface to determine material hardness, elastic modulus, and viscosity. Typically, a very hard material (a probe made of hard materials such as diamond) is pressed onto the surface where mechanical properties can be measured. Pressing hard probe on the surface allows it to penetrate the surface. As the load is increased, it reaches a certain level, and then the load begins to decrease. When the load is decreased, a certain graph is obtained. The Young's modulus value of the material can be calculated by derivation of unloading plot. Material hardness is defined using the following equation:

$$H = \frac{P_{max}}{A_r} \quad eq.1$$

where H is hardness P_{max} maximum load, A_r is the residual indentation area. Nanoindentation is used particularly to determine the mechanical properties of structures at the nanometre scale, such as thin films, coatings, and nanomaterials. This method can be applied to both

composite materials and films composed of a single material. The technique can be used in implant technologies, aerospace applications, coatings, and protective surfaces where mechanical properties and hardness are crucial.

2.1. Micro-Toughness Test

The technique is used to assess the surface hardness of a material. This test determines the material hardness by measuring the deformation on the surface with a force applied to the material surface (usually at the micronewton level). Microhardness testing is employed to analyse hardness differences among various material types, assess the impact of material processes, and control the quality of coatings. This test can be conducted using Vickers or Knoop indenters.

Nanoindentation and microhardness testing are crucial tools for examining the surface properties of materials and understanding their mechanical behaviour. These tests are used in various applications such as material selection, design optimization, coating evaluation, and control of material processes.

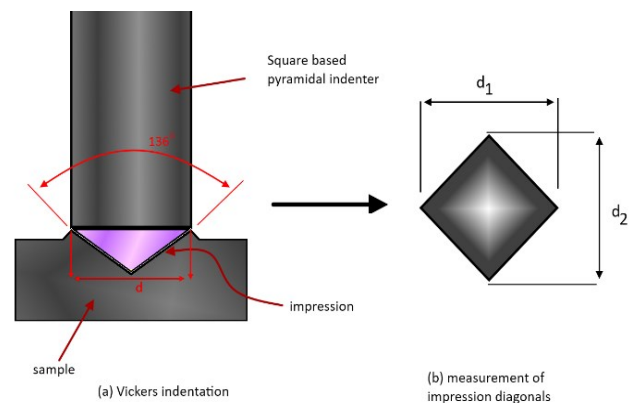


Figure 1: Schematics of Vickers indenter

3. Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) is an analytical method used to evaluate the thermal behaviour of a material by correlating its weight change with temperature or time. This technique is employed to determine the material's thermal stability, decomposition temperatures, moisture absorption capacity, and thermal degradation processes. This information can be understood from the changes in the material's weight. The basic principles and applications of Thermogravimetric Analysis (TGA) include:

Basic Principle: TGA is an experimental method that continuously monitors the weight change of a material. The

technique is based on the principle of heating the material within a controlled container and measuring its weight during this process. As the material is heated at a specific heating rate in an oven or heater, thermal changes occurring on the material lead to weight loss or weight gain. The material goes through various stages during heating. For example, during the initial heating stage, elements such as water vapor evaporate from nanoparticles or powdered molecules, causing a reduction in weight. Alternatively, if the material contains organic molecules, these molecules may combust above a certain temperature, resulting in their removal from the material and a change in weight. Throughout the entire process, the TGA device records the weight change by measuring the weight of the material sample with a sensitive balance. This allows for the quantification of the amount and number of molecules within the material.

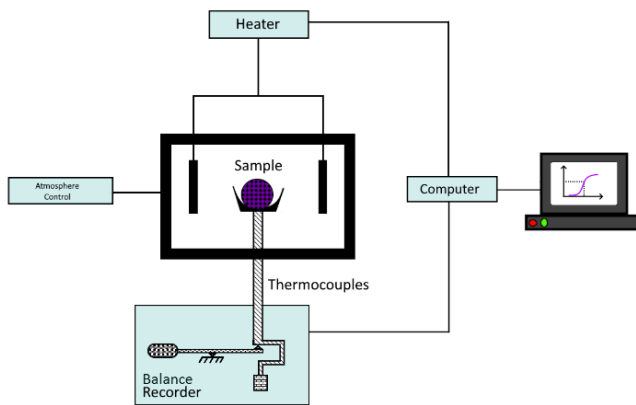


Figure 2: Schematics of thermogravimetric analysis (TGA) device.

Applications: TGA is used in various materials and applications. Some common application areas include:

1. Analysis of the thermal behaviour of polymers: TGA can be used to examine the thermal decomposition temperatures, thermal stability, and decomposition products of polymers.

2. Analysis of solid fuels: Combustion behaviour, ignition temperatures, and calorific values of solid fuels derived from coal, biomass, or waste can be determined using TGA.

3. Analysis of inorganic materials: Thermal decomposition processes and decomposition kinetics of inorganic materials such as ceramics, metal oxides, and salts can be evaluated with TGA.

4. Quality control of polymer coatings: TGA can be employed to control the quality of polymer coatings and determine coating thickness and structure.

Among the advantages of TGA are high sensitivity, a wide temperature range, automatic data recording, and high repeatability. These features support the use of TGA in various fields such as material characterization, material selection, and optimization of manufacturing processes.

4. Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) is an analysis method used to investigate the thermal behaviours of materials. This technique determines thermal events such as transitions, endothermic or exothermic reactions, melting, or crystallization by measuring the response of a material sample to changes in temperature. The basic operating principle and applications of DSC are as follows:

1. Basic Principle: DSC is a device with two thermal chambers, one containing a reference sample and the other containing the test sample. Both the reference and sample chambers undergo the same heating profile. The temperature difference between the material sample and the reference is measured to identify thermal transitions. Thermal events in the sample, occurring during various transitions or reactions, can be observed as heat absorbed or released.

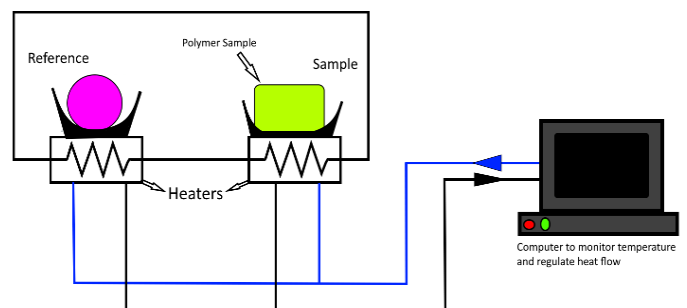


Figure 3: Schematics of differential scanning calorimetry (DSC) device.

2. Applications: DSC finds application in various materials and fields. Some common application areas include:

2.1. Analysis of the thermal behaviours of polymers: DSC can be used to examine the melting and crystallization temperatures of polymers, glass transition temperatures, polymerization reactions, and thermal stability.

2.2. Pharmaceutical industry: Factors such as stability of drug formulations, melting properties of drugs, polymorphic transformations, and reaction kinetics can be evaluated using DSC.

2.3. Combustion analysis: Burning characteristics such as combustion temperatures, energy release, and oxidation kinetics can be determined using DSC.

2.4. Analysis of chemical reactions: DSC allows the detection of endothermic or exothermic reactions, as well as the examination of reaction kinetics and enthalpy.

DSC has several advantages such as high sensitivity, a wide temperature range, automatic data recording, high repeatability, and ease of use, etc. These features support the use of DSC in various areas such as material characterization, product quality control, formulation optimization, and analysis of chemical reactions.

5. Fatigue and Fracture Analysis

Fatigue and fracture analysis is an experimental approach used to assess the durability, strength, and fracture properties of materials. These analyses address important issues such as understanding material performance, optimizing design, and ensuring structural integrity.

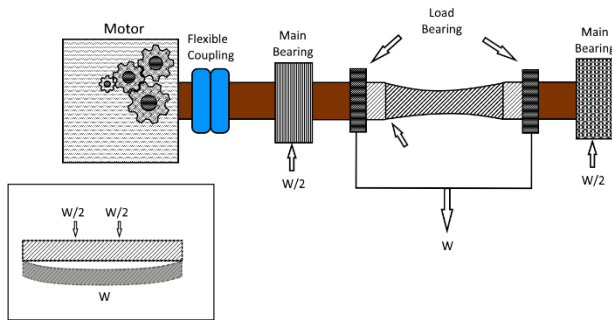


Figure 4: Schematics of fatigue testing machine

Some fundamental techniques used for fatigue and fracture analyses include:

Fatigue Tests: Fatigue tests are employed to evaluate the resistance of materials to repeated loading. In these tests, the material specimen undergoes repeated stress or deformation cycles under a specific load. The number and magnitude of these cycles are crucial in determining the

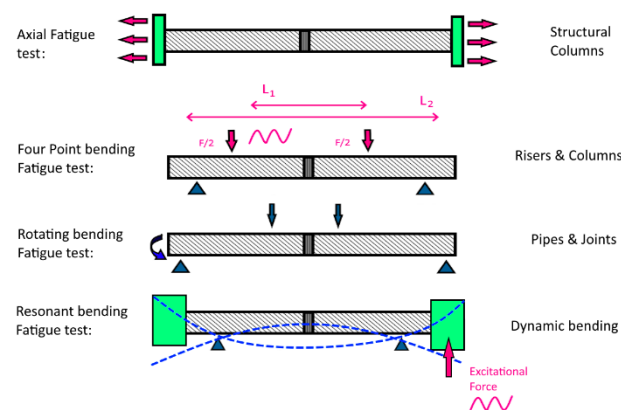


Figure 5: Schematics of illustrating fatigue tests

fatigue strength and life of the material. Fatigue tests are often conducted using various methods to assess parameters such as stress-strain curves, fatigue life, and stress fatigue.

Impact Tests: Impact tests measure the resistance of materials to sudden impact loads. In these tests, the specimen is subjected to impact force or deformation under impact energy. These tests are used to evaluate fracture toughness, crack initiation and propagation, energy absorption, and fracture mode of the material.

Fracture Surface Analysis: Fracture surface analysis is used to examine the fracture mode and mechanisms of materials. The surface of a fractured material specimen is examined under a microscope to obtain information on fracture mechanisms, fracture paths, crack formation, and propagation routes. This analysis is important for understanding the fracture resistance and fatigue properties of the material.

Fracture Mechanics Analysis: Fracture mechanics analysis is used to assess the fracture behaviour of materials subjected to cracks. Parameters such as crack size, stress intensity factor, K-plane integration, and linear elastic fracture mechanics are used in these analyses. These analyses are crucial for determining the durability, fracture resistance, and crack propagation rate of materials.

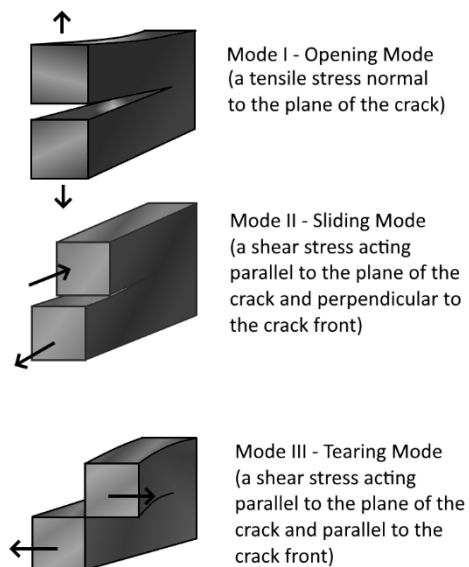


Figure 6: Schematics of illustrating the fracture Mechanics analysis

Fatigue and fracture analyses play a significant role in materials science and engineering applications. These analyses are used in various fields such as structural

component design, material selection, damage detection, and life cycle management. These techniques provide important information to enhance the durability and integrity of materials, detect flaws, and create safe and robust structures. Such tests can also be applied for assessing the mechanical characteristics of the crystals.

6. Conclusion

Mechanical and thermal characterization of the materials has a crucial role for thin films, nanomaterials, and material technologies. Proper characterization techniques enable researchers to develop and produce high quality and functional materials which can be used in different industries. In our report, we provide a brief information about thermal and mechanical characterization techniques which were commonly used for different applications.

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Mümin Mehmet KOÇ: Editing, English check
Burhan COŞKUN: Conceptualization, writing, supervising

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