Microstructural Characterization Methods, Significance & Analysis of Composite Materials

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Abstract— Microstructural analysis and metallography are the predominant terms used in materials research. The characterization and analysis of microstructures have become extremely important in materials science and engineering. Materials fabricated with specific sizes and structures are expected to find a variety of applications. The discovery of novel materials, material processes, new experimental and theoretical approaches for research have good opportunities for the development of innovative materials. This paper emphasizes the importance and methods of microstructural characterization, metallographic sample preparation and different instruments available.

Key words: Microstructural, Characterization, Analysis, Experimental, Metallographic

I. INTRODUCTION

Microstructure is defined as the structure of a prepared surface or a thin foil of material investigated by employing a microscope of high magnification. The microstructure of a material influences the mechanical properties viz., strength, toughness, hardness, corrosion resistance, temperature, wear resistance and so on. These salient properties consequently manage the appropriateness of the materials for industrial applications [1, 2]. Generally, microstructural analysis of composite materials can be carried out by means of optical microscope, scanning electron microscope and transmission electron microscope. Microstructural analysis of composites emphasize on the comprehensive characterization of materials.

Materials characterization refers to the broad and general process by which the structure of the material and properties are explored and measured. The characterization of materials is an essential process in the field of materials science, without which scientific comprehension of engineering materials cannot be established. The scale of the structures noticed in the characterization of materials usually ranges from angstroms to centimeters. Many techniques pertaining to the characterization of materials have been practiced from centuries. The advent of electron microscope and secondary ion mass spectrometry have created revolution in the twentieth century. The major instruments used for the characterization of materials are optical microscope, scanning electron microscope, transmission electron microscope, field ion microscope, scanning tunneling microscope and atomic force microscope. The other techniques used for the characterization of materials are energy dispersive x-ray spectroscopy, wavelength dispersive x-ray spectroscopy, mass spectrometry, secondary ion mass spectrometry and electron energy mass spectroscopy, auger electron microscopy, x-ray photoelectron microscopy, ultraviolet-visible spectroscopy, and thermoluminescence. These techniques characterize chemical composition, composition variation, crystal structure and photoelectric properties of materials.

Microstructural analysis and metallography are the predominant terms used in materials research. The characterization and analysis of microstructures have become extremely important in materials science and engineering. Materials fabricated with specific sizes and structures are expected to find a variety of applications. The discovery of novel materials, material processes, new experimental and theoretical approaches for research have good opportunities for the development of innovative materials. The discovery of novel materials and its characterization are expected to have an impact in areas such as electronics, medicine, energy management and biotechnology. The development of composite materials require advanced tools and skills to investigate at high magnification for the lucid comprehension of structures. For this purpose, specifically optical and electron microscopes are used [3, 4, 5].

One of the critical challenges in functional and engineering materials is the study of morphology controlled process based on the growth of crystal. Microstructural analyses of composite materials have been advantageous for the examination of cohesive interfacial bonding, porosity, particle size, grain size, and grain boundaries, inter dendritic segregation and dispersion concentration of the reinforcements. The properties of a material determine the level of performance for a specific application and the properties are dependent on the structure of the material [3, 4, 5, 6].

II. METALLOGRAPHIC SPECIMEN PREPARATION

Metallography is an investigative study of the microstructure of the materials. The microstructural analysis of materials benefit in determining the exact procedure about material processing. Microstructural analysis is regarded as a crucial step for the evaluation of product reliability. The fundamental steps involved in the metallographic specimen preparation are documentation, sectioning and cutting, mounting, grinding, polishing, buffing and etching [6, 7]. The metallographic procedure adopted for the preparation of the sample play a major role in the characterization of materials.

The fundamental steps involved in metallographic specimen preparation are as follows:

A. Documentation

This is a basic step that ensures the initial condition of the specimen and helps in carrying out microstructural analysis.

B. Sectioning & Cutting

Proper sectioning is required to restrict damage, which may lead to disparities in the microstructure and may result in improper metallographic characterization. The process of cutting has been achieved by the proper selection of abrasive

type, bonding and size, cutting speed, load and application of coolant.

C. Mounting

This step involves three most important functions viz., protection of the edge of the specimen, fills voids in porous materials and improves handling of asymmetrical shaped samples. Mounting has been accomplished by encapsulating the specimen into a compression mounting compound by using a thermosetting plastic.

D. Grinding

The surface layers damaged by the process of cutting will be eliminated by grinding. The specimens that are mounted are to be ground with rotating discs of abrasive paper. The grinding involves several stages. Every stage of grinding eliminates the scratches or striations from the previous coarser paper. This has been easily achieved by orienting the specimen perpendicular to the previous scratches. The specimens have been washed thoroughly with soap water to prevent contamination from coarser grit present on the specimen surface. Characteristically, the optimum grade of paper used is 1200.

E. Polishing

The purpose of polishing is to remove the surface damage. Polishing discs have been covered with soft cloth impregnated with abrasive diamond particles and an oily lubricant or water lubricant. Particles with two different grades have been used : a coarser polish - typically with diamond particles 6 microns in diameter which should remove the scratches produced from the finest grinding stage, and a finer polish – typically with diamond particles 1 micron in diameter, to generate an even surface. Before using a finer polishing wheel, the specimen should be washed thoroughly with warm soapy water followed by alcohol to prevent flaw of the disc. Some of the polishing cloths commonly used are metal mesh cloth, polyester polishing pads, pellon pads, parametric polymer pads and nylon polishing pads.

F. Etching

Etching has been accomplished to reveal the microstructure of the metal through selective chemical process. In alloys with more than one phase, etching creates contrast between different regions through differences in topography or the reflectivity of the distinct phases. The rate of etching will be influenced by crystallographic orientation, so variations have been observed between the grains. This has been resulted in a surface relief that has facilitated grain boundaries, phases and precipitates which can be easily distinguished. Generally, Keller's reagent is used for Aluminium matrix composites, which generally comprises the composition hydrofluoric acid, nitric acid, hydrochloric acid and distilled water.

III. Analysis of Composite Materials

Microstructural analyses of composites have been advantageous for mechanical and thermal characterization of composite materials. The examination of dispersion concentration of the reinforcements, cohesive interfacial bonding, formation of grain boundaries and interdendritic segregation in hybrid composites will influence the determination of mechanical and thermal properties of composites viz., tensile strength, moduli of elasticity, thermal conductivity and thermal expansively [8, 9]. Hence, microstructural characterization has to be carried out to accomplish thermal, mechanical and tribological characterization having desired percentage combinations.

Microstructural characterization with desired percentage reinforcements of hybrid metal matrix composites can be accomplished by employing Optical Microscope, Scanning Electron Microscope and Energy Dispersive X-ray Spectroscope. Optical Microscope can be used to investigate the formation of grain boundaries and interdendritic segregation. The microstructures with usually magnification 200X and 500X shall be used to depict the behavior of composites. Scanning Electron Microscope (SEM) is generally used to examine the porosity, particle sizes and dispersion concentration of the reinforcements. Scanning Electron Microscope coupled to an Energy Dispersive X-ray Spectroscope (EDAX or EDS) shall be used for the chemical or elemental characterization of hybrid composites. EDAX characterizes the elemental composition of hybrid metal matrix composites.

IV. Regular Characterization Methods

An optical microscope is a special type of microscope that utilizes visible light and a system of lenses to magnify images of small samples. The image from an optical microscope can be captured by normal light sensitive camera to generate a micrograph. It is preferably suited for the examination of microstructure, grain size, inclusion rating, nodular count, particle size, coating layer thickness and depth of carburization. This high resolution microscope has a magnification, ranging from 50X to 1000X with computer interface and is equipped with sophisticated and high resolution Charged Coupled Device (CCD) camera with user friendly software [7].

A Scanning Electron Microscope (SEM) is an electron microscope that generates image of a particular sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample and thus produce the signals quickly. It furnishes information about the topography and composition of the surface of the sample. The electron beam will be normally scanned in a raster scan pattern and the position of the electron beam can be combined with the detected signal to produce an image. The most common mode of SEM is the detection of secondary electrons emitted by means of atoms excited by an electron beam. The types of signals that are produced by the use of a scanning electron microscope include secondary electrons, back-scattered electrons, characteristic x-rays and cathodoluminescence, specimen current and transmitted electrons. In this research work, the type of signal produced by SEM is Back Scattered Electrons (BSE). Back scattered electrons are typically the beam electrons that are reflected from the sample by elastic scattering. The images that are produced by back-scattered electrons can make available with accurate information about the distribution of different elements in the sample.

SEM is a powerful tool to comprehend the crystal growth morphology and provides assistance for micro and nanofabrication. Solution based strategies can be employed in this instrument because they can facilitate chemical flexibility and synthetic tunability. The uniform distribution of the reinforcements is an indispensable condition for a composite material to accomplish its performance at extreme superiority [105, 106]. The microstructural examination of hybrid metal matrix composites have been accomplished to characterize dispersoid concentration of the reinforcements, particle size and porosity. The evolution of microstructure, specifically in terms of size, shape and distribution of the reinforcements have been assessed by a systematic approach. The microstructural analyses of hybrid composites have been advantageous to study the morphology and presence of porosity comprehensively [8, 9, 10].

In materials characterization, it is essential to determine the elemental or chemical composition of a composite. Generally, energy dispersive x-ray spectroscope is used to carry out elemental or chemical characterization. In this research work, the microstructural analyses of hybrid composites have been carried out by means of Scanning Electron Microscope coupled to an Energy Dispersive X-ray Spectroscope (EDAX). The primary components of the EDAX setup are the excitation source, x-ray detector, pulse processor and analyzer. It relies on the contact of x-ray excitation and a sample. The detector that has been used commonly is the Silicon-Lithium detector.

REFERENCES

[1] Fei Liu, Junshu Wu, Kunfeng Chen and Dongfeng Xue, "Morphology study using Scanning Electron Microscopy", Microscopy, Science, Technology, Applications and Education, A Mendez-Vilas and J Diaz (Eds.), 2005.
[2] Donald C. Zipperian, "Metallographic specimen preparation basics", Pace Technologies.
[3] Advanced Metallurgical Laboratory Manual, Department of Science and Technology, Government of India.
[4] Gaurav Mahajan, Nikhil Karve, Uday Patil, P Kuppan and K Venkatesan, "Analysis of microstructure, hardness and wear of Al-SiC-TiB_2 hybrid metal matrix composites", International Journal of Science and Technology, ISSN: 0974 - 5645, Vol. 8, No. 5, 2015, pp. 101-105.
[5] Hailong Wang, Rui Zhang, Xing Hu, Chang-An Wang and Yong Huang, "Characterization of a powder metallurgy SiC/Cu-Al composite", Journal of Materials Processing Technology, Vol. 197, Issues 1-3, 2008, pp. 43-48.
[6] J.M. Molina, J. Narciso, L. Weber, A. Mortensen and E. Louis, "Thermal conductivity of Aluminium-Silicon Carbide composites with monomodal and bimodal particle size distribution", Materials Science and Engineering A, Vol. 56, Issue 2, 2008, pp. 483-488.
[7] Qiang Zhang, G. Wu, Guoqin Chen, Longtao Jiang and Bofeng Luan, "The thermal expansion and mechanical properties of high reinforcement content SiC_p/Al composites fabricated by squeeze casting technology", Composites: Part A, Vol. 34, 2003, pp. 1023-1027.
[8] Ali Mazahery and Mohsen Ostad Shabani, "Microstructural and abrasive wear properties of SiC reinforced Aluminium-based composite produced by compocasting", Transactions of Nonferrous Metals, Society of China, 2013, pp. 1905-1914.
[9] Manual on S-3400 Variable Pressure Scanning Electron Microscope, Hitachi High-Technologies Corporation, Japan.
[10] Amarnath G. and K.V. Sharma, "Microstructure and mechanical properties of nanoparticulate WC/Al metal matrix composites", International Journal of Research in Engineering and Science", ISSN: 2320 - 9364, Vol. 1, Issue 7, 2013, pp. 1-7.

