



**INVESTIGATING SOME THERMAL, MECHANICAL, AND  
MICROSTRUCTURE BEHAVIOUR OF ALUMINUM-EGGSHELL  
COMPOSITE WARES**

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ENGINEERING.**

**APRIL, 2025**

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**A PROJECT REPORT SUBMITTED TO THE DEPARTMENT OF  
MATERIALS AND METALLURGICAL ENGINEERING, FACULTY OF  
ENGINEERING, UNIVERSITY OF BENIN, BENIN CITY**

**IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE  
AWARD OF BACHELOR OF ENGINEERING (B.Eng.) HONOURS  
DEGREE IN MATERIALS AND METALLURGICAL ENGINEERING.**

**APRIL, 2025**

## CERTIFICATION

This is to certify that the project work titled “Investigating some Thermal, Mechanical, and Microstructure Behaviour of Aluminum-Eggshells Composite” was carried out by:

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of the Department of Materials and Metallurgical Engineering, Faculty of Engineering, University of Benin, in partial fulfilment of the requirements for the award of Bachelor of Engineering, (B.Eng.) in Materials and Metallurgical Engineering.

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(HEAD OF DEPARTMENT)

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Date

## **DEDICATION**

We hereby dedicate this project work to Almighty God who has been our ultimate source of strength, good health and sustenance. Also, for flourishing us with the requisite fortitude to embark on and complete this project.

## **ACKNOWLEDGEMENT**

This project work would have been an uphill task to complete without the ever available and attainable help and encouragement of our project supervisor Engr. J. Jesumirhewe, our respective family members, the entire staff of the Department of Materials and Metallurgical Engineering, Faculty of Engineering; The Department of Production Engineering; The Department of Mechanical Engineering; The Department of Civil Engineering, University of Benin; and Department of Material Science and Engineering, Obafemi Awolowo University, Osun State.

We specially want to acknowledge the contributions of the Head of Materials and Metallurgical Engineering Department, Dr. U. G. Unueroh and our project supervisor; for the contributions they made to facilitate the completion of our project work on time.

Finally, yet importantly, we would like to express our gratitude to our friends, for in their own way of wishes and moral support, assisted us to achieve our project.

## **ABSTRACT**

*This study investigates the potential of eggshell waste as a reinforcement material in aluminum matrices for kitchenware applications, aiming to enhance material properties.*

*Composites were fabricated with 7%, 10%, and 13% eggshell reinforcement and subjected to tensile testing, Brinell hardness testing, Differential Scanning Calorimetry (DSC), Scanning Electron Microscopy (SEM), and Energy-Dispersive Spectroscopy (EDS) to assess mechanical, thermal, and microstructural properties. Tensile testing revealed a significant increase in Ultimate Tensile Strength (UTS) with 13% reinforcement, reaching 134.29 MPa, though ductility was reduced. SEM analysis of the 10wt% composite showed a finer textured structure but non-uniform particle distribution. EDS confirmed calcium presence, and showed reduced oxygen content. Brinell hardness exhibited a positive correlation between the weight percentage of eggshell in the aluminum composite, which showed that higher eggshell content within the tested range leads to increased hardness. DSC indicated that eggshell addition altered thermal characteristics, with the 13wt% composite showing a slightly higher melting temperature and changes in heat of fusion.*

*These results demonstrate that eggshell reinforcement enhances the tensile strength, hardness and modifies the thermal behaviour of aluminum.*

## TABLE OF CONTENT

COVER PAGE	i
TITLE PAGE	ii
CERTIFICATION	iii
DEDICATION	iv
ACKNOWLEDGEMENTS	v
ABSTRACT	vi
TABLE OF CONTENTS	vii
LIST OF TABLES	x
LIST OF FIGURES	xi
LIST OF PLATES	xii
CHAPTER ONE	1
INTRODUCTION	1
1.1 Background of study	1
1.2 Statement of problem	3
1.3 Aim and objectives of the study	4
1.3.1 Aim of the study	4
1.3.2 Objectives of the study	4
1.5 Significance of the study	4
1.6 Scope and limitations of the study	5
CHAPTER TWO	7
LITERATURE REVIEW	7
2.1 Theory of composite materials	7
2.2 Aluminum matrix composites (AMCs)	7
2.3 Reinforcement of aluminum	7
2.4 Types of reinforcements	8
2.5 Role of reinforcements	8
2.6 Processing of aluminum matrix composites(AMCs)	8
2.7 Properties of aluminum matrix composites	9
2.7.1 Mechanical Properties	9
2.7.2 Physical Properties	9

2.8 Applications of aluminum matrix composites(AMCs)	10
2.9 Eggshell powder as reinforcement	11
(a) Composition and Structure of Eggshells	11
2.9.1 Thermal and mechanical properties of composites	13
2.9.2 Highlighting casting as a fundamental manufacturing process	14
2.9.3. Heat flow in aluminum matrix composites (AMCs) and its relevance	16
2.9.4 Previous works and research gap	18
2.9.5. Applications of the work	20
CHAPTER 3	27
MATERIALS AND METHODS	27
3.1 Materials	27
3.2 Equipment	28
3.3. Method	29
3.3.1. Eggshells processing	29
3.3.2 Determination of sample (prototype) weight	30
3.3.3 The aluminum-eggshell composites were fabricated using the sand mold casting method.	30
3.3.4 Characterization techniques	31
3.3.5 Microstructural analysis	31
3.3.6 Hardness test	34
3.3.7 Tensile test	36
ii). DSC curves	43
CHAPTER 4	46
RESULTS AND DISCUSSION	46
Table 4.1: Chemical composition of aluminum alloy	46
Table 4.2: Weight percentage of composite compositions	46
4.1.1 Analysis the EDS data of aluminum spoon	55
4.1.2 Analysis of the 10 wt.% Eggshell Composite (S1):	56
4.1.3 Discussion and comparisons of the SEM images of 10wt.% eggshell sample(S1) and aluminum spoon	56
4.1.4 Analysis of the EDS Data for S1:	57
4.2. Analysis of the Brinell hardness data	57
Table 4.3: Brinell hardness data	57
4.2.1 Effect of eggshells on hardness and mechanical behavior	57
4.2.2 Overall mechanical behavior:	58

4.3 Analysis of the tensile test data	61
Table 4.4: Tensile test data	61
4.3.1 Pure aluminum baseline:	61
4.3.2 Effect of eggshell reinforcement:	61
4.3.3 Optimum reinforcement percentage:	62
4.3.4 Microstructural implications:	62
4.4.2 Analysis of the DSC curve of S2(13wt.% eggshell)	66
CHAPTER 5	68
CONCLUSION AND RECOMMENDATION	68
5.1 Conclusion	68
5.2 Recommendation	69
REFERENCES	71
APPENDIX I	75
APPENDIX II	76
APPENDIX III	77
APPENDIX IV	78
APPENDIX V	79

## LIST OF TABLES

Table 4.1 Chemical Composition of Aluminum Alloy	46
Table 4.2 Weight Percentage of Composite Composition	46
Table 4.3 Brinell Hardness Data	57
Table 4.4 Tensile Test Data	61

## LIST OF FIGURES

Figure 2.1 Image Showing Metal Matrix Composites Classification-----	10
Figure 2.2 Sand Casting Process -----	16
Figure 2.3 Aluminum Utensils -----	26
Figure 3.1 SEM-EDS (JOEL JSM760F)-----	33
Figure 3.2 Monsanto tensometer (Type W) -----	36
Figure 3.3 Representation of Elongation-----	37
Figure 3.4 Stress and Strain Curve for Brittle. Ductile and Elastomeric Materials -----	40
Figure 3.5 Avery Dennison Tensile Testing Machine -----	41
Figure 3.6 DSC Curve Obtained on Heating Samples from an Amorphous Ribbon of Fe <sub>73</sub> Si <sub>16</sub> B <sub>7</sub> Nb <sub>3</sub> Cu Alloy-----	43
Figure 3.7 Differential Scanning Calorimetry Machine -----	45
Figure 4.9 Bar Chart of Hardness Variation in Samples-----	59
Figure 4.9.1 Graph of the Hardness Variation in Samples -----	60
Figure 4.9.2 Tensile strength (MPa) vs wt.% Eggshell addition -----	63
Figure 4.9.3 DSC Graph of Heat Flow Vs Temperature for Pure Aluminum Sample -----	64
Figure 4.9.4 DSC Graph of Heat Flow Vs Temperature for S2(Al+13wt.% Eggshell)-----	65

## LIST OF PLATES

Plate 4.1 SEM Result of Aluminum Spoon (Prototype)with 10000x Magnification)-----	47
Plate 4.2 SEM Result of Aluminum Spoon (Prototype) with 9000x Magnification ----	48
Plate 4.3 SEM Result of Aluminum Spoon (Prototype) with 8000x Magnification -----	49
Plate 4.4 EDS Results of Aluminum Spoon (Prototype) -----	50
Plate 4.5 SEM Result of Aluminum Alloy with 10wt.% Eggshell Composition (10000x Magnification) -----	51
Plate 4.6 SEM Result of Aluminum Alloy with 10wt.% Eggshell Composition (9000x Magnification) -----	52
Plate 4.7 SEM Result of Aluminum Alloy with 10wt.% Eggshell Composition (8000x Magnification) -----	53
Plate 4.8 EDS Results of Aluminum Alloy with 10wt.% Eggshell Composition-----	54

# CHAPTER ONE

## INTRODUCTION

### 1.1 Background of study

In kitchens around the world, tools like spoons, spatulas, and ladles are indispensable. They facilitate a wide range of cooking tasks and are used multiple times daily. However, the performance and safety of these tools largely depend on the materials from which they are made. Aluminum, with its lightweight, affordability, and corrosion resistance, has become a preferred choice for manufacturers in producing kitchen utensils. It is particularly valued for its utensils of other metals. Aluminum also has a profound ability of forming metal matrix composites. Composites are structural material composed of a duly arranged mixture or the combination of two or more micro, macro or Nano constituents with interfaces. The constituents are combined at a macroscopic level and which are effectively insoluble in each other. The discrete constituent is called the reinforcement and the continuous phase is called the matrix. According to the chemical nature of the matrix phase, composite materials are classified as metal matrix (MMC), polymer matrix (PMC) and ceramic matrix composites (CMC). Low cost reinforcement has always been preferable in all kinds of applications. The production and utilization of using by-products from the industry as reinforcement has given better mechanical properties and wear properties in the earlier studies. (Smith and Hashemi, 2008). Due to the tailored mechanical, physical and thermal properties including their superior abrasion and wear resistance, low densities, improved thermal and electrical conductivity, better strength to density ratio, etc., Metal Matrix Composites (MMC) have been broadly used as a class of materials for the aerospace, electronics, automotive, and wear applications. (Aigbodion and Hassan, 2007; Naresh, 2006).

However, aluminum's high thermal conductivity can lead to excessive heat transfer, making utensil handles hot to the touch during cooking. This issue can compromise the user's comfort and safety, posing risks of burns or discomfort. Historically, the industry has addressed this issue by adding synthetic handle covers made from materials like rubber or plastic. These insulating covers reduce heat transfer to some extent, providing a cooler surface for the user to hold. Nevertheless, these materials are often susceptible to wear and tear due to frequent exposure to

high temperatures and washing. Over time, they may become less effective at insulation, or even crack and break, diminishing the utensil's lifespan. Additionally, the environmental impact (unfavorable disposal option) of these synthetic materials like the thermosetting plastics is detrimental due to the long-lasting non-degradable characteristics of thermoset waste which is an ongoing concern (Huaguo, 2023). With growing awareness of the need for sustainable manufacturing, the use of natural, biodegradable, or renewable materials in consumer products is becoming a priority in many industries (McKinsey, 2020).

Thermal insulation reduces heat transfer between solid objects, fluids, or gases by introducing a barrier between them. Insulation can be critical for a range of industries including energy, industrial, medical, and more. These complex applications demand optimal thermal environments for efficient performance. They use a variety of insulation technologies to solve thermal challenges within their system. Thermal insulation materials limit the rate of heat flow between objects or substances. Materials with the lowest thermal conductivity will provide the highest performance insulation. In addition to thermal conductivity, other physical properties may be considered when selecting insulation materials, such as: density, thickness, specific heat capacity, thermal resistance and thermal diffusivity (Concept Group LLC, 2021)

This study focuses on using eggshells as an additive in aluminum to address the problem of heat transfer in utensils. Eggshells has naturally low thermal conductivity, making it a promising candidate for enhancing the insulating properties of metal composites. It is widely available and, compared to synthetic materials, is environmentally benign and economically viable. Although earlier studies have proved that Chicken Egg Shell (ES) is an agricultural waste mainly considered as futile and discarded mostly because it contributes to pollution of the environment, especially in those countries where the egg product industry is well developed. By integrating eggshells directly into the aluminum casting process, the project seeks to produce kitchen utensils that not only provide improved heat insulation but also contribute to sustainable and environmentally friendly production methods.

It is worthy of note that metal matrix composites (MMCs) are advanced engineering materials consisting of metal (e.g., Al, Cu, Mg, Ti) as the matrix and the second reinforcing/strengthening phase (e.g.,  $\text{Al}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$ , SiC, TiC,  $\text{B}_4\text{C}$ ,  $\text{Cr}_3\text{C}_2$ , BN,  $\text{Si}_3\text{N}_4$ , carbon nanotubes, etc.) to produce properties different from the parent or constituent materials. The reinforcing phase enhances the needed strength and stiffness properties of the composites, and

this strengthening phase could be in the form of short/discontinuous fibers/whiskers or continuous fibers or particulates. In general, the reinforcement of composite materials serves different purposes based on the specific needs for their desired applications. In many instances, all the expected material properties for a particular application may not be found in a single monolithic material; hence, the need to combine two or more materials to achieve these desired properties. Some of these properties (Kainer, 2006; Chung, 2010; Macke et al., 2012) for which composites are made are highlighted below

1. Improved yield and tensile strengths at ambient and elevated temperatures.
2. Increased stiffness at room and high temperatures.
3. Enhanced chemical and corrosion resistance.
4. Increased thermal insulation or conductivity.
5. Improved thermal shock stability.
6. Increased electrical insulation or conductivity.
7. Improved fatigue resistance, especially at higher temperatures.
8. Enhanced abrasion and wear resistance.
9. Controlled thermal expansion coefficient.
10. Reduced material weight (low density).
11. Improved fracture toughness and damage tolerance.
12. Improved creep resistance.
13. Enhanced energy absorption and vibration damping capabilities.
14. Improved electromagnetic shielding effectiveness.

Interestingly, due to the possibility that the properties of composite materials can be engineered to fit the desired engineering application, composites have been employed in the design and manufacturing of diverse engineering components in various technological sectors.

## **1.2 Statement of problem**

Despite aluminum's favorable properties as a material for kitchen utensils, its high thermal conductivity leads to significant heat transfer to the handles during use, making them uncomfortable and potentially dangerous for users. Traditional solutions such as adding plastic or rubber insulation to the handles only provide a partial fix. These materials can degrade over time, especially with prolonged exposure to heat, and are not ideal from an environmental standpoint as they contribute to plastic waste. This project explores a different approach by integrating eggshells directly into the aluminum casting process, creating a composite material

that has better insulating properties without the need for additional handle covers. Eggshell's insulating properties can reduce heat transfer through the utensil, enhancing user comfort and safety, with the possibility of not compromising strength and eliminating the need for synthetic handle covers. This solution, if successful, could address both the safety concerns of users and the sustainability demands of modern consumers.

### **1.3 Aim and objectives of the study**

#### **1.3.1 Aim of the study**

To develop an aluminum-based kitchen utensil with improved thermal insulation properties by incorporating eggshells as an additive.

#### **1.3.2 Objectives of the study**

To achieve the aim, the following objectives have been established

- I. Determination of the optimal eggshell- aluminum ratio for casting
- II. Determination of thermal and some mechanical properties responses of the casts
- III. Establishment of relationship between sizes of eggshell reinforcement and thermal behavior of the material

### **1.5 Significance of the study**

This study has both practical and theoretical significance, as it aims to solve a widespread problem in kitchenware while advancing knowledge in material science, specifically in the use of natural additives for composite materials. The significance of this project is multi-faceted, with implications for user safety, sustainability, economic impact, and research on thermally insulating materials.

- **Enhanced user safety and comfort:** the project's primary contribution lies in improving the safety and usability of kitchen utensils. By incorporating eggshell into the aluminum casting, the utensil's handles are expected to remain cooler, allowing users to cook comfortably without the risk of burns or discomfort. This benefit is especially significant for home cooks and professional chefs who use these tools frequently.
- **Environmental sustainability:** integrating eggshells into kitchen utensils supports the shift toward more environmentally sustainable manufacturing practices. Unlike plastic and rubber,

eggshell is a natural resource. using it as an insulating additive reduces dependency on synthetic materials that contribute to plastic waste. this project could thus set a precedent for future research on biodegradable or renewable additives in kitchenware and other consumer products, aligning with global efforts to reduce plastic consumption and environmental degradation.

- **Advancement in composite material research:** this project contributes to the growing field of composite materials by exploring how natural additives, like eggshell can enhance specific properties, in this case, thermal insulation. Aluminum is widely used in many industries due to its mechanical properties, but thermal management remains a challenge in applications where direct heat transfer is undesirable. the study's findings could inspire similar innovations in other industries, such as automotive and electronics, where managing heat without compromising structural integrity is critical.

- **Innovation in kitchenware design:** beyond the functional aspects, this study opens up possibilities for innovative kitchen utensil designs that are both practical and eco-conscious. By eliminating the need for extra insulation materials, this research could inspire designs that are minimalistic, durable, and sustainable. This project's emphasis on a natural, integrated solution rather than add-on components aligns with consumer trends that favor simplicity, durability, and environmental responsibility in product design.

- **Consumer health and well-being:** the choice of material for kitchen utensils has implications for consumer health. While synthetic materials can sometimes leach harmful substances into food at high temperatures, the natural and inert properties of eggshells make it a safer choice. Incorporating eggshells into aluminum kitchenware could thus provide consumers with an alternative that supports a healthier cooking experience.

## **1.6 Scope and limitations of the study**

This research focuses on the development, testing, and assessment of aluminum-based kitchen utensils enhanced with eggshells for improved thermal insulation. The study will encompass the following stages:

- **Material preparation:** drying, grinding, sieving, and weighing of eggshells
- **Composite fabrication and optimization:** conduction of series of experimental castings, varying the ratio of eggshells to aluminum to optimize thermal insulation without compromising mechanical strength.

- Laboratory testing: perform controlled testing to assess thermal conductivity, tensile strength, and durability of the aluminum-eggshell composite, comparing it to standard aluminum kitchen utensils
- Prototype development and evaluation: produce prototype utensils using the composite and conduct usability testing to assess insulation performance and practical application.

However, several key limitations must be acknowledged despite the inclusion of various analytical methods such as scanning electron microscopy (SEM), differential scanning calorimetry (DSC), tensile testing, and hardness testing. One significant constraint is the intentional exclusion of a detailed analysis of corrosion resistance. While aluminum is generally recognized for its corrosion resistance due to the formation of a protective oxide layer, the introduction of eggshells could alter this property. As a result, the study does not explore how the addition of eggshells affects the material's susceptibility to various corrosive environments, which is particularly important for kitchen utensils exposed to moisture and acidic substances.

## CHAPTER TWO

### LITERATURE REVIEW

#### 2.1 Theory of composite materials

Composite materials are defined as macroscopic combinations of two or more distinct materials, possessing a distinct interface between them (Callister & Rethwisch, 2018). These materials are often classified based on the matrix material, leading to categories like polymer matrix composites (PMCs), metal matrix composites (MMCs), and ceramic matrix composites (CMCs). This project focuses on MMCs, where a metallic matrix, in this case, aluminum, is reinforced with a ceramic or particulate material. The "rule of mixtures" provides a simplified estimate of composite properties based on the volume fraction and properties of the constituents (Agarwal & Broutman, 1990). However, this rule often deviates from experimental results due to factors like interface bonding, particle distribution, and processing conditions. The interface, the boundary between the matrix and reinforcement, is critical for load transfer and overall composite performance (Chawla, 2012). Strong interfacial bonding is essential for effective stress transfer from the matrix to the reinforcement, maximizing the composite's strength and other properties.

#### 2.2 Aluminum matrix composites (AMCs)

Aluminum is a widely used matrix material due to its low density, high strength-to-weight ratio, good corrosion resistance, and ease of processing (Miracle, 2001). These attributes make aluminum and its alloys attractive for various applications, including aerospace, automotive, and consumer goods. Aluminum matrix composites (AMCs) further enhance these properties by incorporating reinforcing materials, leading to improved strength, stiffness, wear resistance, and, potentially, thermal properties.

#### 2.3 Reinforcement of aluminum

To overcome the limitations of monolithic aluminum, reinforcing materials are incorporated into the aluminum matrix to enhance its mechanical and physical properties. The resulting materials, known as AMCs, exhibit improved strength, stiffness, wear resistance, and, potentially, thermal properties.

## 2.4 Types of reinforcements

Various types of reinforcing materials are used in AMCs, including:

- a) Particles: Ceramic particles such as silicon carbide (SiC), alumina (Al<sub>2</sub>O<sub>3</sub>), and boron carbide (B<sub>4</sub>C) are commonly used due to their high hardness, strength, and stiffness (Hashim et al., 2001).
- b) Fibers: Continuous or discontinuous fibers, such as carbon fibers, glass fibers, and ceramic fibers, provide high strength and stiffness in the direction of reinforcement (Chawla, 2012).
- c) Whiskers: Single-crystal fibers with high aspect ratios, such as silicon carbide whiskers, offer excellent reinforcement potential (Kwon et al., 1993).

## 2.5 Role of reinforcements

The incorporation of reinforcement in the aluminum matrix leads to several strengthening mechanisms, including:

- Load transfer: The reinforcement carries a significant portion of the applied load, thereby increasing the overall strength and stiffness of the composite (Kelly, 1973).
- Orowan strengthening: The presence of hard reinforcing particles hinders the movement of dislocations, leading to increased strength (Orowan, 1948).
- Hall-Petch strengthening: The grain refinement of the aluminum matrix due to the presence of reinforcement contributes to increased strength (Hall, 1951; Petch, 1953).

## 2.6 Processing of aluminum matrix composites(AMCs)

Various processing techniques are employed to fabricate AMCs, including:

- Liquid metallurgy: This involves incorporating the reinforcement into molten aluminum, followed by casting or solidification (Surappa, 2003).
- Powder metallurgy: This involves blending aluminum powder with reinforcement particles, followed by compaction and sintering (German, 1990).
- Solid-state processing: This involves consolidating aluminum and reinforcement materials in the solid state, such as by diffusion bonding or friction stir processing (Mishra et al., 2014).

## **2.7 Properties of aluminum matrix composites**

AMCs exhibit a wide range of properties depending on the type, volume fraction, and distribution of reinforcement, as well as the processing technique employed.

### **2.7.1 Mechanical Properties**

- a) **Strength and stiffness:** AMCs generally exhibit higher strength and stiffness compared to unreinforced aluminum
- b) **Wear resistance:** The incorporation of hard ceramic particles significantly improves the wear resistance of AMCs (Bhansali and Lloyd, 1998).
- c) **Fatigue resistance:** The fatigue behavior of AMCs is influenced by various factors, including the type of reinforcement, matrix microstructure, and loading conditions (Llorca, 2002).

### **2.7.2 Physical Properties**

- a) **Density:** The density of AMCs depends on the density of the reinforcement and its volume fraction (Jones, 1999).
- b) **Thermal conductivity:** The thermal conductivity of AMCs can be tailored by selecting appropriate reinforcement materials (Callister, 2007).
- c) **Coefficient of thermal expansion:** The coefficient of thermal expansion of AMCs is influenced by the thermal expansion behavior of the matrix and reinforcement (Dieter, 1988).

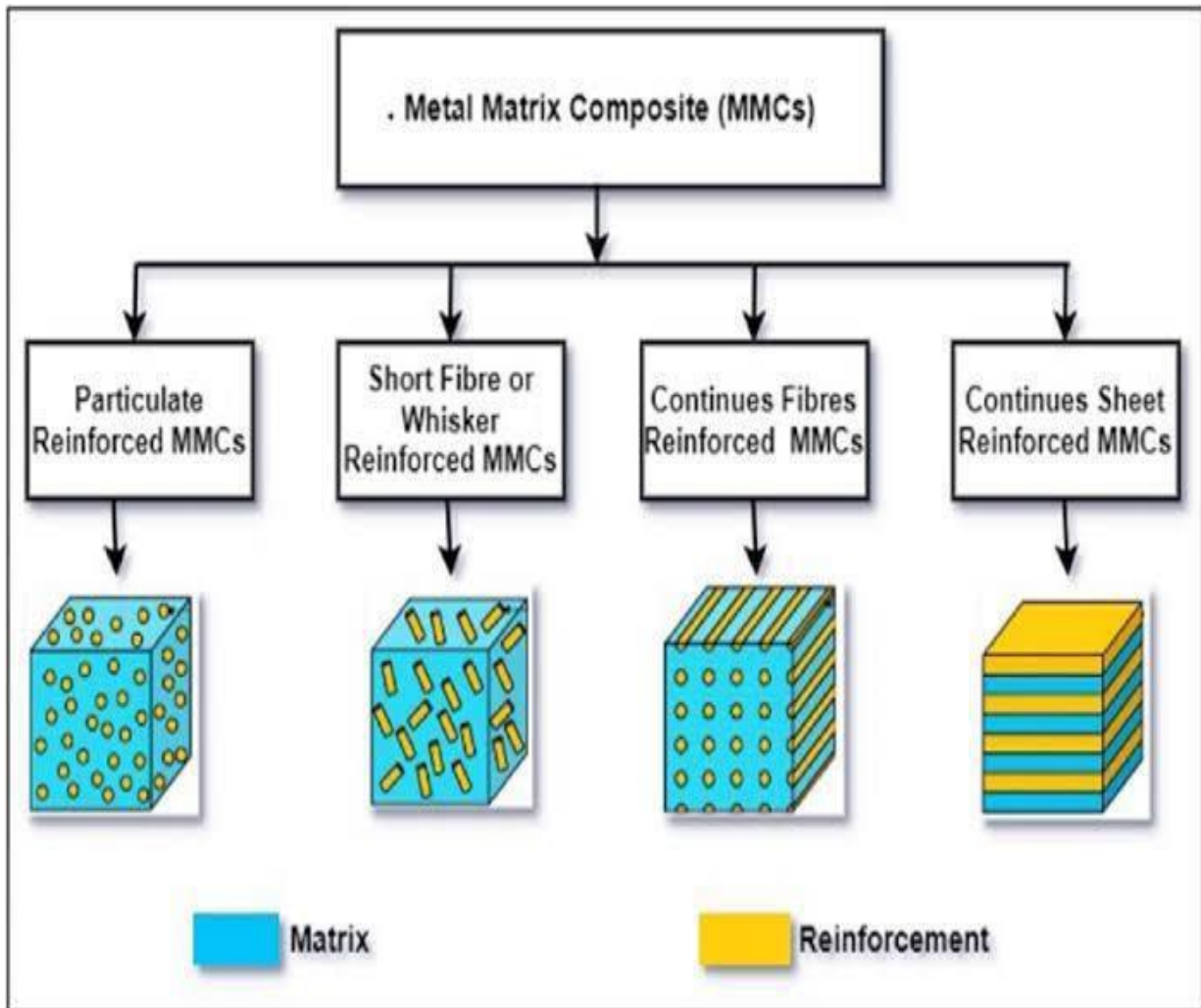


Figure 2.1: Image showing metal matrix composites c (Endalkachew, 2024)

## 2.8 Applications of aluminum matrix composites(AMCs)

AMCs have found applications in various fields, including:

- **Aerospace:** AMCs are used in aircraft components such as fuselage panels, wing structures, and engine parts due to their high strength-to-weight ratio and stiffness (Peters et al., 1998).
- **Automotive:** AMCs are used in automotive components such as brake rotors, pistons, and connecting rods due to their high wear resistance and fatigue strength (Kainer, 2006).
- **Sports and recreation:** AMCs are used in sports equipment such as bicycles, skis, and golf clubs due to their lightweight and high stiffness (Cornie et al., 1991).

## **2.9 Eggshell powder as reinforcement**

Eggshells are primarily composed of calcium carbonate ( $\text{CaCO}_3$ ) (approximately 94%), with trace amounts of other minerals like magnesium carbonate, calcium phosphate, and organic matter (Stadelman, 1995). Calcium carbonate possesses desirable properties, including high thermal stability and relatively low thermal conductivity (though specific values vary depending on the form and density). This makes it a promising candidate for enhancing the thermal insulation of aluminum composites. Moreover, utilizing waste eggshells offers a sustainable and cost-effective approach to material development, addressing environmental concerns related to waste disposal.

### **(a) Composition and Structure of Eggshells**

Eggshells have a complex hierarchical structure consisting of several layers: the cuticle (outermost layer), the spongy layer (main layer), and the mammillary layer (innermost layer). The spongy layer, which constitutes the majority of the eggshell thickness, is composed of calcite crystals arranged in a palisade-like structure. This structure contributes to the mechanical strength of the eggshell. The chemical composition of eggshells is typically:

- Calcium carbonate ( $\text{CaCO}_3$ ): ~94%
- Magnesium carbonate ( $\text{MgCO}_3$ ): ~1%
- Calcium phosphate ( $\text{Ca}_3(\text{PO}_4)_2$ ): ~1%
- Organic matter: ~4% (mainly proteins and polysaccharides) (Stadelman, 1995)

### **(b) Processing of Eggshell Powder**

To utilize eggshells as reinforcement, they need to be processed into powder form. The typical processing steps include:

1. **Cleaning:** Removal of adhering organic matter (e.g., egg white, yolk) by washing with water and sometimes detergents.
2. **Drying:** Drying the cleaned eggshells in an oven or under sunlight to remove moisture.
3. **Crushing/Grinding:** Reducing the size of the dried eggshells using a crusher, grinder, or ball mill to obtain a fine powder.
4. **Sieving:** Separating the powder into different size fractions using sieves to achieve a desired particle size distribution

5. Calcination (Optional): Heating the eggshell powder at high temperatures (e.g., 800-900°C) to remove organic matter and convert  $\text{CaCO}_3$  to  $\text{CaO}$  (calcium oxide). This can influence the interfacial bonding in the composite (Baláž et al., 2017).

### **(c) Eggshell powder as reinforcement in aluminum matrix composites: mechanisms and effects**

The incorporation of eggshell powder into an aluminum matrix can influence the composite's properties through several mechanisms:

- **Strengthening:** The hard  $\text{CaCO}_3$  particles act as obstacles to dislocation movement, contributing to increased strength and hardness (similar to other ceramic particle reinforcements).
- **Stiffness enhancement:** The high modulus of  $\text{CaCO}_3$  can increase the overall stiffness of the composite, although the effect might be less pronounced compared to reinforcements like  $\text{SiC}$  or  $\text{Al}_2\text{O}_3$ .
- **Interfacial bonding:** The quality of the interface between the aluminum matrix and the eggshell powder is crucial for effective load transfer. Factors like particle size, distribution, and processing parameters influence the interfacial bonding. Calcination of eggshell powder can affect the interfacial reactions and bonding characteristics.
- **Porosity:** The presence of eggshell powder can sometimes lead to increased porosity in the composite, which can negatively affect mechanical properties. Careful control of processing parameters is essential to minimize porosity.
- **Tribological properties:** Studies have shown that the addition of ESP can improve the wear resistance of aluminum composites, potentially due to the lubricating effect of calcium oxide formed during processing or testing (Rajeshkumar et al., 2018).

The incorporation of eggshell powder into an aluminum matrix influences both the thermal behavior and mechanical properties of the resulting composite. The presence of hard  $\text{CaCO}_3$  particles enhances the hardness and wear resistance of the aluminum matrix. (Dwivedi, 2014). However, the extent of improvement depends on factors such as particle size, distribution, and interfacial bonding. The thermal conductivity of the composite is also affected by the addition of eggshell powder, as  $\text{CaCO}_3$  has a lower thermal conductivity than aluminum. (Callister & Rethwisch, 2018).

## **(d) Advantages and Challenges of Using Eggshell powder**

### **I. Advantages:**

- Cost-effectiveness: ESP is a low-cost and readily available waste material.
- Sustainability: Utilizing ESP promotes waste recycling and reduces environmental impact.
- Potential for property enhancement: ESP can contribute to improved mechanical and tribological properties of AMCs.

### **II. Challenges:**

- Relatively low hardness compared to other ceramic reinforcements: The strengthening effect of ESP might be less pronounced compared to materials like SiC or Al<sub>2</sub>O<sub>3</sub>.
- Potential for porosity: Careful control of processing parameters is needed to minimize porosity.
- Interfacial bonding: Achieving strong interfacial bonding between the aluminum matrix and ESP can be challenging.
- Thermal stability of CaCO<sub>3</sub>: At high temperatures, CaCO<sub>3</sub> can decompose, which can affect the composite's properties.

## **2.9.1 Thermal and mechanical properties of composites**

### **(a) Thermal properties of composites**

Thermal conductivity, a measure of a material's ability to conduct heat, is a crucial property for kitchenware. Lower thermal conductivity translates to better thermal insulation, preventing heat transfer and improving user safety. The thermal conductivity of a composite is influenced by several factors, including the volume fraction of reinforcement, the thermal conductivities of the individual constituents, and the interfacial thermal resistance (Hasselman & Johnson, 1987). Introducing eggshell powder, with its lower thermal conductivity compared to aluminum, is expected to reduce the overall thermal conductivity of the composite.

### **(b) Mechanical properties of composites**

The mechanical properties of interest in this study include hardness, which indicates resistance to surface indentation. The addition of particulate reinforcement can influence these properties. The degree of improvement or degradation depends on factors such as particle size, distribution, and the strength of the interface bond (Clyne & Withers, 1993).

## 2.9.2 Highlighting casting as a fundamental manufacturing process

Casting is a manufacturing process in which a liquid material, typically molten metal, is poured into a mold containing a hollow cavity of the desired shape. The liquid solidifies within the mold, replicating the mold's form. The solidified part, known as the casting, is then removed from the mold.

Types of Casting Processes:

### 1. Sand casting:

- Description: Uses sand as the mold material. It's versatile and cost-effective for both small and large production runs.
- Applications: Engine blocks, large machine components, and artistic sculptures (Degarmo, et al., 2011).

### 2. Die casting:

- Description: Molten metal is injected under high pressure into a reusable metal mold (die). Ideal for high-volume production of complex shapes with good surface finish.
- Applications: Automotive parts, electronic housings, and small appliances. (Davis,2000).

### 3. Investment casting (Lost-Wax Casting):

- Description: A wax pattern is created, coated with a ceramic, and the wax is melted out, leaving a ceramic mold. Used for intricate shapes and high-quality castings.
- Applications: Turbine blades, jewelry, and medical implants. (Beeley,2001).

### 4. Stir casting:

- Description: A liquid metal matrix is stirred vigorously while reinforcing particles are introduced. This method is relatively simple and low-cost.
- Applications: Production of metal matrix composites, particularly for research and development. (Hashim. el al., 2001).

## 5. Squeeze Casting:

- Description: Molten metal is poured into a mold containing the reinforcement, and high pressure is applied during solidification. Improves density and interfacial bonding.
- Applications: High-strength automotive and aerospace components. (Chadwick,1996).

For metal matrix composite fabrication stir casting is commonly used. It is a relatively simple and cost-effective method suitable for producing particulate-reinforced MMCs (Hashim et al., 2001). In this process, the reinforcement particles are introduced into the molten metal while stirring to ensure uniform distribution. However, challenges such as particle segregation, porosity, and poor wettability between the matrix and reinforcement need to be addressed.

These challenges include the following:

- Wettability: Poor wettability between the molten metal and the reinforcement particles can lead to weak interfacial bonding and poor mechanical properties. (Asthana, 1998). Liquid metals processing: applications to materials manufacturing. Springer Science & Business Media.) Surface treatment of the particles or the addition of alloying elements can improve wettability
- Particle distribution: Achieving uniform particle distribution in the matrix is crucial for consistent material properties. Agglomeration and settling of particles can occur due to density differences and interparticle forces. (Surappa, 2003).
- Interfacial reactions: Chemical reactions at the interface between the matrix and the reinforcement can occur during processing. These reactions can lead to the formation of brittle intermetallic compounds, which can degrade the mechanical properties. (Jones, 2003).
- Porosity: Gas entrapment during mixing and solidification can lead to porosity in the casting, which can reduce the mechanical properties and increase the risk of corrosion. (Stefanescu, 2008).

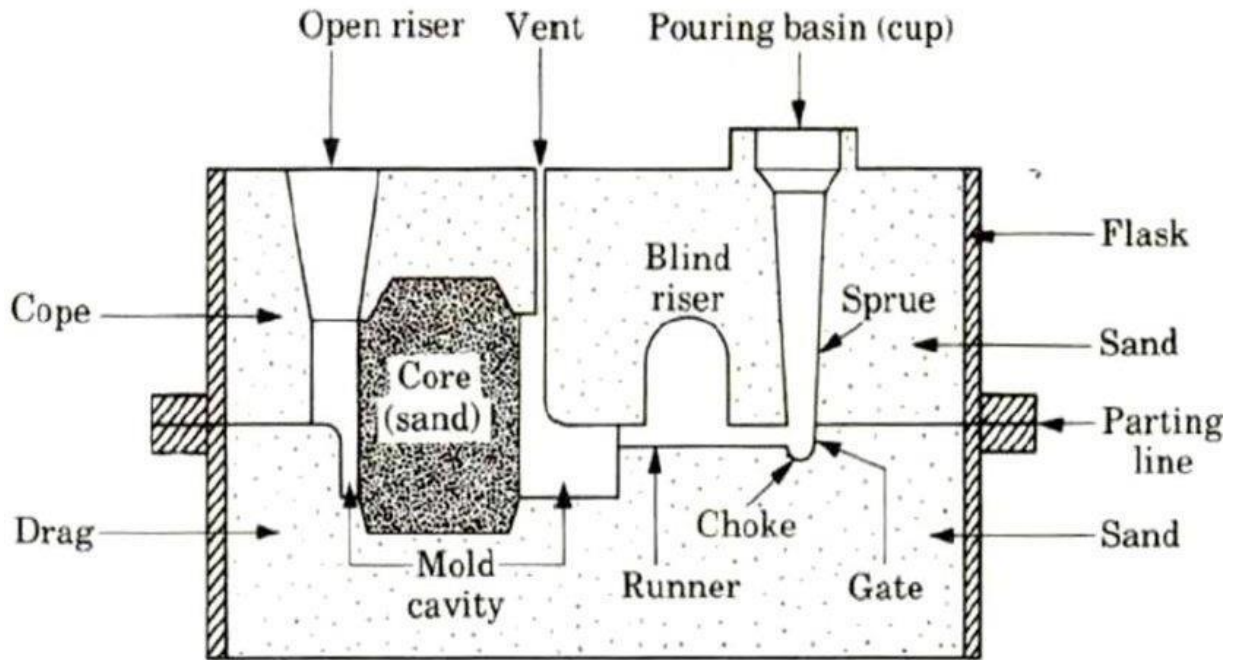


Figure 2.2: Sand casting process (Mohammed, 2019)

### 2.9.3. Heat flow in aluminum matrix composites (AMCs) and its relevance

Understanding heat flow is essential in materials processing and performance, especially for composites. Heat transfer occurs through three primary mechanisms:

- **Conduction:** Heat transfer through direct contact between molecules due to a temperature gradient. In AMCs, conduction occurs within the aluminum matrix, the reinforcement particles (ESP), and at the interface between them.
- **Convection:** Heat transfer through the movement of fluids (liquids or gases). During AMC processing (e.g., liquid metallurgy), convection can occur in the molten aluminum. In service, convection can occur due to air or fluid flow around the composite component.
- **Radiation:** Heat transfer through electromagnetic waves. Radiation becomes significant at high temperatures, such as those encountered during some AMC processing techniques or high-temperature applications. (Incropera et al., 2007)

### **(a) Heat Flow during AMC processing**

Heat flow plays a critical role in various AMC processing techniques:

- **Liquid metallurgy:** During casting or melt infiltration, heat transfer governs the solidification rate of the aluminum matrix. The cooling rate influences the microstructure of the matrix, the distribution of the reinforcement particles, and the formation of interfacial reactions (Stefanescu, 2008). Non-uniform cooling can lead to residual stresses and defects in the composite. The thermal conductivity of the reinforcement (eggshell powder in this case) relative to the matrix influences the overall heat dissipation during cooling.
- **Powder metallurgy:** In powder metallurgy, heat is applied during sintering to consolidate the powder compact. Heat flow during sintering affects the densification process, the bonding between particles, and the final properties of the composite. (German, 1990)
- **Solid-state processing:** In techniques like friction stir processing (FSP), heat generation due to friction and plastic deformation is crucial for achieving bonding and microstructural refinement. The heat input needs to be carefully controlled to avoid excessive temperatures that could lead to undesirable reactions or degradation of the reinforcement (Mishra et al., 2014).

### **(b) Influence of eggshell powder (ESP) on Heat Flow in AMCs**

The incorporation of ESP into the aluminum matrix introduces complexities in heat flow due to the different thermal properties of ESP compared to aluminum:

- **Thermal Conductivity:**  $\text{CaCO}_3$ , the primary component of ESP, has a lower thermal conductivity than aluminum. This means that the addition of ESP can reduce the overall thermal conductivity of the composite. The volume fraction and distribution of ESP significantly influence this effect.
- **Thermal expansion:** The coefficient of thermal expansion (CTE) of  $\text{CaCO}_3$  is also different from that of aluminum. This mismatch in CTE can lead to thermal stresses at the interface between the matrix and the reinforcement during temperature changes. These thermal stresses can influence the mechanical behavior and integrity of the composite. (Callister, 2007)
- **Thermal stability of  $\text{CaCO}_3$ :** As mentioned earlier,  $\text{CaCO}_3$  can decompose at high temperatures, typically above  $800^\circ\text{C}$ , releasing  $\text{CO}_2$  and forming  $\text{CaO}$ . This

decomposition can affect the thermal properties and microstructure of the composite. Therefore, processing temperatures need to be carefully controlled to avoid this decomposition, especially in liquid metallurgy techniques.

### **(c) Modeling heat flow in AMCs**

Computational modeling can be a valuable tool for understanding and predicting heat flow in AMCs. Techniques like finite element analysis (FEA) can be used to simulate heat transfer during processing and in service. These simulations can help optimize processing parameters, predict temperature distributions, and assess the impact of reinforcement distribution and thermal properties on the overall thermal behavior of the composite.

### **(d) Relevance to mechanical and tribological properties**

Heat flow considerations are directly linked to the mechanical and tribological properties of AMCs:

- Residual stresses: Non-uniform cooling during processing can lead to residual stresses in the composite, which can affect its strength, fatigue behavior, and dimensional stability.
- Interfacial bonding: The temperature at the interface between the matrix and the reinforcement during processing influences the quality of the interfacial bonding. Proper heat flow management is essential for achieving strong bonding.
- Wear behavior: During wear testing or service, frictional heating can occur at the contact surfaces. The thermal conductivity of the composite influences the dissipation of this heat, which can affect the wear rate and wear mechanisms. This is especially relevant in applications where ESP has shown promise in improving wear resistance.

## **2.9.4 Previous works and research gap**

The ever-increasing demand for low cost reinforcement stimulated the interest towards production and utilization of using by-products from industry as reinforcement since they are readily available or are naturally renewable at affordable cost. (Aigbodion, 2007) have used Kankara clay (alumino-silicate) in reinforcing Al–Si alloy, (Bienia et al., 2003) used fly ash in the reinforcement of aluminum matrix, (Naresh, 2006), worked on the development and characterization of metal matrix composite using red mud an industrial waste for wear resistant

applications, they all reported good dispersion and recovery of the particles in the composites castings. Previous studies have proved that Chicken eggshell (ES) is an aviculture byproduct that has been listed worldwide as one of the worst environmental problems, especially in those countries where the egg product industry is well developed. In the U.S. alone, about 150,000 tons of this material is disposed of in landfills (Shuhadah et al., 2008). ES contains about 95% calcium carbonate in the form of calcite and 5% organic materials such as type X collagen, sulfated polysaccharides, and other proteins (Shuhadah et al., 2008; Patricio et al., 2007; Hussein et al., 2011). Although there have been several attempts to use eggshell components for different applications, its chemical composition and availability makes eggshell a potential source of filler in polymer composites (Shuhadah et al., 2008; Patricio et al., 2007; Hussein et al., 2011). Report has shown, among other characteristics, that ES has a relatively lower density compared to the mineral calcium carbonate. Egg shell is a biomaterial containing 95% by weight of calcium carbonate in the form of calcite and 5% by weight of organic materials, such as ( $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ , S, Cl, P, and  $\text{Cr}_2\text{O}_3$ , MnO) (Hussein al., 2011). The generalized egg shell structure, which varies widely among species, is a protein lined with mineral crystals, usually of a calcium compound such as calcium carbonate. These characteristics qualify ES as a good candidate for bulk quantity,

inexpensive, lightweight and low load-bearing composite applications, such as the automotive industry, trucks, homes, offices, and factories. (Patricio et al., 2007), studied ES, a new bio-filler for poly-propylene composites. The work proved that ES composite showed lower modulus of elasticity (E) values than talc composites, talc filler could be replaced by up to 75% with ES while maintaining a similar stiffness and E compared to the talc composites. (Hussein et al., 2011) studied the water absorption and mechanical properties of high – density polyethylene/ ES

composite, It was found that the addition of egg shell powder to the polymer leads to decrease in the tensile strength, modulus of elasticity and shore-D hardness. On other hand it increases the % elongation at break and impact strength. Water absorption of the composites behavior as function of days was also investigated, and it increases by increasing exposure time for the same filler content, while the absorbed amount of water increases, by increasing the wt.% of ES constant exposure time. Further research has explored combining eggshells with other reinforcing materials, such as silicon carbide, in aluminum matrices. A study published in IRJET investigated the effects of waste eggshells and silicon carbide addition in the synthesis of aluminum alloy 6061 hybrid green metal matrix composite, observing improvements in hardness and tensile strength with the addition of carbonized eggshell particles. Comparing the

effects of uncarbonized and carbonized eggshells on a magnesium-alumina matrix, a study in the International Journal of Innovative Science and Research Technology found that carbonized particles led to better tensile strength. A review article on Research Gate provides a comprehensive overview of how eggshell particles influence the mechanical properties of aluminum matrix composites, discussing various factors like particle size, distribution, and processing techniques. A review article on Research Gate provides a comprehensive overview of how eggshell particles influence the mechanical properties of aluminum matrix composites, discussing various factors like particle size, distribution, and processing techniques. These studies indicate the potential of eggshell powder as a reinforcement material in MMCs, particularly when carbonized.

While limited research exists specifically on aluminum-eggshell composites for kitchenware, studies on aluminum composites reinforced with other ceramic particles, including  $\text{CaCO}_3$ , provide valuable insights. Studies have also explored the effect of  $\text{CaCO}_3$  on the mechanical properties of aluminum composites (e.g., Usca et al., 2016, who investigated Al-Si alloy reinforced with  $\text{CaCO}_3$ ). These studies generally show that adding  $\text{CaCO}_3$  can increase hardness but may sometimes decrease ductility. There's a research gap regarding the specific use of eggshell powder (with its inherent variations in composition and structure compared to pure  $\text{CaCO}_3$ ) in aluminum for kitchenware applications, especially concerning thermal insulation.

### **2.9.5. Applications of the work**

#### **(a) Enhanced kitchenware**

One promising application of aluminum matrix composites reinforced with eggshell powder lies in the enhancement of kitchenware. This application leverages the potential improvements in mechanical and thermal properties offered by the composite material to create more durable, efficient, and potentially more sustainable kitchen utensils and cookware.

- Durability and wear resistance:

A primary advantage of incorporating eggshell powder into an aluminum matrix is the potential for increased durability and wear resistance. Eggshell is primarily composed of calcium carbonate ( $\text{CaCO}_3$ ), a hard ceramic material (Balázs & Weber, 2016). The inclusion of these

hard particles within the softer aluminum matrix can significantly enhance the composite's resistance to scratching, abrasion, and deformation during regular use. This is consistent with general principles of particulate reinforcement in MMCs, where the hard reinforcing phase carries a significant portion of the applied load, leading to improved hardness and wear resistance (Dwivedi, 2014). Studies on similar particulate-reinforced aluminum composites have demonstrated improvements in hardness and wear resistance with the addition of ceramic particles (Hashim et al., 1999). Specifically, research focusing on eggshell reinforcement has shown promising results in improving the mechanical properties of aluminum composites. For instance, studies have shown that incorporating carbonized eggshell particles can enhance the hardness, tensile strength, and fatigue strength of aluminum. This enhanced durability translates to kitchenware that is less prone to damage from everyday use, such as cleaning with abrasive sponges or contact with metal utensils, thereby extending its lifespan.

- Thermal properties and cooking performance:

Aluminum is already a widely used material in cookware due to its excellent thermal conductivity, which allows for rapid and even heat distribution. However, the addition of eggshell powder could potentially influence the thermal behavior of the composite. While  $\text{CaCO}_3$  has a lower thermal conductivity than aluminum (Callister & Rethwisch, 2018). The overall effect on the composite's thermal conductivity will depend on the volume fraction and distribution of the eggshell particles. Further investigation is needed to determine the precise impact of eggshell addition on the thermal conductivity of the composite and its implications for cooking performance. It's possible that the fine distribution of eggshell particles could promote more even heat distribution, minimizing hot spots and ensuring more consistent cooking. This could be particularly beneficial for cooking delicate foods that are prone to burning or uneven cooking.

- Potential for non-stick properties:

Another interesting area of potential benefit is the development of non-stick properties. While further research is required to confirm this, the presence of calcium carbonate on the surface of the composite could potentially influence its surface energy and create a naturally non-stick surface. This could reduce the reliance on synthetic non-stick coatings, which can degrade over time and release potentially harmful chemicals. This aspect aligns with the growing consumer demand for healthier and more environmentally friendly cookware.

- Sustainability and cost-effectiveness:

The use of eggshell powder as a reinforcement material offers a significant advantage in terms of sustainability. Eggshells are a readily available waste product from the food industry. Utilizing them in the production of kitchenware provides a valuable application for this waste material, reducing landfill waste and promoting a circular economy. Furthermore, eggshells are a low-cost material, which could contribute to the overall cost-effectiveness of producing aluminum-eggshell composite kitchenware. This aligns with the increasing focus on sustainable manufacturing practices and the development of eco-friendly materials.

- Specific Kitchenware Applications:

The aluminum-eggshell composite could be used in a variety of kitchenware applications, including:

1. Pots and pans: The enhanced durability and potential for even heat distribution make this composite suitable for pots and pans used for various cooking methods.
2. Utensils: Spoons, spatulas, and other utensils made from this composite would be more resistant to wear and tear.
3. Baking Sheets and Molds: The potential non-stick properties could be particularly beneficial for baking applications.

### **(b). Beyond kitchenware**

While enhanced kitchenware represents a compelling application for aluminum-eggshell composites, the potential extends to various other sectors, leveraging the unique combination of properties offered by this material.

1. Automotive applications:

The automotive industry is constantly seeking lightweight materials to improve fuel efficiency and reduce emissions. Aluminum alloys are already widely used in automotive components due to their low density. Reinforcing aluminum with eggshell powder can further enhance its specific strength (strength-to-weight ratio) and stiffness, making it a potential candidate for various non-critical automotive components.

- Non-critical structural components:

Components such as interior panels, dashboard supports, and certain brackets could benefit from the enhanced stiffness and reduced weight offered by the composite. While not subjected to high stress levels, these components contribute to the overall weight of the vehicle.

Replacing conventional materials with lighter composites can contribute to fuel economy. (Dieter, 2017).

- Brake rotors (potential):

While more research is needed, the enhanced wear resistance imparted by the hard  $\text{CaCO}_3$  particles could potentially make the composite suitable for brake rotors. However, thermal conductivity and high-temperature performance under braking conditions would need careful investigation. Studies on similar particulate-reinforced aluminum composites have explored their potential in braking applications, highlighting the importance of optimizing particle distribution and interfacial bonding for improved performance under thermal cycling and wear (Ibrahim, et al., 1991).

- Recycled aluminum and eggshell composites:

Research has explored the use of recycled aluminum in conjunction with eggshells to create composites for automotive applications. Studies have evaluated the mechanical and wear resistance properties of these composites, demonstrating their potential for use in specific automotive components

## 2. Construction and building materials:

The construction industry also benefits from lightweight and durable materials. Aluminum-eggshell composites could find applications in various building components:

- Non-load-bearing panels and cladding: The composite's enhanced stiffness and reduced weight can be advantageous in non-load-bearing applications like wall panels, cladding, and roofing elements. These applications prioritize lightweight materials for ease of handling and installation. (Neville, 2011).
- Decorative elements: The composite's potential for near-net-shape casting allows for the creation of intricate decorative elements for building facades or interiors. This could offer architects and designers new possibilities for incorporating sustainable materials into building designs.
- Potential for concrete reinforcement: While requiring further investigation, the composite could potentially be explored as a reinforcement material in concrete. The  $\text{CaCO}_3$  from the eggshells could interact with the cement hydration process, potentially influencing the concrete's properties. (Mehta & Monteiro, 2014).

### 3. Biomedical Applications:

Calcium carbonate, the primary constituent of eggshells, is known for its biocompatibility (Hench,1998). This opens up potential applications in the biomedical field, although extensive research is required to ensure safety and efficacy:

- Drug delivery systems: The porous nature of eggshell-derived materials has been investigated for drug delivery applications. Incorporating these materials into an aluminum matrix could potentially create novel drug delivery platforms. (Balázsi & Weber, 2016).
- Bone implants: While highly speculative at this stage, the composite's potential for Osseo integration (bonding with bone tissue) could be explored for bone implant applications. However, significant research is needed to optimize the composite's composition and surface properties for biocompatibility and mechanical compatibility with bone. (Ratner et al., 2012).

#### **(c) Sustainability**

A key driving force behind the investigation of aluminum-eggshell composites is the inherent sustainability aspect of utilizing a readily available waste material.

##### 1. Waste valorization and circular economy:

One of the most significant sustainability benefits of this project is the valorization of eggshell waste. Globally, vast quantities of eggshells are generated as a byproduct of the food processing industry, poultry farms, and households. These shells often end up in landfills, contributing to waste management challenges and potential environmental issues. (Balázsi & Weber, 2016). By utilizing eggshells as a reinforcement material in aluminum composites, this project directly addresses the issue of waste disposal by transforming a waste product into a valuable resource. This aligns with the principles of a circular economy, which aims to minimize waste and maximize resource utilization by keeping materials in use for as long as possible. (Geissdoerfer, et al.,2017).

##### 2. Reduced reliance on virgin materials:

The incorporation of eggshells as a reinforcement can potentially reduce the reliance on virgin materials in the production of certain components. While aluminum itself is highly recyclable, the extraction and processing of virgin aluminum are energy-intensive processes with

associated environmental impacts. (Das, 2010). By partially replacing aluminum with eggshell powder, the demand for virgin aluminum can be reduced, contributing to resource conservation and minimizing the environmental footprint associated with aluminum production. This is particularly important given the increasing global demand for aluminum and the associated environmental concerns.

### 3. Lower environmental impact compared to synthetic reinforcements:

Many conventional reinforcing materials used in composites, such as synthetic fibers or ceramic powders, require energy-intensive manufacturing processes with potentially significant environmental impacts. In contrast, eggshells are a natural and readily available material that requires minimal processing before incorporation into the composite. This translates to a lower embodied energy and reduced environmental burden compared to synthetic reinforcements. (Ashby, 2009). The simple processing typically involves cleaning, drying, and grinding the eggshells, which consumes significantly less energy than the production of synthetic materials.

### 4. Potential for biodegradability (end-of-life considerations):

While the composite itself is not fully biodegradable due to the aluminum matrix, the presence of the natural eggshell component introduces a degree of biodegradability. When the composite reaches the end of its life cycle, the eggshell component can decompose under appropriate conditions, reducing the overall environmental impact compared to composites made entirely from non-biodegradable materials. However, the aluminum matrix would still need to be recycled through conventional aluminum recycling processes. (Hopewell, et al.,2009).

### 4. Cost-effectiveness and accessibility:

Eggshells are a low-cost and readily available waste material, which contributes to the cost-effectiveness of producing the aluminum-eggshell composite. This can make the composite more accessible for various applications, particularly in developing countries where cost considerations are often a major factor. This accessibility can further promote the adoption of sustainable materials and contribute to broader environmental benefits.



Figure 2.3: Aluminum utensils (Institute of Industrial Development, 2020)

## CHAPTER 3

### MATERIALS AND METHODS

#### 3.1 Materials

The materials used for this project work are Aluminum ingot (Matrix) scrap, Eggshells, plumbago, mold seal, Molding Sand, Spoon prototype, and 15.7mm diameter rod

1. Aluminum ingot (Matrix) scrap: The matrix material used in this study was commercially available aluminum ingot scrap. This material was chosen for its cost-effectiveness and availability, making it suitable for exploratory research. The chemical composition of aluminum alloy is shown in table 1
2. Reinforcement (Eggshell Powder): Fresh chicken eggshells were collected from the local market at Ekosodin, Benin City. The use of fresh eggshells minimizes potential degradation of the calcium carbonate.
3. Plumbago: Plumbago is a naturally occurring form of graphite with a flaky or powdery texture. In casting, plumbago acts as a parting agent. This means it's dusted onto the pattern (the spoon prototype) before the molding sand is packed around it. This created a thin layer that prevented the sand from sticking to the pattern, allowing for a clean separation and a smooth mold cavity. A clean separation ensures that the details of your spoon prototype are accurately transferred to the mold, resulting in a high-quality casting.
4. Mold seal: A mold seal is a material used to seal the joint between the two halves of a mold (the cope and drag). It can be made of clay, wax, or other pliable substances. The mold seal prevents molten metal from leaking out of the mold cavity during pouring. A good seal ensures that all the molten metal fills the mold cavity completely, resulting in a complete and accurate casting.
5. Mold sand: Mold sand is a special type of sand used for creating molds in metal casting. It's typically a mixture of silica sand, clay, and moisture. The mold sand was packed around the spoon prototype to create the mold cavity. It needed to be able to withstand the high temperatures of molten aluminum and hold its shape during pouring. The properties of the mold sand (grain size, clay content, moisture content) affect the surface finish of the casting, the mold's strength, and its ability to vent gases during pouring.

6. Spoon Prototype: This is the physical model of the spoon that was used to create the mold cavity. It can be made of wood, metal, plastic, or other materials. The spoon prototype determines the shape and size of the final cast spoon. The prototype needs to be accurately shaped and finished to ensure that the cast spoon meets your design requirements. It also needs to be durable enough to withstand the molding process.

### **3.2 Equipment**

The equipment used in this project work includes

1. High-performance heavy duty blender: Used for grinding of the eggshells into smaller fragments before further processing. This reduces the particle size
2. Set of sieves: Used for separating the ground eggshell particles into different size fractions. This ensures a consistent particle size distribution for the reinforcement, which is crucial for achieving uniform properties in the composite. Different mesh sizes allow for precise control over the particle size used in the composite.
3. Vernier caliper: A precision measuring instrument used to measure the dimensions of the cast composite samples before and after testing. Accurate dimensional measurements are essential for calculating properties like cross-sectional area for tensile testing and ensuring consistency in sample geometry.
4. Weight meter (balance/scale): Used for accurately measuring the weight of the, eggshell powder. Precise weight measurements are necessary for calculating the volume fraction of the reinforcement in the composite, which is a key parameter affecting its properties.
5. Crucible furnace: Used for melting the aluminum alloy. The crucible holds the aluminum during the melting process, and the furnace provides the high temperatures necessary for melting.
6. Blower for the furnace: Provides forced air to the furnace, enhancing combustion and achieving the high temperatures required for melting aluminum. It ensures efficient and controlled heating of the furnace.

7. Tongs: Used for handling hot crucibles, molds, and samples during the casting process. They are essential for safety when working with high temperatures.
8. Metal stirrer: Used to mix the eggshell powder into the molten aluminum during the casting process. Effective stirring is crucial for achieving a uniform distribution of the reinforcement particles in the matrix, preventing settling or agglomeration.

### **3.3. Method**

#### **3.3.1. Eggshells processing**

The eggshells were processed as follows:

- Cleaning: The eggshells were thoroughly washed with tap water to remove any residual yolk, membrane, and other contaminants. This step is essential to prevent contamination of the composite and ensure proper bonding between the matrix and reinforcement.
- Drying: The cleaned eggshells were dried in the sun for 24 hours to remove moisture completely. Complete drying prevents porosity formation during the casting process due to vaporization of trapped water.
- Grinding: The dried eggshells were then ground into a fine powder using a high performance heavy duty blender for 10 minutes at 2500 rpm. The process reduces the particle size to the desired range, which affects the properties of the final composite.
- Sieving: The ground powder was sieved using a set of standard sieves arranged in descending order of 1800 $\mu\text{m}$ , 600 $\mu\text{m}$ , 425 $\mu\text{m}$ , and 300 $\mu\text{m}$ , to obtain a specific particle size range. Controlling the particle size range is crucial as it influences particle distribution, surface area, and interfacial bonding in the composite. For example, smaller particles provide a larger surface area for bonding but can also lead to agglomeration.

### 3.3.2 Determination of sample (prototype) weight

Volume of cylindrical rod( $v$ ) =  $\pi r^2 h$

Where:

Diameter( $d$ ) = 15.7mm = 1.57cm

Radius ( $r$ ) = Diameter ( $d$ )/2 = 1.57cm/2 = 0.785cm.

Height ( $h$ ) = 21.664cm

Volume of the cylindrical rod( $v$ ) =  $\pi \times 0.785^2 \times 21.664 = 41.94\text{cm}^3$

Mass of rod in grams = Density of rod  $\times$  volume of rod =  $2.7 \times 41.94 = 13.238\text{g}$

### 3.3.3 The aluminum-eggshell composites were fabricated using the sand mold casting method.

The following steps were involved:

- Melting: The aluminum ingots were melted in a crucible furnace at a temperature of  $670^\circ\text{C}$ , which was maintained slightly above the melting point of the aluminum alloy. The type of furnace affects the heating rate and control of the melting process.
- Mixing: The eggshell powder was added to the molten aluminum while stirring vigorously for 3 minutes. The stirring was continued to ensure uniform distribution of the eggshell particles in the molten aluminum. The volume fraction of eggshell powder used in this study were 10 wt.%, 13 wt.%, and 7 wt.%. The stirring process is crucial for achieving a homogenous distribution of the reinforcement particles in the matrix. The type of stirrer and stirring parameters affect the mixing efficiency and particle distribution.
- Casting: The molten composite mixture was then poured into a sand mold of desired dimensions.
- Solidification: The molten composite was allowed to solidify in the mold under ambient conditions. The solidification rate affects the microstructure and properties of the casting.

### **3.3.4 Characterization techniques**

The fabricated composites were characterized using the following techniques:

1. Microstructural analysis
2. Hardness testing
3. Tensile testing
4. Thermal analysis

### **3.3.5 Microstructural analysis**

The microstructure of the composites was examined using Scanning Electron Microscopy (SEM) in the Material Science and Engineering Department, Obafemi Awolowo University. The samples were prepared by standard metallographic procedures, including grinding, polishing, and etching. Microstructural analysis reveals the distribution of the reinforcement particles, the presence of any porosity or defects, and the nature of the interface between the matrix and reinforcement. Metallography is the study of the crystalline structure of metals and alloys and the relationship of this structure to the physical properties of metals. Microscopic examination of suitably prepared specimens makes it possible for the determination of size, structure, and orientation of the metal crystals. By means of such examinations, metallurgists can frequently identify a metal or alloy and check on the effectiveness of heat treatments for hardening or annealing. Metal specimens for metallographic examination are usually highly polished and then etched with etchants; this treatment brings out the grain structure by attacking the boundaries between the grains or by attacking one of the constituents of an alloy. Then, metals are examined under high magnification of a low power microscope, a thin, electron-transparent replica or cast of the etched surface can be made, because bulk metals do not transmit an electron beam. Alternatively, an extremely thin specimen can be made; the microstructure that is observed is a projection of that contained within the thin specimen.

Scanning electron microscopy/energy dispersive X-ray spectrometry (SEM/EDS) is a widely applied elemental microanalysis method capable of identifying and quantifying all elements in the periodic table except H, He, and Li. By following the “*k*-ratio” (unknown/standard) measurement protocol development for electron-excited wavelength dispersive spectrometry (WDS), SEM/EDS can achieve accuracy and precision equivalent to WDS and at substantially lower electron dose, even when severe X-ray peak overlaps occur, provided sufficient counts are recorded. Achieving this level of performance is now much more practical with the advent of the high-throughput silicon drift detector energy dispersive X-ray spectrometer (SDD-EDS).

However, three measurement issues continue to diminish the impact of SEM/EDS: (1) In the qualitative analysis (i.e., element identification) that must precede quantitative analysis, at least some current and many legacy software systems are vulnerable to occasional misidentification of major constituent peaks, with the frequency of misidentifications rising significantly for minor and trace constituents. The use of standard less analysis, which is subject to much broader systematic errors, leads to quantitative results that, while useful, do not have sufficient accuracy to solve critical problems, e.g. determining the formula of a compound. EDS spectrometers have such a large volume of acceptance that apparently credible spectra can be obtained from specimens with complex topography that introduce uncontrolled geometric factors that modify X-ray generation and propagation, resulting in very large systematic errors, often a factor of ten or more (Newbury, et al., 2013)

**(a). Metallographic examination:** Visual examination is good enough for macro-examination but on the micro-level, there is the need for aided media. The samples under consideration were prepared for micro-examination.

**i). Sample preparation:** This is the primary stage involved in metallographic examination processes. These include grinding, polishing, etching before final examination under the metallurgical microscope.

**ii). Grinding:** This operation aims at producing a perfectly flat and smooth surface. Silicon carbide papers of different grades placed on the grinding machine was used in the order of 220,320,400 and 600, i.e. from coarse grade to fine grade. The grinding process was done under running water to wash away the grits and also to avoid overheating. The samples were turned through 900 while changing from one grit size to another in the Materials Science and Engineering laboratory at OAU Ile – Ife. This is to neutralize the scratching effect of the previous grinding of the former grit size.

**iii). Polishing:** A universal polishing machine was employed. A polishing cloth was placed on the polisher for the initial polishing swamped with solution of one micron of silicon carbide solution, then, followed by the final polishing stage with selves cloth swamped with solution of 0.5 $\mu$ m Silicon carbide until a mirror-like surface is attainable. It is then washed and dried.

**iv). Etching:** This is done to reveal the microstructure of the polished surface. Etching is the selective attack on the grain boundaries being a region of high energy and dislocation density. The mirror-like surface was etched in 2% NITAL (2% Nitric acid and 98% of Ethyl Alcohol) while Sodium hydroxide is for non-ferrous materials Again, it is washed, dried and later viewed

under the metallurgical microscope (Accuscope microscope with camera (Serial no 0524011, Maker: Princeton, US) with magnification 400x and 800x respectively.



Figure 3.1: SEM-EDS (JEOL JSM-7600F)

### **3.3.6 Hardness test**

The hardness testing was carried out in the material science and engineering department, Obafemi Awolowo University. Hardness testing measures the material's resistance to indentation. Hardness is the ability of a material to resist deformation, indentation, or penetration. In Brinell tests, as in Rockwell measurements, a hard, spherical indenter is forced into the surface of the metal to be tested. The diameter of the hardened steel (or tungsten carbide) indenter is 10.00 mm (0.394 in). Standard loads range between 500 and 3000kg in 500-kg increments; during a test, the load is maintained constant for a specified time (between 10 and 30s). Harder materials require greater applied loads. The Brinell hardness number, HB, is a function of both the magnitude of the load and the diameter of the resulting indentation. This diameter is measured with a special low-power microscope, utilizing a scale that is etched on the eyepiece. The measured diameter is then converted to the appropriate HB number using a chart; only one scale is employed with this technique. Semi-Automatic techniques for measuring Brinell hardness are available. These employ optical scanning systems consisting of a digital camera mounted on a flexible probe, which allows positioning of the camera over the indentation. Data from the camera are transferred to a computer that analyzes the indentation, determines its size, and then calculates the Brinell hardness number. For this technique, surface finish requirements are normally more stringent than for manual measurements. Maximum specimen thickness as well as indentation position (relative to specimen edges) and minimum indentation spacing requirements are the same as for Rockwell tests. In addition, a well-defined indentation is required; this necessitates a smooth flat surface in which the indentation is made.

#### **(a) Correlation between hardness and tensile strength**

Both tensile strength and hardness are indicators of a metal's resistance to plastic deformation. Consequently, they are roughly proportional, for tensile strength as a function of the HB for cast iron, steel, and brass. The same proportionality relationship does not hold for all metals as a rule of thumb for most steels, the HB and the tensile strength are related according to

$$TS \text{ (MPa)} = 3.45 \times HB$$

$$TS \text{ (psi)} = 500 \times HB$$

## (b) Procedure

Sample was provided and it was cut in order to get a specific length, after that the sample being cut was filed using a hand file in order to harden the surface of the sample, this was said to have been done properly provided one could see the image of the teeth of the surface of the filed sample. It was later grinded by using a grinding machine in which polishing the surface came after then. After which the sample was fixed into the torsionmeter where it was subjected to compression of load of 250 kg for about 15 seconds after which the indented diameter was measured by eye scope. We now used the conversion table to know the Brinell or hardness number of the material. **Brinell Hardness Number (BHN)** is calculated using the following formula:

$$HB = \frac{2P}{\pi D (D - \sqrt{D^2 - d^2})}$$

Where

- HB= Brinell Hardness Number (Kgf/mm<sup>2</sup>)
- P= Applied Load in Kilogram-force (Kgf)
- D= Diameter of Indenter (mm)
- d= Diameter of Indenter (mm)

When quoting a Brinell hardness number (BHN or more commonly HB), the conditions of the test used to obtain the number must be specified. The standard format for specifying tests can be seen in the example "HBW 10/3000". "HBW" means that a tungsten carbide (from the chemical symbol for tungsten) ball indenter was used, as opposed to "HBS", which means a hardened steel ball. The "10" is the ball diameter in millimeters. The "3000" is the force in kilograms' force.

The hardness may also be shown as XXX HB YYD2. The XXX is the force to apply (in kgf) on a material of type YY (5 for aluminum alloys, 10 for copper alloys, 30 for steels). Thus a typical steel hardness could be written: 250 HB 30D2. It could be a maximum or a minimum.

A convenient conversion fact is that of Brinell hardness to ultimate tensile strength. For quenched and tempered steel, the tensile strength (psi) is about 500 times the Brinell hardness number (provided the strength is not over 200,000



Figure 3.2: Monsanto tensometer (Type W)

### 3.3.7 Tensile test

Tensile testing, a fundamental technique, provides essential information about a material's response to tensile forces, including its strength, ductility, and stiffness. The samples of the composite were subjected to tensile test examining the influence of this reinforcement on the composite's mechanical properties using the Avery Dennison universal tensile testing machine with load range 0 to 600 KN in the mechanical workshop faculty of engineering, university of Benin.

**(a) Procedure**

- i). Specimen Preparation: Composite specimens were machined according to ASTM E8 standards to ensure consistent geometry and dimensions.
- ii). Mounting: Specimens were carefully mounted in the grips of the tensile testing machine, ensuring proper alignment to minimize bending stresses.
- iii). Loading: A tensile load was applied at a constant strain rate. Load and extension data were recorded continuously.
- iv). Data Analysis: The Ultimate Tensile Strength (UTS), and percentage elongation of samples were determined.

**(b) Elongation:** Elongation refers to the increase in length of the specimen after fracture, expressed as a percentage of the original length.

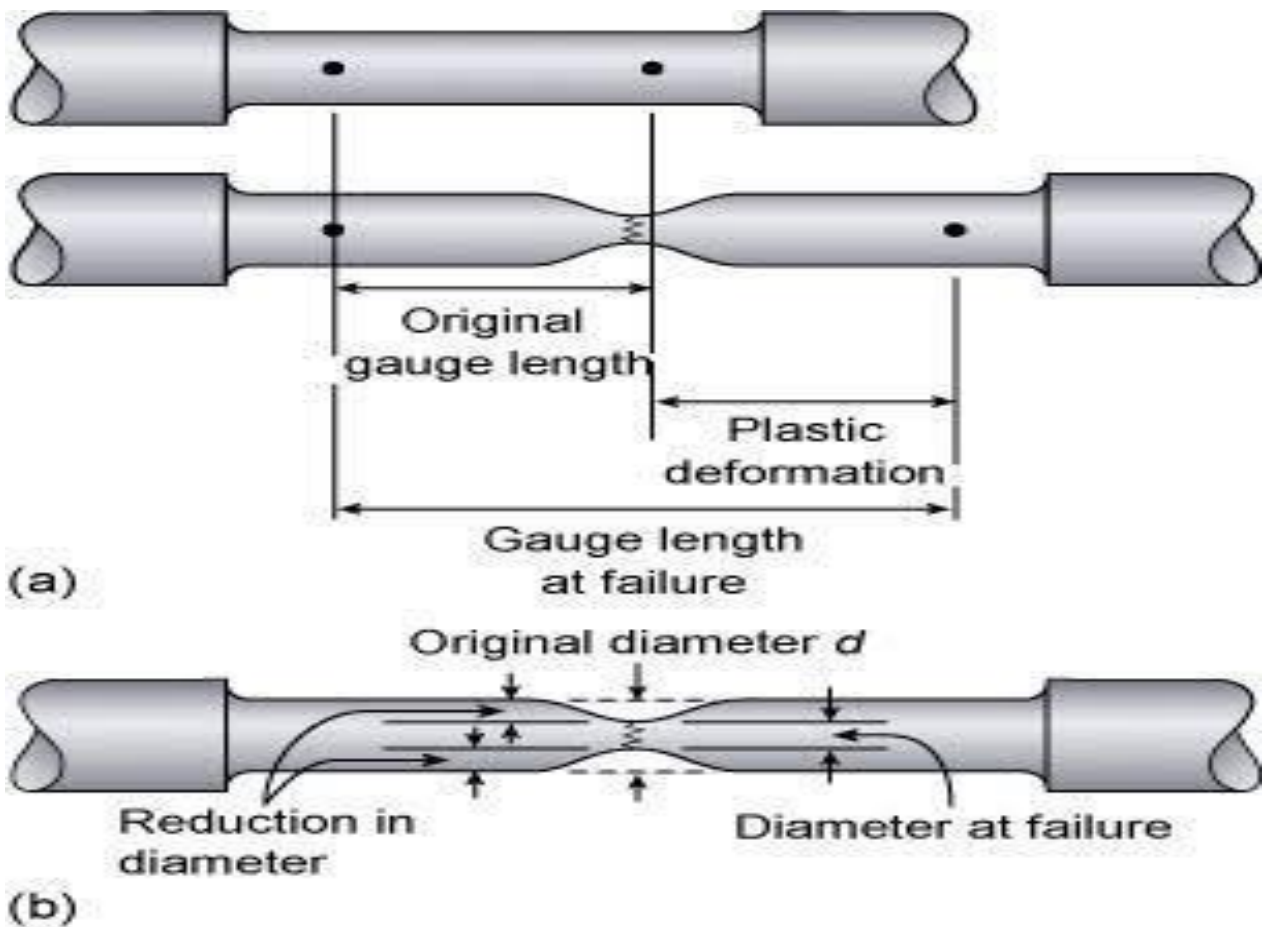


Figure 3.3: Representation of elongation(Wilkinson,2020)

### **(c) Percentage Elongation of samples**

The percentage elongation is calculated using the following formula:

$$\text{Percentage Elongation} = (\Delta L / L_0) \times 100\% = [(L_f - L_0) / L_0] \times 100\%$$

Where:

$\Delta L$  = Change in length ( $L_f - L_0$ )

$L_0$  = Original gauge length

$L_f$  = Final length after failure

### **(d) Ultimate tensile strength (UTS)**

Ultimate Tensile Strength (UTS), also known as tensile strength with a standard unit of Mega Pascal (MPa) which is the equivalence of  $\text{N/mm}^2$ , is the maximum stress a material can withstand before it starts to neck while being stretched or pulled.

Formula:

$$\text{UTS} = F/A$$

where:

UTS = Ultimate Tensile Strength

F = Force applied

A = Original cross-sectional area = cross-sectional area of cylindrical sample =  $\pi d^2/4$

d = 15.7mm

$$A = \pi (15.7^2) / 4 = 193.59\text{mm}^2$$

### **(e). Principles of elongation in cast alloys**

When a tensile load is applied to a cast alloy specimen, deformation occurs. Initially, this deformation is elastic, meaning the material returns to its original dimensions upon load

removal. Beyond the elastic limit, plastic deformation occurs, resulting in permanent changes in shape. Several factors influence elongation in cast alloys:

- **Composition:** The specific alloying elements and their proportions significantly impact strength and ductility.
- **Microstructure:** Grain size, shape, and phase distribution within the alloy matrix are critical. Cast alloys often exhibit a coarser microstructure than wrought alloys, which can affect ductility. Porosity, a common characteristic of some casting processes, can also reduce elongation.
- **Processing conditions:** Casting parameters, such as pouring temperature, cooling rate, and mold material, influence the resulting microstructure and, consequently, mechanical properties.
- **Reinforcement (Eggshell particles):** The addition of eggshell particles as reinforcement introduces a significant factor. Their size, shape, distribution, and the quality of their interfacial bond with the aluminum matrix directly affect the composite's overall mechanical behavior, including elongation. Typically, particulate reinforcement increases strength but may decrease ductility

#### **(f). Stress-strain curve**

The stress-strain curve is a fundamental representation of a material's mechanical behavior under tensile loading. It plots stress (force per unit area) against strain (change in length divided by original length). The curve reveals key characteristics:

- **Elastic Region:** The initial linear portion where stress is proportional to strain.
- **Yield Point:** The stress at which plastic deformation begins.
- **Ultimate Tensile Strength (UTS):** The maximum stress the material can withstand.
- **Fracture Point:** The point at which the material fails.

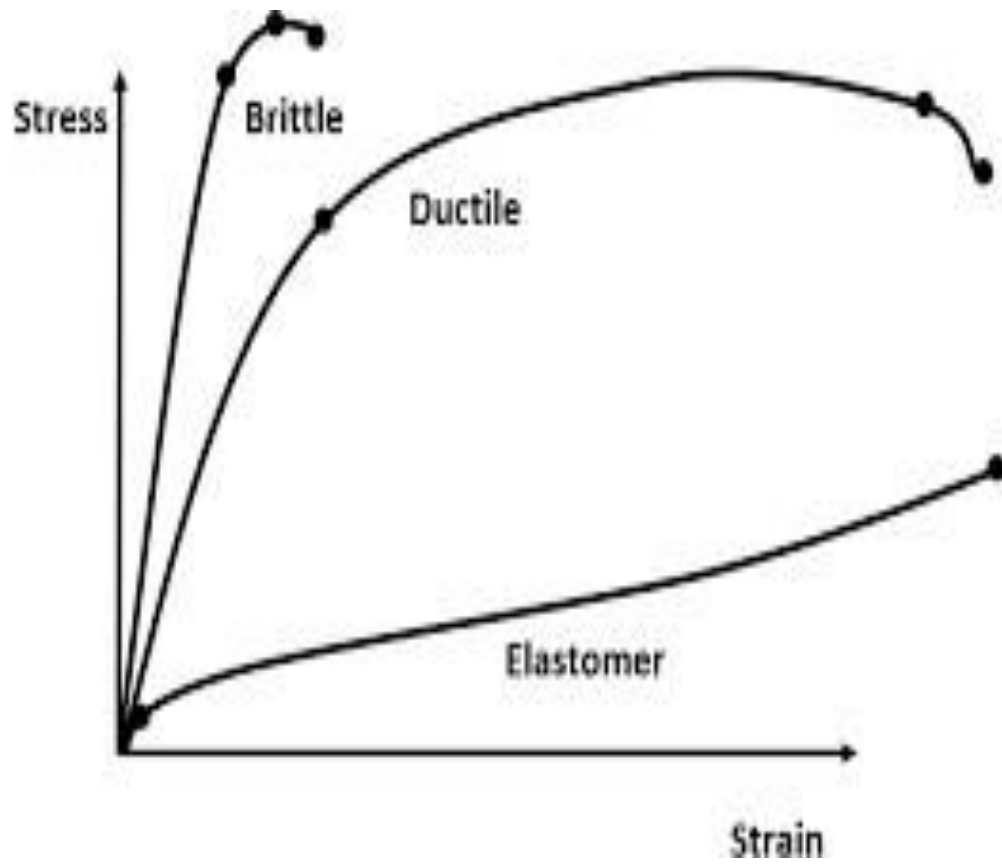


Figure 3.4: Stress and strain curve for brittle, ductile and elastomeric materials (Vendatu,2025)



Figure 3.5: Avery Dennison tensile testing machine

### **3.3.8 Thermal analysis**

The differential Scanning Calorimetry (DSC) was used to analyze the thermal behavior of the composite, particularly its thermal stability and any phase transitions that might occur. This can provide insights into the interaction between the aluminum and the eggshell powder at different temperatures. It is less directly relevant to thermal conductivity measurement but can provide supporting information.

#### **(a) Methodology and principle of DSC – differential scanning calorimetry**

This is a technique in which the difference in the amount of heat required to increase the temperature of a sample and reference are measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned.

The main application of DSC is in studying phase transitions, such as melting, glass transitions, or exothermic decompositions. These transitions involve energy changes or heat capacity changes that can be detected by DSC with great sensitivity.

#### **i). Detection of phase transitions**

The basic principle underlying this technique is that, when 2g of the sample undergoes a physical transformation such as phase transitions, more or less heat will need to flow to it than the reference to maintain both at the same temperature. Whether less or more heat must flow to the sample depends on whether the process is exothermic or endothermic. For example, as a solid sample melts to a liquid it will require more heat flowing to the sample to increase its temperature at the same rate as the reference. This is due to the absorption of heat by the sample as it undergoes the endothermic phase transition from solid to liquid. Likewise, as the sample undergoes exothermic processes (such as crystallization) less heat is required to raise the sample temperature. By observing the difference in heat flow between the sample and reference, differential scanning calorimeter are able to measure the amount of heat absorbed or released during such transitions. DSC may also be used to observe subtler phase changes; such as glass transitions. It is widely used in industrial settings as a quality control instrument due to its applicability in evaluating sample purity and for studying polymer curing.

## ii). DSC curves

The result of a DSC experiment is a curve of heat flux versus temperature or versus time. This curve can be used to calculate enthalpies of transitions. This is done by integrating the peak corresponding to a given transition. It can be shown that the enthalpy of transition can be expressed using the following equation:

$$\Delta H = KA$$

where  $\Delta H$  is the enthalpy of transition,  $K$  is the calorimetric constant, and  $A$  is the area under the curve. The calorimetric constant will vary from instrument to instrument, and can be determined by analyzing a well-characterized sample with known enthalpies of transition.

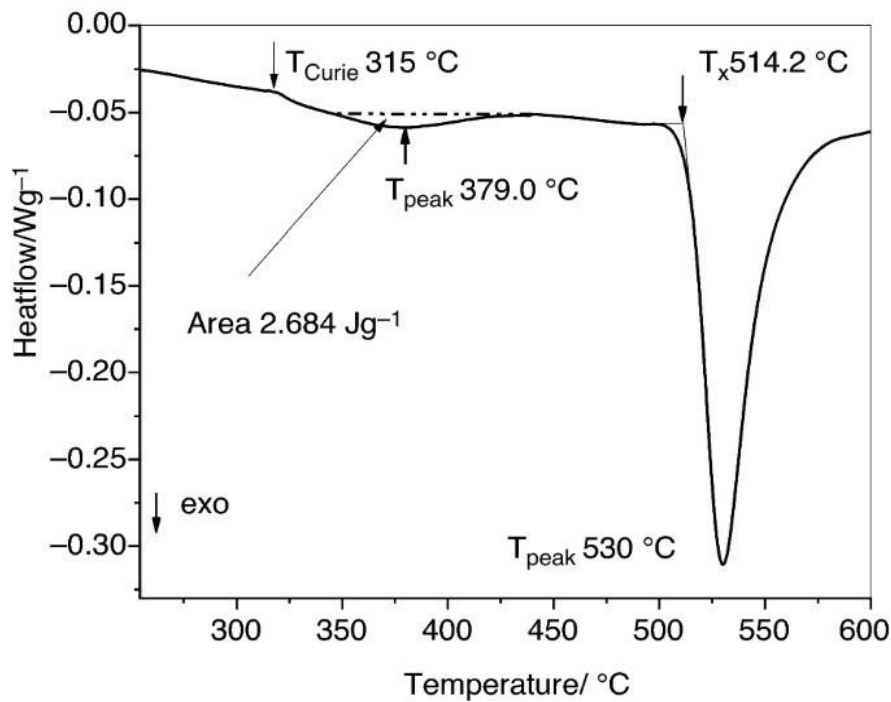


Figure 3.6: DSC curve obtained on heating samples from an amorphous ribbon of 1 Fe73Si16B7Nb3Cu alloy (heating rate: 8 °C min<sup>-1</sup>) (Ferenc,2021)

### iii). Applications of DSC

Differential scanning calorimetry can be used to measure a number of characteristic properties of a sample. Using this technique it is possible to observe fusion and crystallization events as well as glass transition temperatures  $T_g$ . DSC can also be used to study oxidation, as well as other chemical reaction. Glass may occur as the temperature of an amorphous solid is increased. These transitions appear as a step in the baseline of the recorded DSC signal. This is due to the sample undergoing a change in heat capacity; no formal phase change occurs. As the temperature increases, an amorphous solid will become less viscous. At some point the molecules may obtain enough freedom of motion to spontaneously arrange themselves into a crystalline form. This is known as the crystallization temperature ( $T_c$ ). This transition from amorphous solid to crystalline solid is an exothermic process, and results in a peak in the DSC signal. As the temperature increases the sample eventually reaches its melting temperature ( $T_m$ ). The melting process results in an endothermic peak in the DSC curve. The ability to determine transition temperatures and enthalpies makes DSC a valuable tool in producing phase diagrams for various chemical systems.

Differential scanning calorimetry can also be used to obtain valuable thermodynamics information about proteins. The thermodynamics analysis of proteins can reveal important information about the global structure of proteins, and protein/ligand interaction. For example, many mutations lower the stability of proteins, while ligand binding usually increases protein stability. Using DSC, this stability can be measured by obtaining Gibbs Free Energy values at any given temperature. This allows researchers to compare the free energy of unfolding between ligand-free protein and protein-ligand complex, or wild type and mutant proteins. DSC can also be used in studying protein/lipid interactions, nucleotides, drug-lipid interactions in studying protein denaturation using DSC, the thermal melt should be at least to some degree reversible, as the thermodynamics calculations rely on chemical equilibrium.



Figure 3.7: Differential scanning calorimetry machine (Shimadzu Dsc-60 Machine)

## CHAPTER 4

### RESULTS AND DISCUSSION

**Table 4.1: Chemical composition of aluminum alloy**

ELEMENT	Al	Mg	Zn	Si	Ni	Mn
%	85.65	3.42	7.97	0.34	2.39	0.23

**Table 4.2: Weight percentage of composite compositions**

Weight of pure aluminum alloy sample (PS) = 13.24g (100%)

Wt.% of eggshell = 0%

Material No.	Al		E.S	
	wt. %	( g )	wt. %.	( g )
Sample1	90	101.92	10	11.32
Sample 2	87	98.52	13	14.72
Sample 3	93	105.31	7	7.93

#### 4.1 Analysis of the scanning electron microscopy/energy dispersion spectroscopy

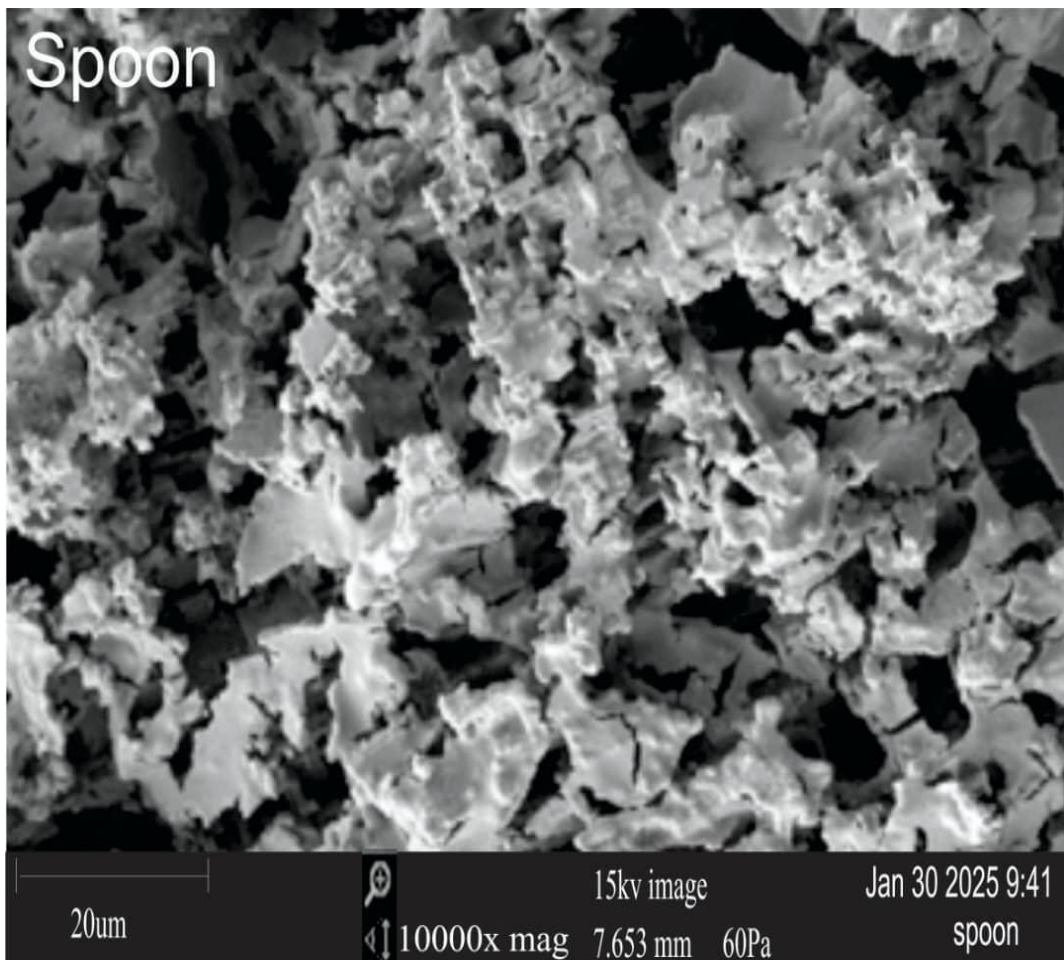


Plate 4.1: SEM result of aluminum spoon(prototype) with 1000x magnification

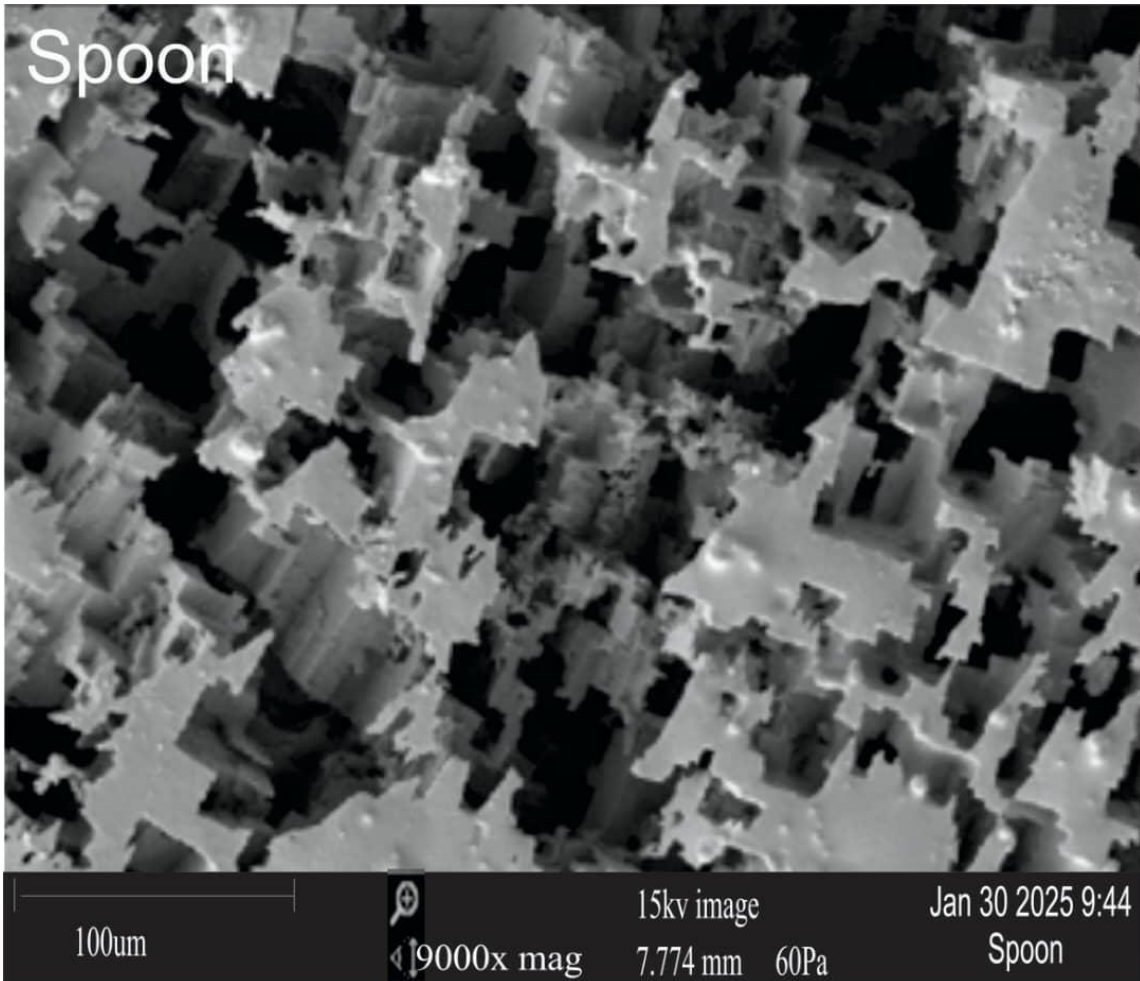


Plate 4.2: SEM result of aluminum spoon(prototype)with 9000x magnification

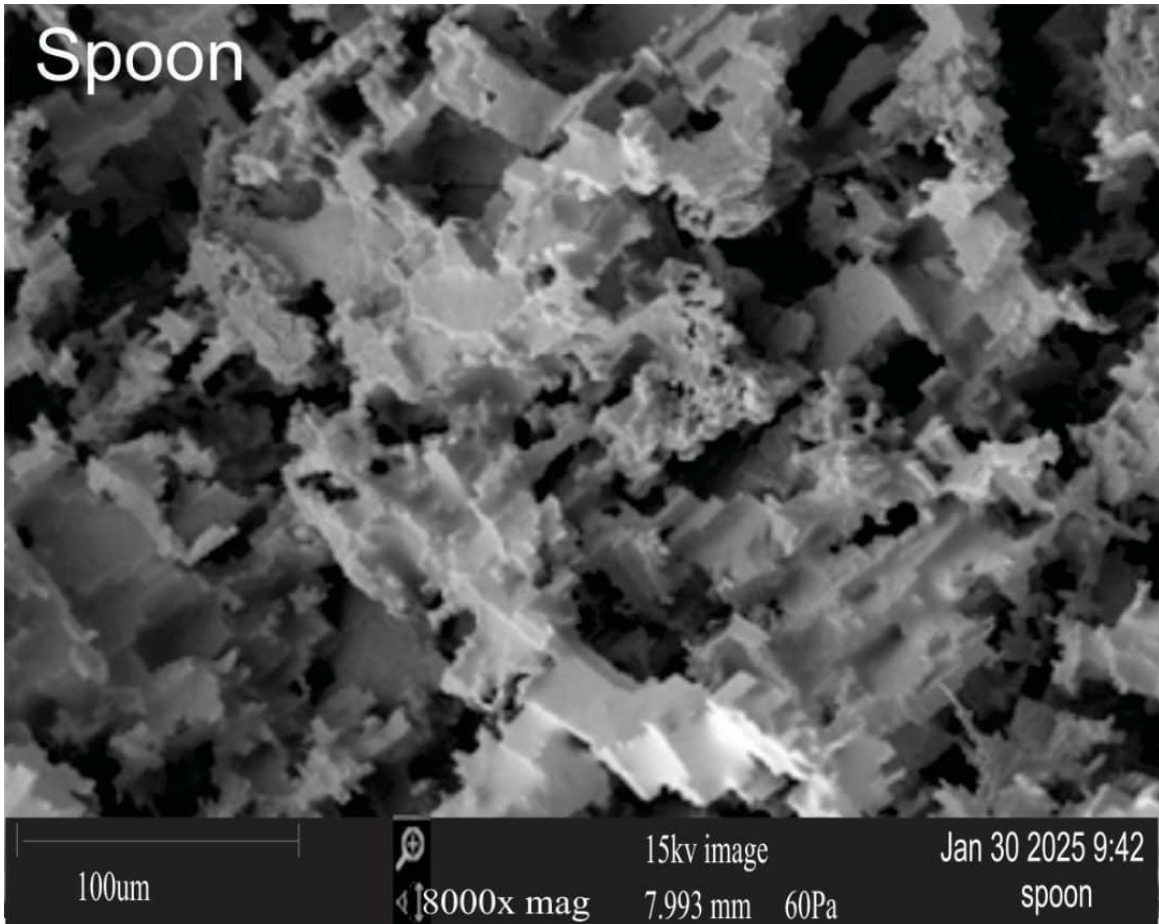


Plate 4.3: SEM result of aluminum spoon (Prototype) with 8000x magnification

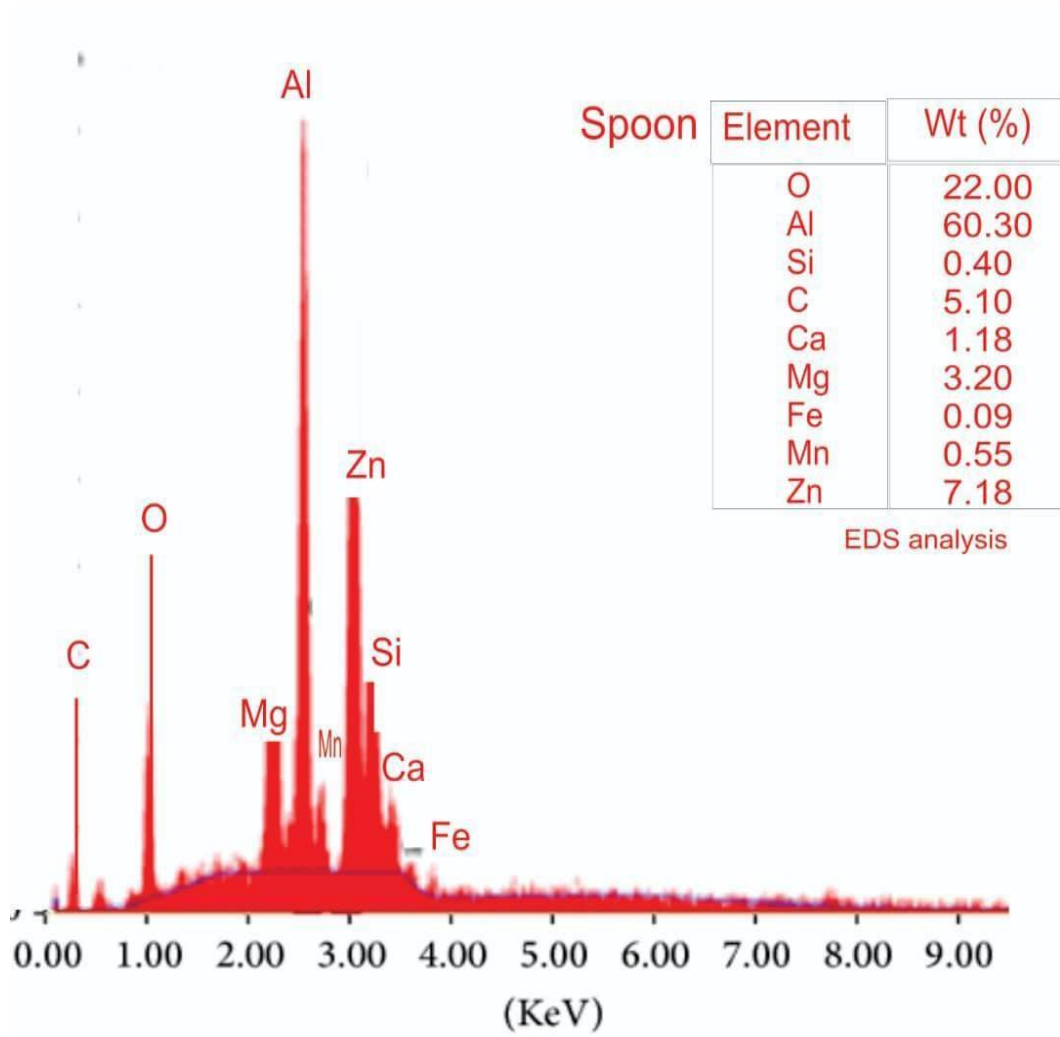


Plate 4.4: EDS results of aluminum spoon (prototype)

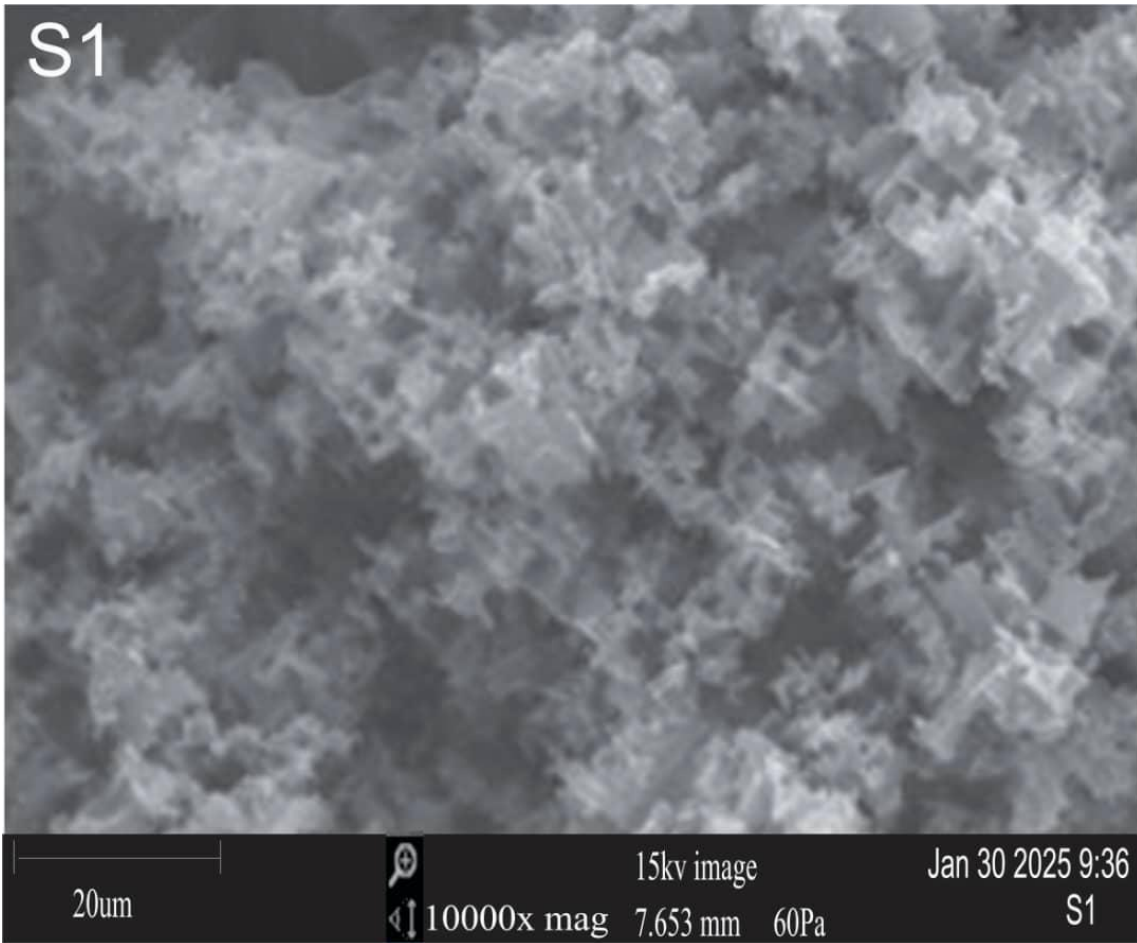


Plate 4.5: SEM result of aluminum alloy with 10wt % eggshell composition (1000x magnification)

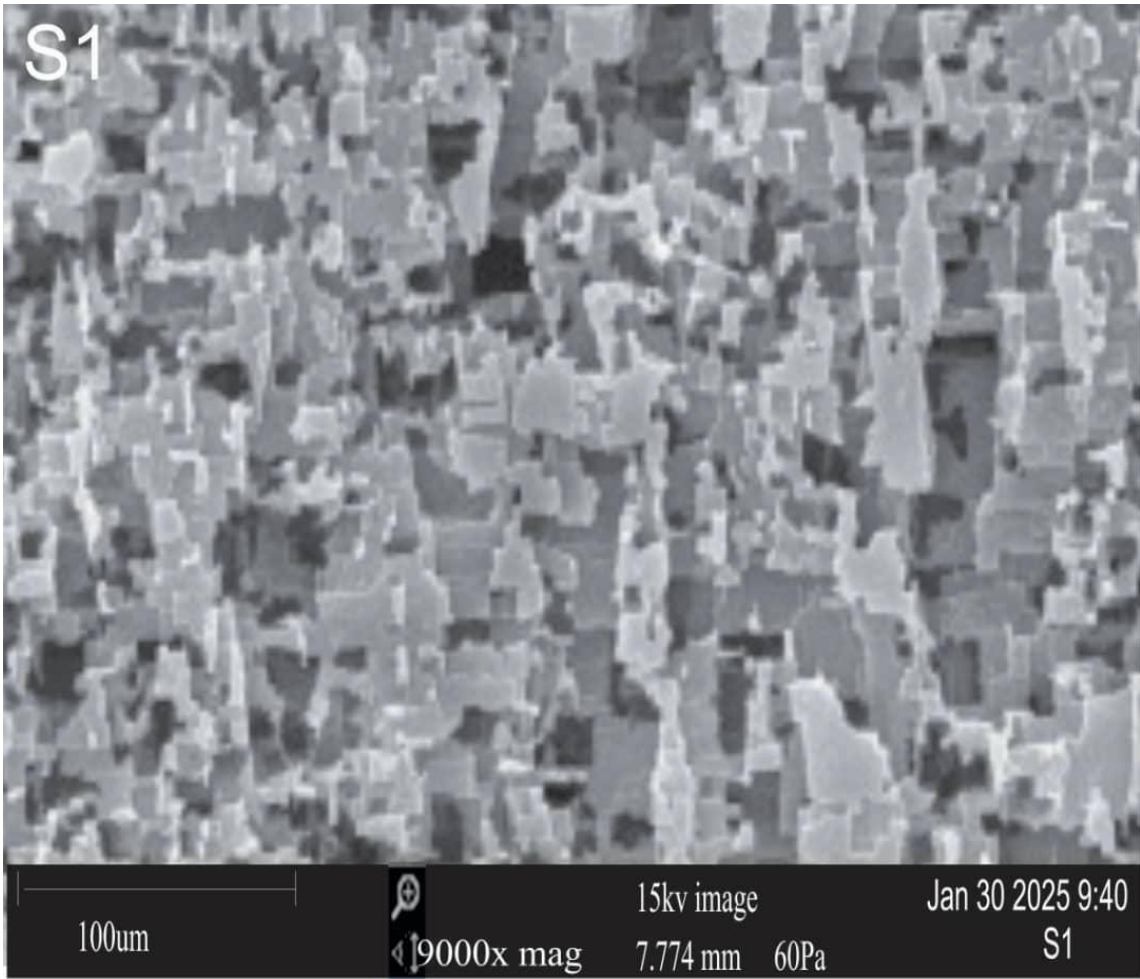


Plate 4.6: SEM result of aluminum alloy with 10wt % Eggshell composition (9000x magnification)

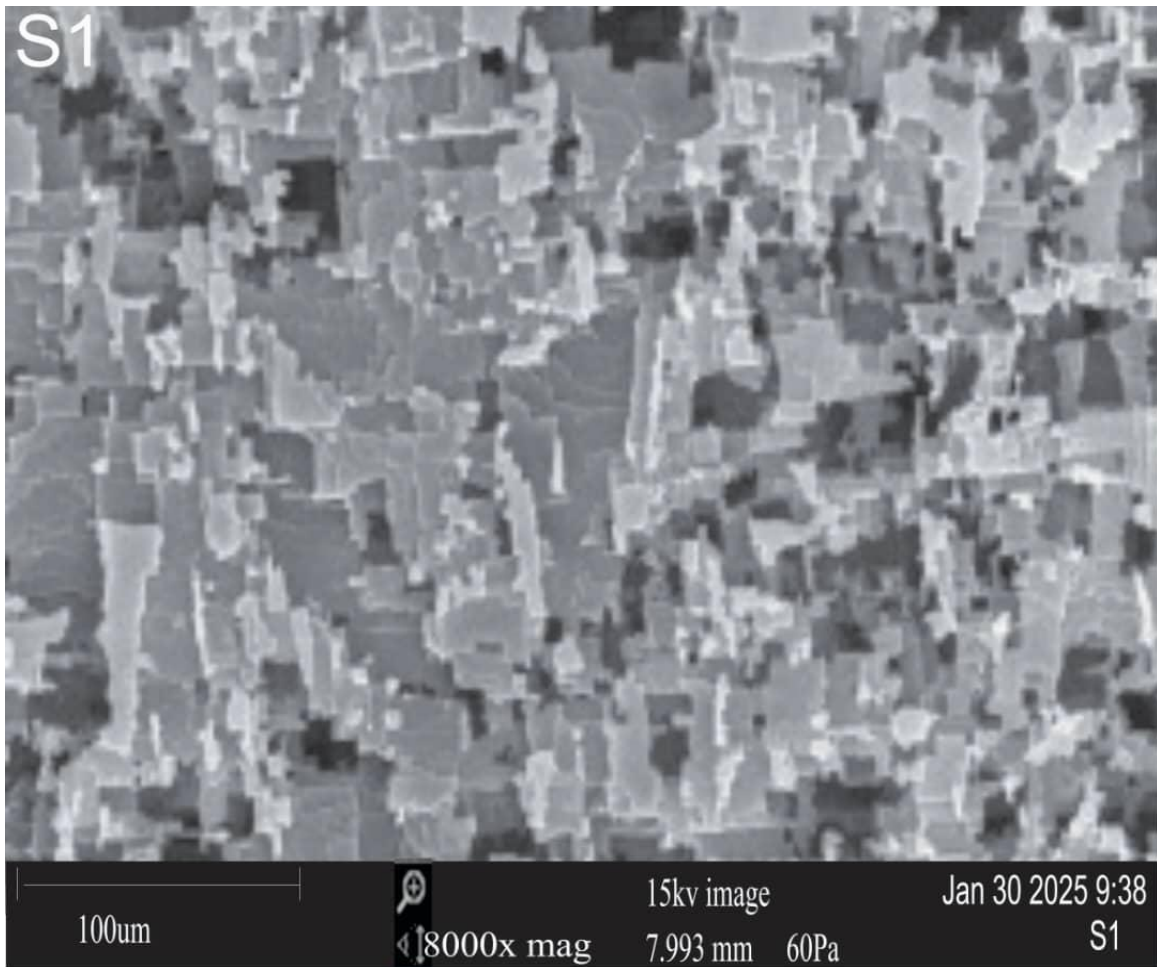


Plate 4.7: SEM result of aluminum alloy with 10wt % eggshell composition (8000x magnification)

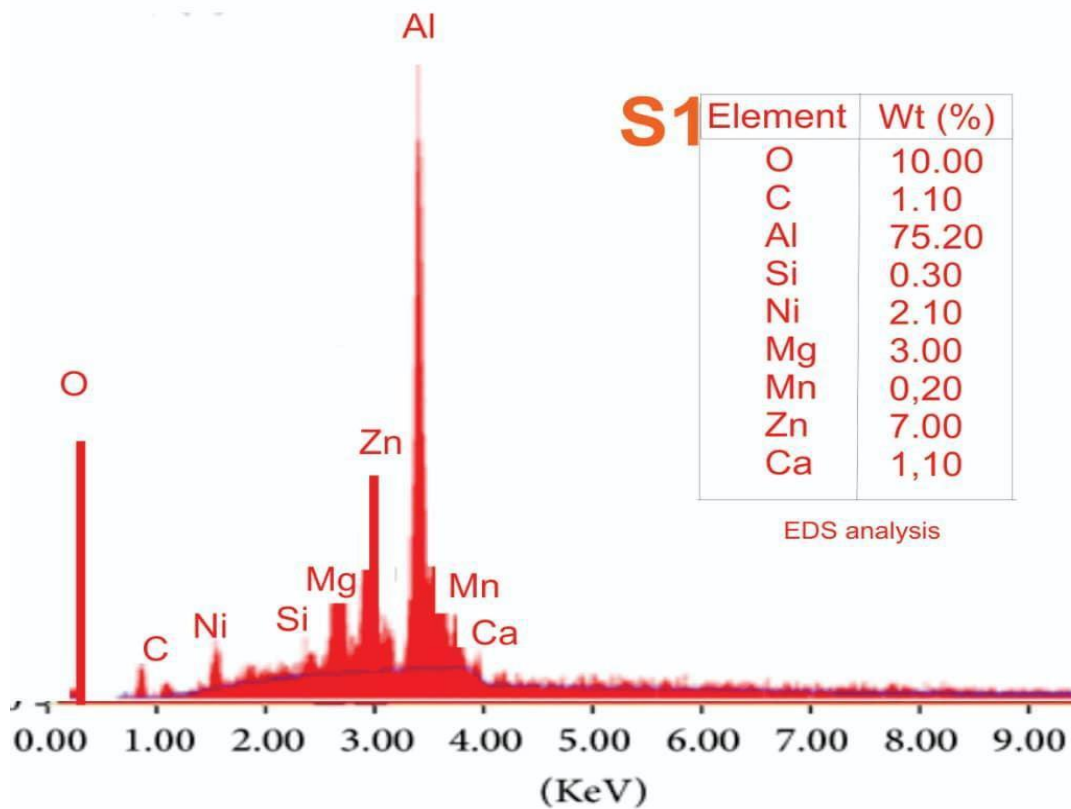


Plate 4.8: EDS results of aluminum alloy with 10wt % eggshell composition

**Plate 4.1** shows the surface morphology of the aluminum spoon prototype, revealing a complex microstructure at a magnification of 10000x. The surface is characterized by a notable roughness, exhibiting a faceted texture with irregularly shaped features. The presence of these features, as highlighted by the varying contrasts in the image (lighter and darker regions), suggests a non-uniform surface structure. The lighter areas may correspond to regions of higher electron reflectivity, potentially indicating differences in surface orientation or localized variations in chemical composition. Conversely, the darker regions may be attributed to surface irregularities such as pits or pores, or possibly areas where the electron beam is less efficiently reflected. The sharp edges and angular shapes observed throughout the microstructure underscore the irregular nature of the spoon's surface. This baseline analysis of the aluminum spoon provides a crucial reference point for evaluating the impact of eggshell incorporation on the microstructure of the aluminum matrix composites developed in this study.

#### 4.1.1 Analysis the EDS data of aluminum spoon

Plate 4.4 shows the **weight percentage (wt.%)** of each element detected on the surface of the aluminum spoon.

- **Aluminum (Al) - 60.30 wt.%:** It's an aluminum spoon, so aluminum is the dominant element. The percentage is not 100%, which is expected. surfaces rarely are perfectly pure.
- **Oxygen (O) - 22.00 wt.%:** This is a significant presence. It suggests the aluminum has likely formed some **aluminum oxide (Al<sub>2</sub>O<sub>3</sub>)** on the surface due to exposure to air. Aluminum readily oxidizes, creating a thin layer that can affect its properties.
- **Carbon (C) - 5.10 wt.%:** Carbon is common. It could be from atmospheric contamination, residues from manufacturing, or even from the carbon tape used in the SEM sample preparation itself.
- **Zinc (Zn) - 7.18 wt.%:** This is an interesting find. Zinc is often used as a coating to protect metals from corrosion (galvanizing). It might suggest the spoon is made from a specific aluminum alloy containing zinc, or that it underwent some surface treatment.
- **Magnesium (Mg) - 3.20 Wt.%:** Magnesium is another common alloying element in aluminum alloys, often added to improve strength and other properties.
- **Calcium (Ca) - 1.18 wt.%:** This points out that the prototype aluminum spoon is not pure aluminum alloy. Calcium present may be an impurity and it's also worth noting that calcium is a major component of eggshells, so it'll be important to track how this changes in the composites.
- **Silicon (Si) - 0.40 wt.%:** Silicon is another common element found in aluminum alloys, often as a strengthening agent.
- **Iron (Fe) - 0.09 wt.% and Manganese (Mn) - 0.55 wt.%:** These are present in very small amounts and are likely impurities.

The EDS analysis presented in Plate 4.4 reveals the elemental composition of the aluminum spoon prototype. As expected, aluminum is the primary constituent (60.30 wt.%), confirming the base material. However, the presence of a substantial oxygen content (22.00 wt.%) suggests the formation of a surface oxide layer, likely aluminum oxide, which is common in aluminum exposed to atmospheric conditions. This oxide layer can influence the surface properties of the spoon and should be considered when evaluating the performance of the aluminum-eggshell composites. Notably, the analysis also detected significant amounts of zinc (7.18 wt.%) and

magnesium (3.20 wt.%), indicating the possible presence of alloying elements. These additions could be related to the specific grade of aluminum used or potentially from a galvanizing process. The presence of calcium (1.18 wt.%) is particularly interesting, especially in the context of this study involving eggshells, which are rich in calcium. While the origin of calcium in the spoon remains unclear (possibly from recycled aluminum or a specific alloy), it establishes a baseline level for comparison with the composites, where calcium will be intentionally introduced via the eggshell reinforcement. The minor presence of silicon, iron, and manganese suggests the possible presence of impurities.

#### **4.1.2 Analysis of the 10 wt.% Eggshell Composite (S1):**

- i). Texture and morphology: The image labeled "S1" shows a surface that is still rough but appears somewhat different from the aluminum spoon prototype. There is a more interconnected, almost web-like structure in some areas. This could be due to the eggshells integrating (or attempting to integrate) with the aluminum matrix.
- ii). Feature size and distribution: The texture seems finer in some regions, suggesting the eggshells might be influencing the aluminum's microstructure at this scale.

#### **4.1.3 Discussion and comparisons of the SEM images of 10wt.% eggshell sample(S1) and aluminum spoon**

While the surface of the 10 wt.% composite remains rough, a characteristic inherited from the base aluminum, there are indications of a more interconnected and potentially finer textured structure in certain regions. This variation in texture could be attributed to the interaction between the added eggshell particles and the aluminum during processing. The eggshells, with their inherent calcium carbonate and other mineral constituents, may be acting as nucleation sites or influencing the solidification behavior of the aluminum, leading to the observed microstructure. However, it is crucial to examine the distribution of the eggshell particles. Areas exhibiting a more uniform texture may indicate a more homogeneous dispersion of the reinforcement within the aluminum matrix. Conversely, regions displaying localized agglomerations or clusters of eggshells could suggest challenges in achieving a uniform distribution, which is a common issue in composite materials. The porosity of the 10wt% composite appears to be generally comparable to that of the unreinforced aluminum spoon. This suggests that the addition of eggshells, at this concentration and under the processing conditions used, did not significantly alter the overall porosity of the material.

#### 4.1.4 Analysis of the EDS Data for S1:

- **Aluminum (Al) - 75.20 wt.%:** The percentage is higher than in the aluminum spoon (60.30%). This is likely because the relative amount of other elements is reduced due to the addition of eggshells, which don't contain much aluminum.
- **Oxygen (O) - 10.00 wt.%:** A significant decrease compared to the spoon. This suggests that the eggshells are influencing the surface oxidation of the aluminum. It might also be related to the way the composite was processed.
- **Carbon (C) - 1.10 wt.%:** A drastic reduction from the spoon. This is also significant and suggests that the carbon in the composite might be coming primarily from the eggshells (which contain calcium carbonate).
- **Zinc (Zn) - 7.00 wt.%:** Still present at a similar level to the spoon, indicating it's likely an alloying element in the base aluminum.
- **Magnesium (Mg) - 3.00 wt.%:** Likely another alloying element.
- **Calcium (Ca) - 1.10 wt.%:** It's at a similar level to the spoon, despite adding eggshells. This suggests that either the eggshells aren't dispersing well, or the calcium in the eggshells isn't being detected effectively by EDS (possibly due to it being bound in the form of calcium carbonate).
- **Nickel (Ni) - 2.10 wt.%:** Nickel was not detected in the spoon sample. This could indicate contamination during processing or an unexpected component of the eggshells themselves.
- **Silicon (Si) - 0.30 wt.% and Manganese (Mn) 0.20 wt.%:** These are still present at low levels.

#### 4.2. Analysis of the Brinell hardness data

**Table 4.3: Brinell hardness data**

SAMPLE	BRINELL HARDNESS NUMBER (BHN)
Al + 7% weight of Egg-shell(S3)	43.70
Al + 10% weight of Egg-shell(S1)	48.12
Al + 13% weight of Egg-shell(S2)	55.74

##### 4.2.1 Effect of eggshells on hardness and mechanical behavior

Table 4.3 presents the Brinell Hardness Number (BHN) for aluminum-eggshell composites with varying eggshell percentages.

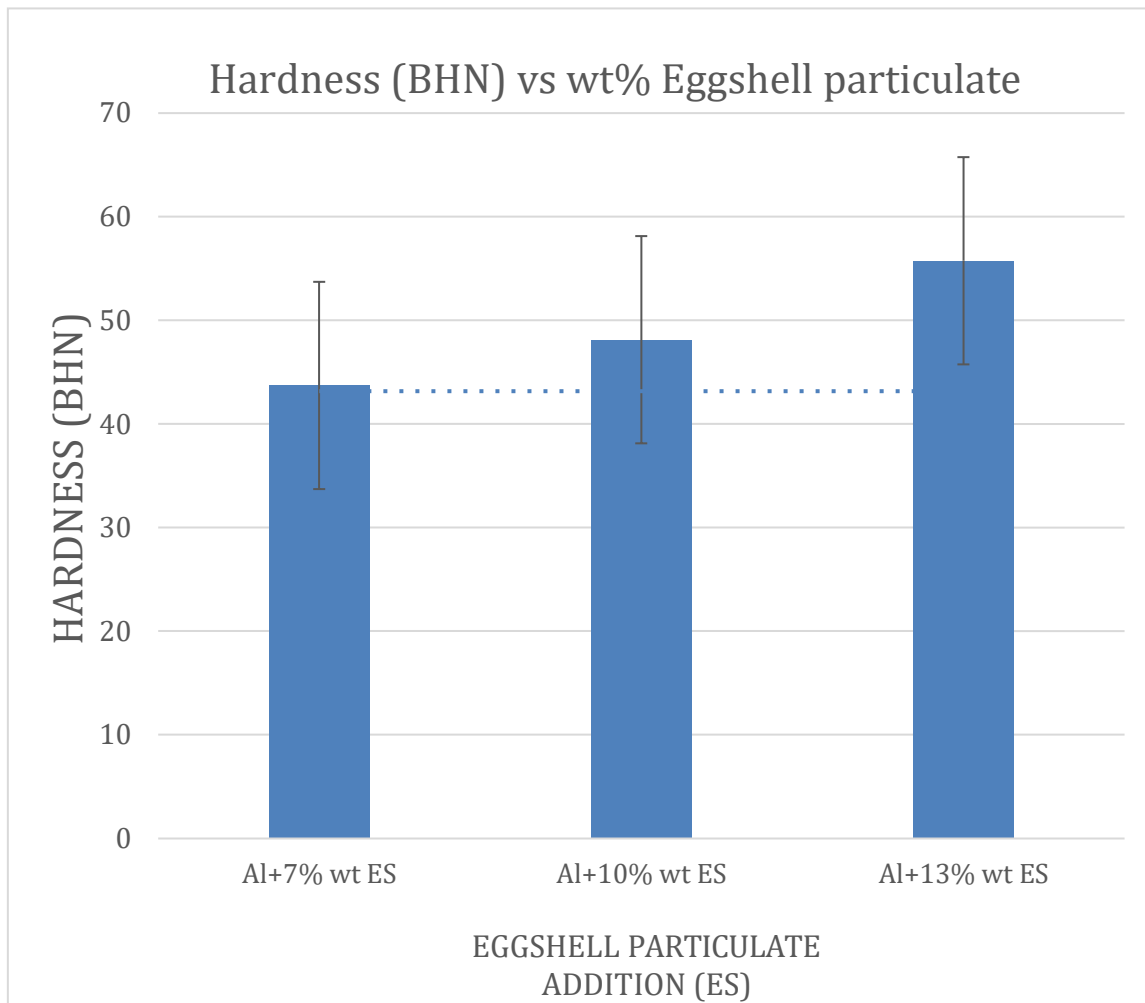
**LOW EGGSHELL CONTENT:** The composite containing the lowest amount of eggshell, specifically Al + 7% weight of Egg-shell (S3), exhibited a Brinell Hardness Number (BHN) of 43.70. This value represents the baseline hardness among the tested composite samples. At this lower concentration, the contribution of the eggshell reinforcement to the overall hardness appears to be less pronounced compared to samples with higher eggshell content.

**MEDIUM EGGSHELL CONTENT:** When the eggshell content was increased to a medium level, as seen in the Al + 10% weight of Egg-shell (S1) composite, the Brinell Hardness Number showed an increase to 48.12. This suggests that a moderate addition of eggshell particles begins to have a more noticeable effect on enhancing the hardness of the aluminum matrix. The greater presence of the reinforcing phase starts to impede deformation more effectively.

**HIGH EGGSHELL CONTENT:** The composite with the highest proportion of eggshell in the tested samples, Al + 13% weight of Egg-shell (S2), displayed the highest Brinell Hardness Number of 55.74. This outcome indicates a clear trend: as the weight percentage of eggshell increases within the aluminum matrix, the resulting composite exhibits a greater resistance to indentation, signifying improved hardness. The higher volume fraction of the reinforcing eggshell particles likely provides a more significant barrier to plastic deformation.

#### **4.2.2 Overall mechanical behavior:**

The data from the table demonstrates a positive correlation between the weight percentage of eggshell in the aluminum composite and its Brinell Hardness Number. Higher eggshell content within the tested range leads to increased hardness.



Samples

Figure 4.9: Bar chart of hardness variation in samples

Here is the hardness variation bar chart for the given material samples. The Brinell Hardness (BHN) values are plotted for different compositions, showing how hardness increases with the addition of eggshell reinforcement, reaching a peak at 13%.

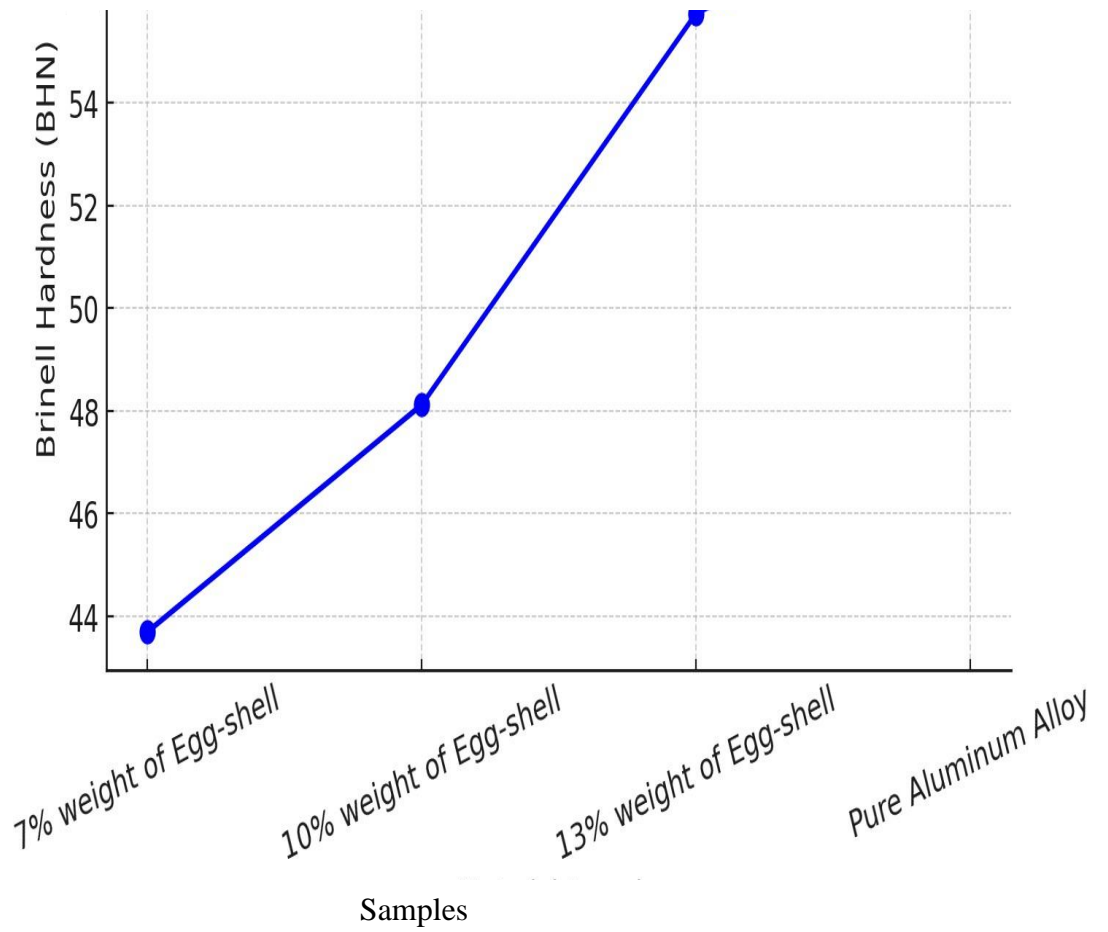


Figure 4.9.1: Graph of the hardness variation in samples

### 4.3 Analysis of the tensile test data

**Table 4.4: Tensile test data**

Sample	Initial Length(m)	Final Length (mm)	D <sub>o</sub> Diameter	D <sub>f</sub> Diameter	Load (KN)	Stress (MPa)	Strain (% Elongation)	UTS (MPa)
Pure Al	200.00	210.00	15.7	15.7	13.00	67.143	5.00	13.83
Pure Al	182.00	183.00	15.7	15.7	8.00	41.318	0.55	75.12
Al+10wt.% Egg-shell	203.00	204.00	15.7	15.7	8.00	41.318	0.50	82.64
Al+13wt.% Egg-shell	192.00	193.00	15.7	15.7	13.00	67.143	0.50	134.29
Al+7wt.% Egg-shell	203.00	207.00	15.7	15.7	18.50	95.549	1.97	48.50
Al+7wt% Egg-shell	201.00	206.00	15.7	15.7	11.50	59.395	2.49	23.85

#### 4.3.1 Pure aluminum baseline:

- The table provides data for two pure aluminum samples. This establishes a baseline for comparing the composite materials.
- The table also shows the variation in UTS and strain between the two pure aluminum samples. This highlights the inherent variability in material testing and the importance of multiple samples.

#### 4.3.2 Effect of eggshell reinforcement:

- The table shows results for aluminum composites with 7%, 10%, and 13% egg-shell reinforcement.
- **UTS:** Observe the trend in UTS. The 13% egg-shell composite exhibits the highest UTS (134.29 MPa), significantly higher than pure aluminum. This indicates that egg-shell reinforcement can substantially increase the tensile strength of aluminum.

- **Strain (%Elongation):** The strain values for the egg-shell composites are generally lower than pure aluminum. This suggests that the addition of egg-shell reduces the ductility of the material, making it more brittle.
- **Stress:** The stress values follow a similar trend to UTS, with the 13% composite showing the highest stress.

#### **4.3.3 Optimum reinforcement percentage:**

- The data suggests that 13% egg-shell reinforcement provides the highest strength enhancement. However, it's crucial to consider the trade-off with ductility.
- The 7% egg shell sample had a great stress value, but a lower UTS than the 13% sample. The 10% sample had the lowest stress and UTS values of the egg shell added samples.
- Further investigation with more data points would be needed to determine the precise optimal percentage for desired properties.

#### **4.3.4 Microstructural implications:**

- The changes in mechanical properties (UTS and ductility) are directly related to the micro-structure of the composites.
- Egg-shell particles likely act as reinforcement by hindering dislocation movement within the aluminum matrix, leading to increased strength.
- The distribution, size, and bonding of the egg-shell particles with the aluminum matrix are crucial factors affecting the mechanical behavior.

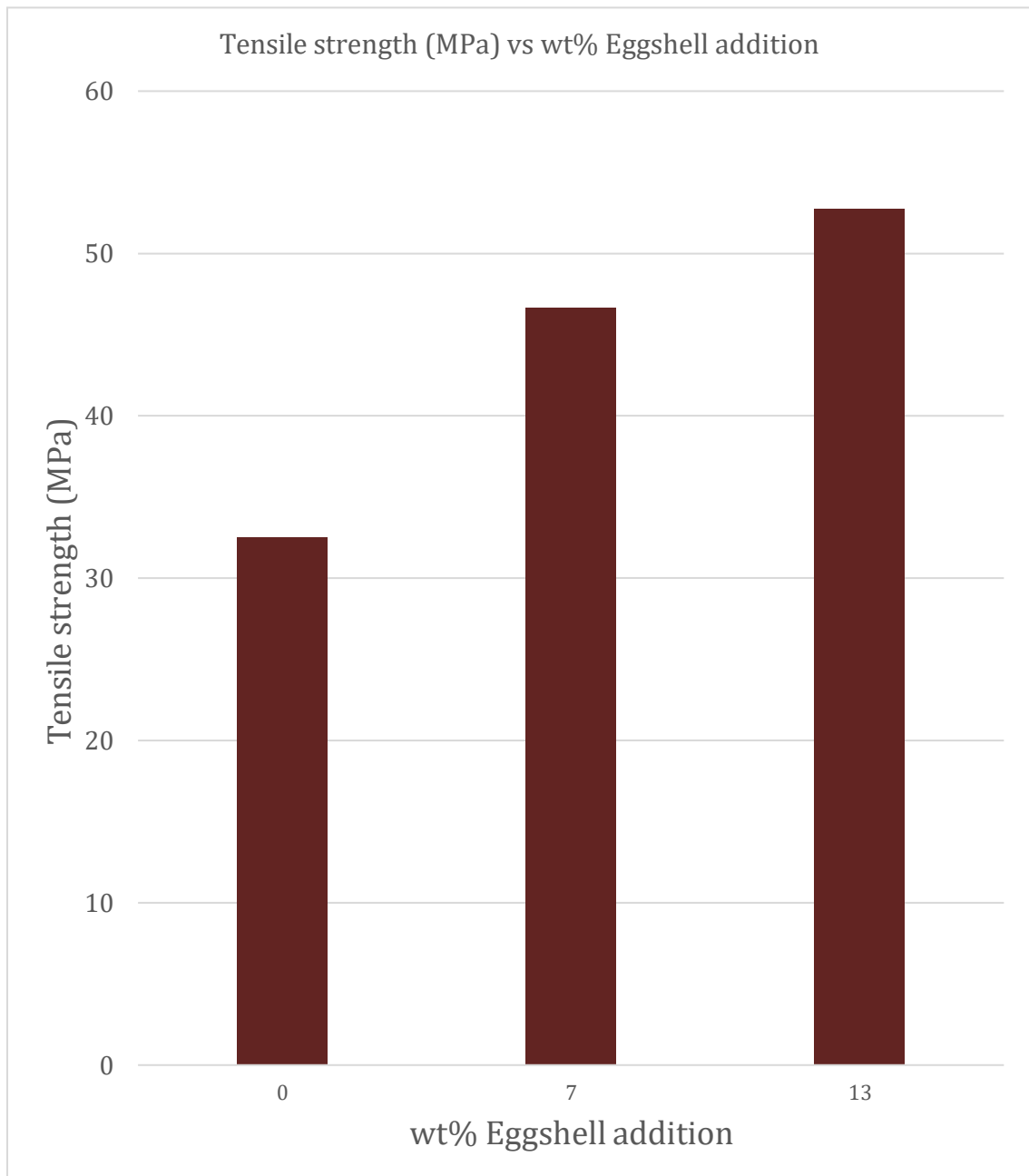


Figure 4.9.2: Tensile strength (MPa) vs wt.% Eggshell addition

#### 4.4 Analysis of the differential scanning calorimetry results

Sample: Sample PS  
Size: 125.35mg  
Method: 4C/mon tp 900C

DSC File: C:/sample ps2025  
Operator: CRA  
Run Date: 10-Feb-25 10:30

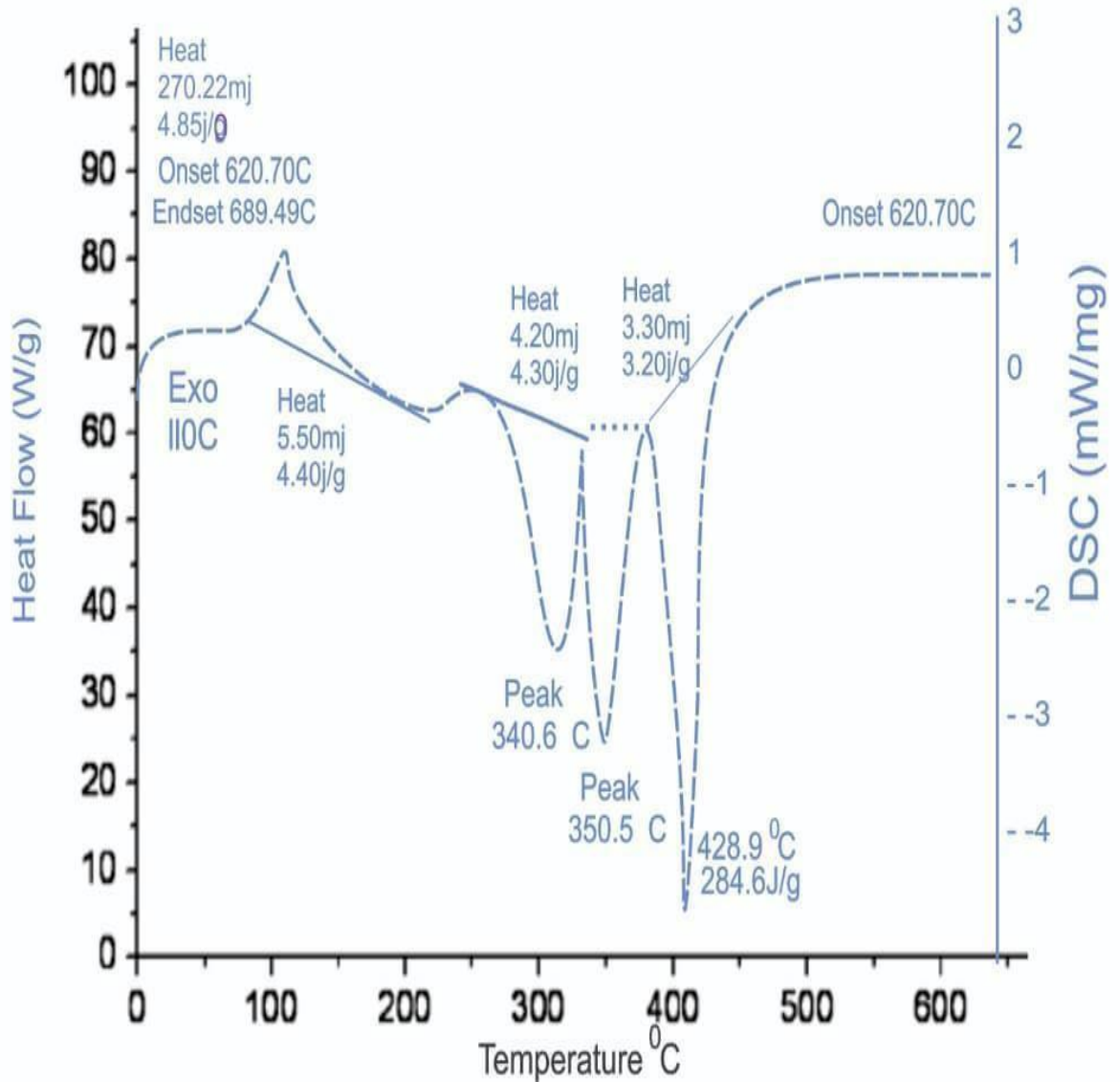


Figure 4.9.3: DSC graph of heat flow Vs temperature for pure aluminum sample

Sample: Sample S2  
Size: 125.35mg  
Method: 4C/mon tp 900C

DSC File: C:/sample s22025  
Operator: CRA  
Run Date: 10-Feb-25 10:22

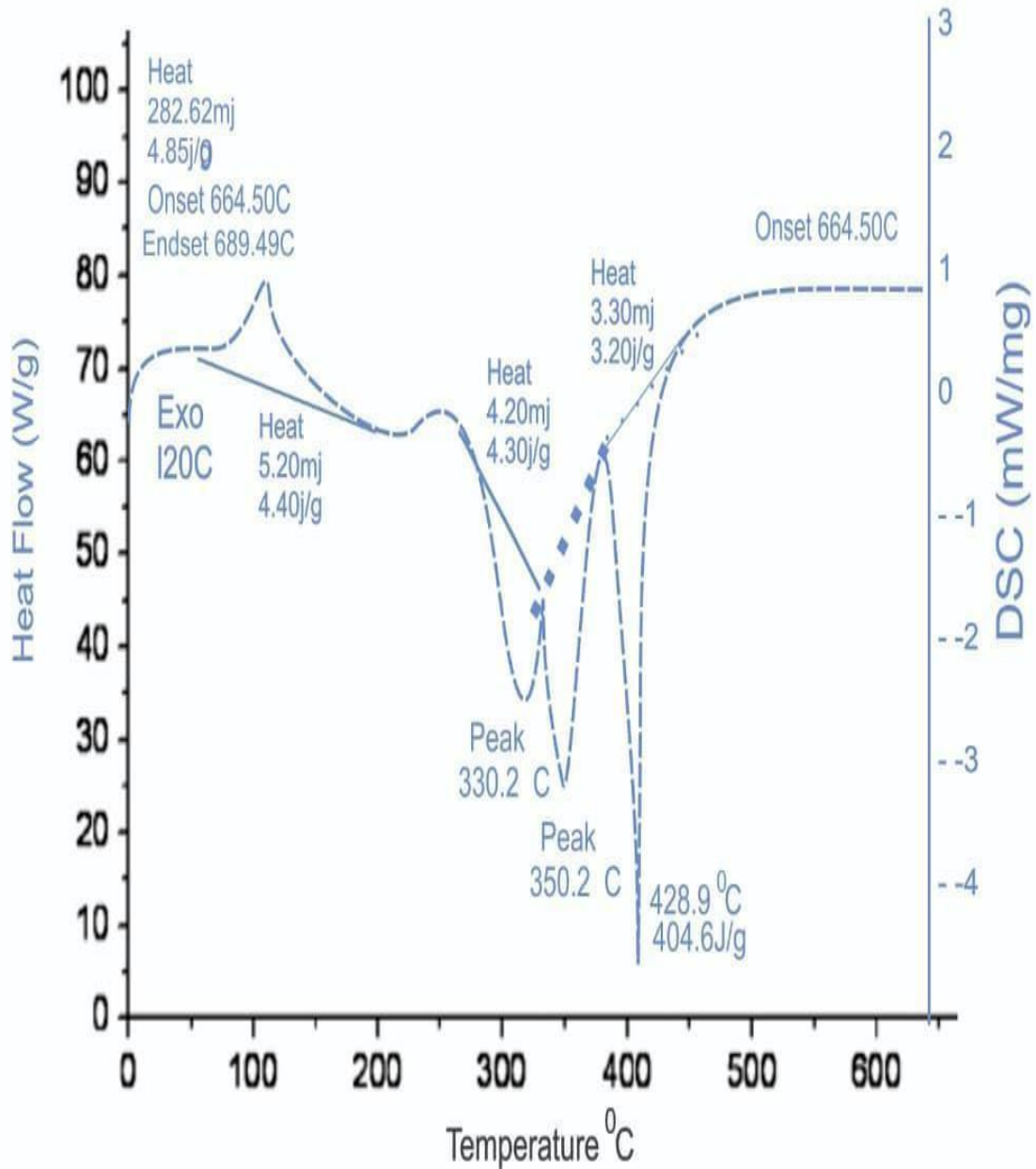


Figure 4.9.4: DSC graph of heat flow Vs temperature for S2(AI+13wt.% eggshell)

#### 4.4.1 Analyzing the DSC Curve of PS (pure aluminum)

**Baseline:** Similar to S2 sample, the initial flat region indicates thermal stability in that temperature range.

- Small exothermic event around 110°C: This small, broad exothermic peak is likely due to:

1. Surface oxidation: Aluminum can react with oxygen in the air, forming a thin oxide layer. This reaction can release a small amount of heat.

2. Possible contaminants: There might be trace impurities or residues on the sample surface that react or decompose in this temperature range.

- Exothermic Events between roughly 280°C to 450°C: This region, while it showed a sharp peak in S2 sample (likely from eggshell decomposition), is less pronounced here. It could indicate:

1. Aluminum carbide formation: If there's any carbon present (even as a trace contaminant), it could react with aluminum at these temperatures to form aluminum carbide ( $Al_4C_3$ ). This reaction is exothermic.

2. Other intermetallic formation: Depending on the specific aluminum alloy, there might be other minor intermetallic phases that form or transform in this range, releasing heat.

- Endothermic events around 620°C - 690°C: This is the melting region of aluminum. The broadness of the peak suggests:

3. Alloy composition: Pure aluminum should have a sharp melting point. The broader peak indicates your sample is likely an aluminum alloy, not commercially pure aluminum. Different alloying elements melt at slightly different temperatures, causing the broader range.

4. Instrumental factors: Factors like the heating rate and sample size can also influence the peak shape.

#### 4.4.2 Analysis of the DSC curve of S2 (13wt.% eggshell)

- Endothermic events: These absorb heat (going upwards on the graph) and often relate to transitions like melting.

- Exothermic events: These release heat (going downwards) and can indicate crystallization or reactions.
- Baseline: The relatively flat region before major peaks indicates the stable thermal behavior of the material in that temperature range.

Broad Endothermic Peak around 120°C: This broad peak might be due to:

- Moisture Evaporation: Eggshells contain organic matter and carbonates, which can hold moisture. This peak could represent the energy required to remove this moisture.
- Decomposition of Organic Components: Some initial decomposition of organic matter in the eggshell might be initiated around this temperature.
- Exothermic Events between roughly 250°C to 400°C: This region is crucial and likely indicates the decomposition and transformation of the calcium carbonate ( $\text{CaCO}_3$ ), the major component of eggshells.

The sharp exothermic peak suggests a more rapid reaction. This is typical of  $\text{CaCO}_3$  decomposition, which releases  $\text{CO}_2$ . This decomposition is essential because it can influence the interfacial bonding between the aluminum matrix and the eggshell reinforcement.

Smaller Endothermic Events around 664°C - 689°C: This is in the range where you might expect to see the onset of melting for the aluminum matrix. However, it's not a sharp, distinct melting peak. This suggests that:

1. The presence of the eggshells might be hindering or interfering with the clear melting behavior of the aluminum.
2. The smaller peak could also indicate that only a portion of the aluminum is melting, possibly due to variations in the composite's microstructure.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

This study investigated the influence of eggshell reinforcement on the microstructure, mechanical properties, and composition of aluminum alloy-eggshell composites. The research encompassed a detailed analysis of the composite's surface morphology using Scanning electron microscopy (SEM), elemental composition via energy dispersive spectroscopy (EDS), tensile testing and hardness evaluation through Brinell hardness testing. The key findings and conclusions derived from this investigation are summarized below:

- **Microstructural changes:** The SEM analysis revealed distinct differences in the surface morphology of the aluminum alloy reinforced with 10wt% eggshells compared to the unreinforced aluminum spoon. The composite exhibited a more interconnected, finer textured structure in certain regions, suggesting that the incorporation of eggshells influences the aluminum matrix's microstructure. However, the distribution of eggshell particles appeared non-uniform, indicating potential challenges in achieving homogeneous dispersion.
- **Hardness behaviour:** Brinell hardness testing revealed a relationship between eggshell content and hardness. It also provided insight into the overall mechanical behaviour of the composites. Hardness is related to a material's resistance to indentation and, to some extent, its strength. The data from the results demonstrates a positive correlation between the weight percentage of eggshell in the aluminum composite and its Brinell Hardness Number. This shows that higher eggshell content within the tested range leads to increased hardness.
- **Tensile behaviour:** Eggshell reinforcement demonstrably boosts the strength of aluminum. Specifically, 13% eggshell content elevates the Ultimate Tensile Strength (UTS) to 134.29 MPa, a significant improvement over pure aluminum. This indicates eggshell particles effectively hinder aluminum deformation. However, increased eggshell content correlates with reduced strain, signifying greater brittleness. While

strength improves, the material's ability to bend or stretch diminishes. The 7% eggshell sample exhibited a strong stress resistance, despite a lower UTS than the 13% sample. This suggests that at 7%, the eggshell distribution may be more uniform, allowing the material to withstand considerable applied load before failure, though its overall maximum force capacity is less than the 13% sample. The 10% sample displayed the lowest stress and UTS values, suggesting potential mixing irregularities at this reinforcement level. 13% eggshell yields the highest strength, but optimizing reinforcement requires balancing strength and flexibility. Further testing with varied eggshell percentages is necessary to determine the optimal balance.

- Thermal behaviour: DSC analysis revealed that the addition of eggshells influences the thermal characteristics of the aluminum matrix. The 13wt% eggshell composite exhibited a slightly *higher* onset and peak melting temperature compared to the unreinforced aluminum, suggesting that the presence of eggshells may be hindering the initiation of melting (Zhang, 2010). The heat of fusion for the composite was also observed to be higher than that of the pure aluminum sample, indicating a change in the energy required for the phase transition. Furthermore, shifts were observed in the exothermic and additional endothermic peaks in the composite's DSC curve, indicating that the eggshells are likely interacting with the processes responsible for these thermal events (Urena, 2003).

## 5.2 Recommendation

Based on the findings of this study, the following recommendations are proposed for future research:

- Optimize Processing Parameters: Further research should focus on optimizing the processing parameters for composite fabrication. This includes exploring different mixing methods, casting techniques, and potentially incorporating steps like sonication or mechanical stirring to improve the dispersion of eggshell particles within the aluminum matrix and minimize agglomeration.
- Enhance Interfacial Bonding: Strategies to improve the interfacial bonding between the eggshells and the aluminum matrix should be investigated. This might involve

surface treatment of the eggshells (e.g., using coupling agents) or exploring different alloying elements in the aluminum matrix that promote better chemical bonding.

- **Control Porosity:** Careful control of the processing atmosphere and parameters is essential to minimize porosity in the composites. Techniques such as vacuum casting or hot pressing could be explored to reduce the formation of voids.
  
- **Advanced Characterization:** More advanced characterization techniques are recommended to gain a deeper understanding of the composite's microstructure and properties. This includes:
  - **Higher magnification SEM and EDS mapping:** To analyze localized regions and confirm the distribution of eggshells and other elements.
  - **X-ray Diffraction (XRD):** To identify the crystalline phases present in the composites, especially calcium-containing compounds from the eggshells.
  - **Tensile Testing:** To directly assess the strength, ductility, and elastic modulus of the composites.
  - **Thermal Analysis (e.g., Differential Scanning Calorimetry - DSC):** To investigate the thermal behavior of the composites and how eggshell addition affects it.
  
- **Modeling and Simulation:** Computational modeling could be employed to simulate the mechanical behavior of the composites and predict the effects of different eggshell distributions and interfacial bonding strengths.

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## APPENDIX I



Eggshell processing

## APPENDIX II



Aluminum Ingot

### APPENDIX III



Mold Preparation

## APPENDIX IV



Crucible furnace and Molten Metal

### APPENDIX III



Fabricated Spoon



Samples Machining