BRNO UNIVERSITY OF TECHNOLOGY

FACULTY OF MECHANICAL ENGINEERING

INSTITUTE OF MATERIALS SCIENCE AND ENGINEERING

WASTE HEAT STORAGE SYSTEMS USING PHASE CHANGE MATERIALS

BATCHELOR THESIS

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SUPERVISOR   Mgr. Jan Čupera

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Assignment Bachelor's Thesis

Institut:	Institute of Materials Science and Engineering
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As provided for by the Act No. 111/98 Coll. on higher education institutions and the BUT Study and Examination Regulations, the director of the Institute hereby assigns the following topic of Bachelor's Thesis:

Waste heat storage systems using phase change materials

Brief Description:

Waste heat storage systems using phase change materials are an efficient way to store thermal energy (eg. industrial waste heat) and have the advantages of high storage density and isothermal nature. I.e. such systems have the ability to absorb a large amount of thermal energy, without increasing the temperature of the system itself. In practice, such a system can be achieved by creating a binary system of metals with complete insolubility in the solid state, in which the matrix (a metal with a higher melting point) ensures sufficient thermal conductivity and a filler metal with a low melting point and a high enthalpy ensures the accumulation of thermal energy. For example, it could be a binary system Al–Sn or Fe–Cu. Such systems could then be used for the accumulation and subsequent gradual release of thermal energy from components with complex shapes, on which this binary system could be applied using the cold spray method.

Bachelor's Thesis goals:

The student will aim to study literary sources and create research dealing with the topic of systems capable of accumulating thermal energy. In the practical part, the student creates a list of technically important metals in the range of melting temperatures, e.g. 30–700 °C, their melting temperatures, thermal conductivities and melting enthalpies, and subsequently selects suitable candidates from this list for the creation of a binary system capable of accumulating thermal energy. In the next phase, the selected binary system will be created. Finally, the selected system will be subjected to the examination of the microstructure by microscopic techniques (light, electron microscopy) and the ability to accumulate thermal energy will be analyzed by the method of differential scanning calorimetry (DSC). Technical skills and the ability to study documents in English are required for the successful completion of a bachelor's thesis.

Recommended bibliography:

PTÁČEK, Luděk. Nauka o materiálu I. 2., opr. a rozš. vyd. Brno: Akademické nakladatelství CERM, c2003. ISBN 8072042831

SUGO, H.; et al. Miscibility gap alloys with inverse microstructures and high thermal conductivity for high energy density thermal storage applications. Applied Thermal Engineering 2013, 51/1, 1345–1350

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FURQAN, J.; HAMZA, B. Thermal Energy Storage, 2021th ed.; Springer: Singapore

Deadline for submission Bachelor's Thesis is given by the Schedule of the Academic year 2022/23

In Brno,

L. S.

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Director of the Institute	FME dean

Abstract

This bachelor thesis deals with waste heat storage systems using phase change materials as an efficient way to store thermal energy (e.g., industrial waste heat) and have the advantages of high storage density and isothermal nature. I.e., such systems have the ability to absorb a large amount of thermal energy, without increasing the temperature of the system itself. In practice, such a system can be achieved by creating a binary system of metals with complete insolubility in the solid state, in which the matrix (a metal with a higher melting point) ensures sufficient thermal conductivity and a filler metal with a low melting point and a high enthalpy ensures the accumulation of thermal energy. For example, it could be a binary system Al–Sn or Fe–Cu. Such systems could then be used for the accumulation and subsequent gradual release of thermal energy from components with complex shapes, on which this binary system could be applied using the cold spray method.

Keywords

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Declaration

I declare that I prepared my bachelor's thesis on the topic of Waste Heat Storage Systems Using Phase Change Materials independently using professional literature and sources that are listed in the bibliography.

In Brno 2.6. 2023   Signature:...............................

Acknowledgment

Index

Introduction

Thermal Energy Storage (TES) systems using Phase Change Materials (PCMs) can store heat or cold to be used later under varying conditions such as temperature, place, or power. The main use of TES is to overcome the discrepancy between energy generation and energy use. In TES systems energy is supplied to a storage system to be used at a later time, involving three steps: charge, storage, and discharge. [1]

The main requirements for designing a TES system are high energy density in the storage material (storage capacity), good heat transfer between the Heat Transfer Fluid (HTF) and the storage material, mechanical and chemical stability of the storage material, compatibility between the storage material and the container material, complete reversibility of several cycles, low thermal losses during the storage period, and easy control. Moreover, the most important design criteria are the operation strategy, the maximum load needed, the nominal temperature and enthalpy drop, and the integration into the whole application system. [1]

The benefits that can be obtained when implementing storage in an energy system.

are:

1- Energy Efficiency: By catching and reusing waste heat that would otherwise be wasted, waste heat storage devices can assist enhance energy efficiency. This can result in considerable energy savings as well as lower greenhouse gas emissions.
2- Cost Savings: Businesses and industries may lower their energy bills and operational expenses by capturing and utilizing waste heat, resulting in enhanced profitability and competitiveness.
3- Increased System Reliability: Waste heat storage systems that employ phase change materials can provide a dependable and stable supply of heat that can be used to augment or replace less reliable or more expensive sources of heat.
4- Flexibility: Waste heat storage systems may be built to work with a wide range of heat sources and end-use applications, making them a versatile and adaptable option for a number of industries and processes.
5- Reduced Environmental effect: Waste heat storage systems can assist minimize the environmental effect of industrial operations and power production by lowering energy consumption and greenhouse gas emissions. [2]

The storage material can be water, air, oil, bedrock, brick, concrete, etc. Each material has its advantages and disadvantages, but usually, the material is selected according to its heat capacity and the available space for storage. The amount of energy stored is calculated following eq. (1):

$$Q = m \cdot c_p \cdot \Delta T \qquad (eq.1)\ [1]$$

where Q is the amount of heat stored in the material (J), m is the mass of storage material (kg), c_p is the specific heat of the storage material (J/kg·K), and ΔT is the temperature difference (K).

Chapter 1

Methods of Storing Heat Energy in Metal by PCM

Phase Change Materials (PCMs) are materials that can store and release a large amount of energy in form of heat while transitioning from one physical state to another, such as from a solid to a liquid or from a liquid to a gas. A PCM's heat storage capability can be categorized as positive or negative heat storage. Figure (1.1) shows the cycle of storing heat energy in metals by PCMs. [5]
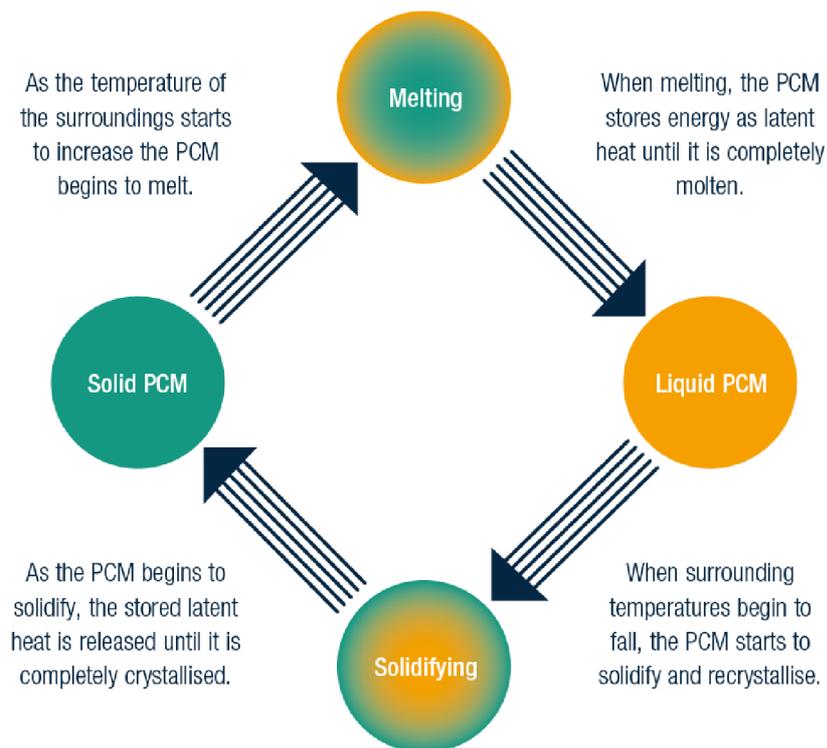

Figure 1.1: cycle of storing heat energy in metals by PCMs. [12]

Positive heat storage indicates that as the PCM transitions from a solid to a liquid phase, it absorbs heat energy, this is known as the melting process. The PCM absorbs heat energy from the surrounding environment throughout this process, resulting in a reduction in the temperature of the surrounding environment. The absorbed heat energy is retained as latent heat in the PCM. [6]

Negative heat storage, on the other hand, indicates that the PCM loses heat energy as it transitions from a liquid to a solid phase. This is referred to as the solidification process. During this process, the PCM emits heat energy into the surrounding environment, raising the temperature of the surrounding environment. The latent heat stored in the PCM during the melting process is responsible for the released heat energy. [6]

As the world moves toward more sustainable and renewable energy sources, energy storage is becoming increasingly important. Energy storage that is efficient and effective is critical for guaranteeing a steady and predictable energy supply, especially for intermittent renewable energy sources like solar and wind power. Heat storage is an important type of energy storage because it allows surplus energy to be kept and released when needed. Heat storage is divided into three forms based on the type of heat energy stored: latent heat storage, sensible heat storage, and thermochemical heat storage. Each method of heat storage has distinct advantages and disadvantages, and the storage type used is determined by the individual application needs. The following context will explore the different types of heat storage and their applications. [7]

a) Latent Heat Storage

Storing energy by latent heat is one of the most popular methods of storing heat energy in metal is latent heat storage. In this method, a phase change material (PCM) is used to store the energy. When heat is applied to the PCM, it undergoes a phase change, either from a solid to a liquid or from a liquid to a gas, depending on the material used. The energy required for this phase change is stored within the material and can be released later when the material undergoes the reverse phase change. Metal containers are often used to store the PCM, as metals are good conductors of heat and can transfer energy efficiently. [1][3]

Calculating the amount of heat absorbed or released during a phase change using Latent Heat Equation:

$$Q_L = mL.$$

Where Q_L is the amount of heat absorbed of released during the phase change (J), m is the mass of the phase change material (kg), and L is the specific latent heat of the fusion or solidification (J/kg).

The principle of latent heat storage is to store energy by changing the phase of a material, such as melting or vaporizing it. This allows for a much larger amount of energy to be stored than with sensible heat storage. Metals can be used as latent heat storage materials by using their high melting or boiling point to store and release energy. [1][3]

Latent heat storage uses the phase transition of a material. Usually, the solid-liquid phase change is used, by melting and solidification of a material. Upon melting heat is transferred to the material, storing large amounts of heat at constant temperature; the heat is released when the material solidifies. Materials used for latent heat storage are called phase change materials (PCM); Metals such as copper, aluminum, and their alloys have been used as PCMs due to their high thermal conductivity and high melting or boiling point. [1]

Many materials have been studied as PCM, but only a few of them have been commercialized, mainly due to problems such as phase separation, subcooling, corrosion, long-term stability, and low heat conductivity, which have not yet been totally solved. Usually, PCMs are selected based on the appropriate melting enthalpy and temperature, availability, and cost. [1]

b) Sensible Heat Storage

Sensible heat storage is another method of storing heat energy in metals. In this method, the metal is heated directly, and the energy is stored as sensible heat by changing the temperature of the storage material without any phase change occurring. This method is often used in conjunction with latent heat storage, as the metal can act as a backup system when the PCM is fully charged. Sensible heat storage can also be used to store energy from sources such as solar panels, which generate heat directly. That's why metals are ideal for sensible heat storage due to their high thermal conductivity and heat capacity. [1]

The equation to the sensible heat storage in waste heat storage systems using phase change materials is the same as the equation to calculate the energy storage eq (1).

The basic principle of sensible heat storage is to heat a material, such as a metal, to an elevated temperature and then store it in an insulated container. When the stored energy is required, the material can be allowed to cool down and release the stored energy. The amount of heat energy that can be stored in a material depends on its heat capacity, which is the amount of heat required to raise its temperature by one degree Celsius. [1]

Metals have a high heat capacity as we mentioned, which means they can store large amounts of heat energy. They are also highly conductive, which allows them to transfer heat quickly and efficiently. [1]

c) Thermochemical Heat Storage

Thermochemical energy storage is another method of storing heat energy in metal. In this method, a chemical reaction is used to store the energy the reactions involve the use of metal oxides or salts that can undergo endothermic or exothermic reactions when exposed to heat. The metal is first heated to a high temperature and then exposed to a chemical reactant, which absorbs the heat energy and undergoes a chemical reaction. The metal can then be cooled, and the energy can be released by reversing the chemical reaction. This method has the advantage of being able to store energy at very high temperatures. [1]

An example of a material is Calcium oxide (CaO), which reacts with water to form calcium hydroxide $(Ca(OH)_2)$ and releases heat:

$$Ca + H_2O \longrightarrow Ca(OH)_2 + Heat \text{ [1]}$$

The equation for estimating a material's Thermochemical Heat Storage capacity is:

$$Q_T = mC_P\Delta T + \Delta H_{rxn} \text{ [4]}$$

Where Q_T is the total heat storage capacity, m is the mass of the material, Cp is the specific heat capacity of the material, ΔT is the temperature change of the material during the reaction, and ΔH_{rxn} is the enthalpy change of the reaction. [1][4]

The total heat storage capacity is the sum of the sensible heat storage ($mC_P\Delta T$) and the heat of reaction (ΔH_{rxn}) associated with the chemical reaction, as shown by this equation. The reaction's enthalpy change (ΔH_{rxn}) might be positive or negative depending on whether the reaction is exothermic or endothermic. [1][4]

Applications of Heat Storage by PCMs

a) Solar Thermal Energy Storage

Latent heat storage in metals can be used for thermal energy storage, where it is used to store excess heat energy generated by various industrial processes, such as metallurgical processes, for later use. The stored energy can then be used to generate electricity or for heating applications. [9]

Sensible heat storage in metals is also used for solar thermal energy storage, where it is used to store the energy generated by solar collectors during the day for use at night or during cloudy weather. Metal-based storage systems are often enclosed in a container and exposed to solar collectors, where they absorb the energy and store it for later use. [9]

Thermochemical heat storage in metals can be used for solar thermal energy storage, where it is used to store the energy generated by solar collectors during the day for use at night or during cloudy weather. Metal oxides or salts are often used as storage materials, which can absorb, or release heat energy as needed. [9]

b) Building Heating and Cooling

Latent heat storage in metals can also be used in building heating and cooling systems, where it is used to regulate the temperature of a building by storing and releasing heat energy as needed. Metal-based PCMs are often integrated into the building's structure, such as the walls or ceilings, and can absorb or release energy as needed. [8][9]

Sensible heat storage in metals is also used in heating, ventilation, and air conditioning (HVAC) systems, where it is used to regulate the temperature of a building by storing and releasing heat energy as needed. Metal-based storage systems are often integrated into the building's structure, such as the walls or ceilings, and can absorb or release energy as needed. [8]

Thermochemical heat storage in metals can also be used in building heating and cooling systems, where it is used to regulate the temperature of a building by storing and releasing heat energy as needed. Metal oxides or salts are often integrated into the building's structure, such as the walls or ceilings, and can absorb or release energy as needed. [8][9]

c) Waste Heat Recovery

Thermochemical heat storage in metals can also be used for waste heat recovery, where it is used to recover the heat energy generated by various industrial processes for later use. The stored energy can then be used for heating applications or to generate electricity. [1]

d) Industrial Processes

Sensible heat storage in metals is widely used in industrial processes, where it is used to store excess heat energy generated by the process for use later. This helps to reduce energy costs and improve efficiency. Metal-based storage systems are often used in conjunction with other storage technologies, such as hot water tanks or heat exchangers, to provide a more efficient and cost-effective solution. [1]

Chapter 2

History of Heat Storage

The use of ice as a phase change material for cold storage is, of course, quite old. Blocks of ice, cut in the winter from frozen lakes or rivers, were stored in "ice houses". Ice is even stored in containers filled with frozen low-melting salt mixtures such as sulfates of magnesium and sodium; potassium, sodium, ammonium, calcium, or magnesium chlorides; or sodium bromide even at summer's end. [8]

Not only in cooling but also in heating applications. Over nearly a century, PCMs have been utilized in "hot bottle" applications for the direct application of warmth to the human body, and in foot warmers, hot plates, and coffee pots. Space heating was attempted around the turn of this century when salt-hydrate-filled, containers were employed to break the winter chill in British rail coaches. [8]

Hydrated sodium acetate was commonly employed for hot bottles but performed erratically. Crooker invented, and with Sterling patented in 1928 an improved sodium acetate PCM, with carefully controlled water content and calcium chloride added to soften the crystalline mass. [8]

In 1929 Jost and Byleveld patented a sealed receptacle in which the PCM- e.g., hydrated sodium acetate, thiosulfate, or sulfate- was mixed with finely divided sand, and contained chunks of metal or pebbles. Heat release was initiated by shaking the container and thus crystallizing the supercooled salt solution. [8]

Figure 2.1: Sodium sulfate decahydrate. [13]

T. P. Bell in 1932 invented and assigned to Thermophore Manufacturing Co. His improved $Na_2SO_4 \cdot 10H_2O - Na_2SO_4$ mixture (Sodium sulfate decahydrate shown in Figure 2.1) for providing heat. Hogan patented a PCM heat storage device containing hydrated sodium thiosulfate. Glucose was added to regulate the crystallization process. [8]

Bowen developed a flexible therapeutic pad containing PCM, suggesting hydrates of sodium metaborate, manganese dichloride, or calcium nitrate as the latent heat storage medium. [8]

In 1932, a Scotsman, A. A. H. Douglas invented a heat storage apparatus containing sealed tubes of PCM in a liquid heat transfer medium. He suggested organic PCMs, such as paraffin, naphthalene, or stearic acid, or hydrated inorganics, such as magnesium sulfate or sodium thiosulfate. Douglas' device was intended for providing hot water supplies or radiant heating for domestic or industrial use. The heat store was charged with off-peak electrical power. [8]

In 1944, A. B. Newton patented a device for radiant nighttime cooling and solar heating, in which the water used for heat transfer and thermal storage was augmented by submerged containers of PCM. Both low melting (10°C) and higher melting (55°C) materials were used. Wood's metal was suggested for the heat storage PCM. For coolness storage, several organic materials, including undecyl alcohol and pentadecane, were proposed. J. R. Swanton, Jr. in 1947 applied for a patent, assigned to A. D. Little, Inc., on a solar heating system employing PCM heat storage. He favored disodium phosphate dodecahydrate as the storage medium and also suggested calcium chloride hexahydrate. He considered Glauber's salt undesirable because of the precipitation of the anhydrous salt. [8]

In 1950 J. E. Feldman of the U.S. Rubber Co. patented heat-producing articles, fabricated of sponge rubber laminated between two rubber sheets. The sponge was filled with an inorganic salt hydrate PCM melting from 28 to 45°C. One article was roughly in the form of a sleeping bag, intended as an arctic rescue bag for injured or incapacitated persons. Another item was a mitten, designed for work under freezing conditions. With this background of PCM applications on Earth and under the oceans, it was natural that PCM thermal control was developed for applications in outer space. [8]

In 1965 Mavleos and Desy invented and patented for Avien Inc., a suit with a warming backpack for divers, explorers, aviators, and others exposed to prolonged cold. Lithium hydride was used as the PCM heat source, and water was circulated to the suit from a heat exchanger in the pack. Hearst and Plum later developed for the Defense Department a similar device with low-melting point PCMs. They suggested, compounds melting from 45 to 90°C, such as stearic acid, naphthalene, hydrated $Mg(NO_3)_2$, or eutectics melting up to 200°C, e.g., $NaNO_3/Mg(NO_3)_2$, or $NaNO_3/ Mg(NO_3)_2/KNO_3$. [8]

The 1960s were years of rapid development of manned space technology. Included in this effort were NASA-sponsored programs to develop PCM thermal control devices. [8]

Working as a subcontractor for M.I.T., in 1962 Cryo-Therm, Inc. studied the application of PCMs as heat sinks for inertial navigation systems. Although the application was limited to a narrow temperature range, 49 to 57°C (120 to 135°F), the literature survey generated information on materials melting from 50 to 3200°C. A technique for estimating the entropy of fusion and calculating the enthalpy of fusion was tested for materials lacking literature values of these data. [8]

In 1964 workers at Northrup Space Laboratories, funded by Marshall Space Flight Center, launched an effort to apply PCM heat storage technology to the space program. Such applications as temperature control for electronics packages, spacecraft temperature modulation, and heat storage for solar electric power systems were investigated. As reported by Shlosinger and Bentilla, a variety of organic PCMs was considered, and normal paraffin was selected as the most promising. Thermal and physical properties were collected or measured for the candidate PCMs. The thermal performance of temperature control units was modeled and verified in a test apparatus. [8]

A study was conducted at Republic Aviation by S. Z. Fixler and coworkers to predict the performance of paraffin PCM spacecraft systems. This showed a 75% or greater reduction in temperature fluctuation for a passive PCM system, and a reduction in weight over other passive thermal control devices. [8]

Grodzka and Fan at Lockheed Missiles and Space Company performed a theoretical study on the operation of PCM devices in the space environment. A group of organic and inorganic candidates was screened experimentally, and four were chosen as prime candidates: lithium nitrate trihydrate, acetamide, methyl fumarate, and myristic acid. [8]

In 1971, Flight systems were developed and used in the manned lunar landing and Skylab programs. The Apollo 15 Lunar Rover Vehicle (LRV) (shown in Figure 2.2) employed three PCM systems on the Signal Processing Unit (SPU) (shown in Figure 2.3), Drive Control Electronics (DCE), and Lunar Communications Relay Unit (LCRU). The heat generated during LRV sorties was stored in paraffin PCM. Between sorties, movable insulating devices allowed the heat to dissipate by radiation. [8][16]
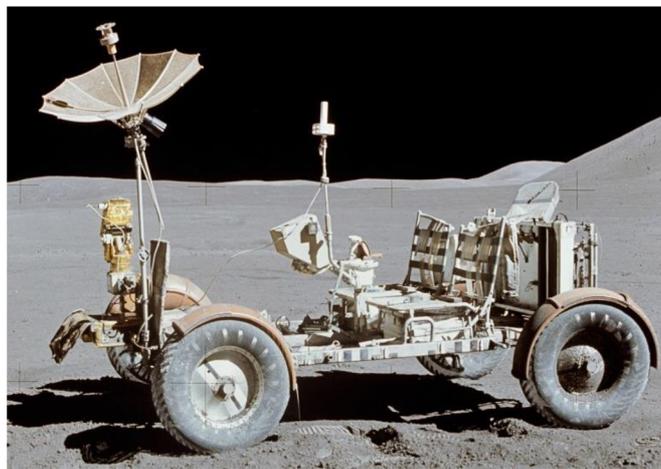

Figure 2.2: The Apollo 15 Lunar Rover Vehicle (LRV)
[14]
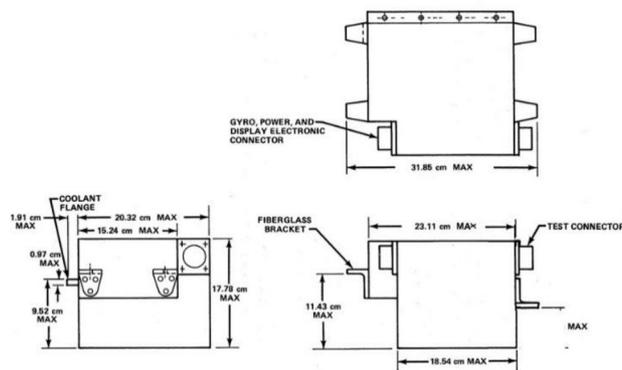
Figure 2.3: Signal Processing Unit (SPU) [15]

The Skylab SL-1 utilized a PCM system for temperature stabilization of containers of human waste during the transfer from the orbital workshop to the command module for return to Earth. Skylab also was fitted with a PCM system to temper the cooling fluid returned from the liquid loop radiator system, preventing excessive temperature excursions in this system. [8]

Since then, several researchers have studied the use of PCMs for thermal energy storage. In the 1970s, researchers continued to explore the use of metal hydrides as PCMs, Metal hydrides have high energy density and can store and release energy during the absorption and desorption of hydrogen. with particular focus on their potential as hydrogen storage materials. However, issues with low thermal conductivity, limited availability of some metal hydrides, and high cost limited their practical applications. [8]

In the 1980s, researchers turned their attention to eutectic mixtures as PCMs, which have lower melting points and higher latent heat storage capacities than pure materials. Eutectic mixtures are formed by combining two or more materials to create a mixture with a lower melting point than any of the individual components. Researchers found that eutectic mixtures of organic compounds, such as fatty acids and alcohols, had promising thermal storage properties. [8]

In the 1980s, researchers explored the use of eutectic mixtures as PCMs. Eutectic mixtures are alloys that melt and solidify at a lower temperature than their constituent metals, making them useful for thermal energy storage. The use of eutectic mixtures in thermal energy storage was first proposed by F. P. Incropera and D. P. DeWitt in their book, Fundamentals of Heat and Mass Transfer, in 1981. Since then, various eutectic mixtures have been studied, including Aluminum-Silicon, Copper-Nickel, and Calcium Chloride-Magnesium Chloride eutectics. [8]

Since then, researchers have continued to study various PCMs, including inorganic salts, organic materials, and composite materials. In recent years, nanomaterials, including graphene and carbon nanotubes, have been investigated as potential PCM additives to enhance thermal conductivity and stability. [8]

Despite the progress made in the development of PCM-based thermal energy storage systems, there are still many challenges to be addressed, including high costs and limited commercial availability. Nonetheless, PCMs have the potential to revolutionize various industrial processes and reduce energy consumption. [10]

In recent years, researchers have focused on developing new PCMs with improved properties, such as higher energy density and thermal conductivity. One such material is graphene oxide (GO), which has a high surface area and thermal conductivity, making it an ideal material for thermal energy storage applications. In 2016, a study reported the use of GO as a PCM in aluminum matrix composites. [10]

Chapter 3

phase transformation for heat storage

Phase transformation is a widely used method for heat storage, which involves using a phase change material (PCM) to store and release heat energy. In this work, we will discuss the principles of phase transformation for heat storage, the types of phase change materials used, and the various applications of this technology.

Principles of Phase Transformation for Heat Storage

Phase transformation for heat storage involves using a PCM, which can absorb or release large amounts of heat energy during a phase transition. The most used PCMs for heat storage are those that undergo a solid-liquid phase transition, such as paraffin wax, fatty acids, and salt hydrates. When heat is applied to the PCM, it melts and absorbs the energy as latent heat. Similarly, when the PCM is cooled, it solidifies, and releases stored energy. This process can be repeated many times without any significant degradation in the PCM. [8][6]

Types of Phase Change Materials

The most commonly used PCMs for heat storage include paraffin wax, fatty acids, and salt hydrates. Paraffin wax has a high latent heat of fusion and can store large amounts of energy. It is also non-toxic and widely available. Fatty acids, such as stearic acid, also have high latent heat of fusion and are used in many commercial applications. Salt hydrates, such as sodium sulfate decahydrate, have the advantage of being able to store energy at high temperatures. [6]

This Table [3.1] shows us some important metals with their melting temperature, thermal conductivity, and melting enthalpies.

The metal	Melting Temperature (°C)	Thermal conductivity (W/m.K)	Melting Enthalpies (KJ/mol)
Gallium (Ga)	29.76	40.6	5.59
Tin (Sn)	231.93	67	7.09
Zinc (Zn)	419.53	116	7.35
Cadmium (Cd)	321.07	97	6.21
Aluminum (Al)	660.32	237	10.71

Table 1 3.1: List of important metals in the range of melting temperatures (30-700°C)

In this work, the mentioned metals in Table 1.1 are going to be compared to choose the most suitable for this work according to the capability of accumulating thermal energy.

Metallic PCMs

Metals have high or low thermal conductivity and thermal stability for different applications that need high or low thermal properties, which make them attractive candidates for PCM applications. Some of the commonly used metallic PCMs are discussed below:

1) Gallium (Ga)[31]

has a low melting point of 29.76 °C, which makes it a suitable candidate for low-temperature thermal energy storage applications. However, gallium is expensive and has limited availability, which can make it less practical for large-scale applications. Gallium does not crystallize in any of the simple crystal structures. The stable phase under normal conditions is orthorhombic with 8 atoms in the conventional unit cell as shown in Figure (3.1). Within a unit cell, each atom has only one nearest neighbor (at a distance of 0.244 pm). [11][17]
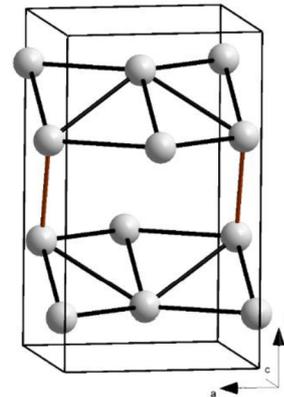
Figure 3.1: Gallium crystal [17]

2) Tin (Sn)[50]

Tin is a chemical element belonging to the carbon family, Group 14 (IV a) of the periodic table with atomic number 50, an atomic weight of 118.69, and a density of 7.7 Mg/m^3. It has a melting temperature of 232 °C. At room temperature β-Tin (white tin) has a tetragonal crystal structure with a basis of one tin atom per lattice point. Below 13.5 C Beta Tin is a high-temperature allotrope of tin that has a body-centered tetragonal crystal structure. The beta Tin lattice parameters are about b = 0.5835 nm and c = 0.3182 nm. [11]

We may use these lattice characteristics to compute the distance between the atoms because the beta Tin atoms are grouped in a regular lattice arrangement.

α-Tin (gray Tin) is the stable form and has the diamond cubic structure with a lattice parameter of 0.495 nm (shown in Figure 3.2). It is another metal that has been investigated for its potential as a phase change material (PCM). Tin has a melting point of 232 °C and a relatively high thermal conductivity of 67 W/m.K, which makes it a suitable candidate for high-temperature thermal energy storage applications.
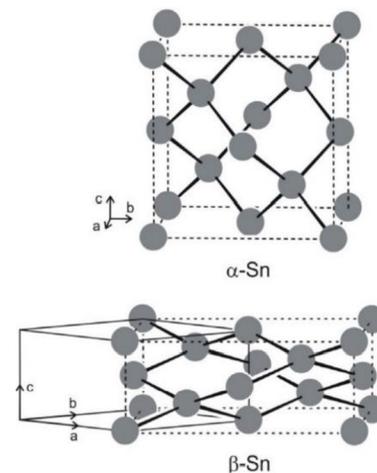

Figure 3.2: α and β Tin lattice [18]

3) Zinc (Zn)$_{30}$

Zinc is a metal in group II B of the periodic table with atomic number 30, an atomic weight of 65.37, and a density of 7.14 Mg/m3. It has a melting temperature of 419.5 °C. At room temperature, the metal has a hexagonal close-packed crystal structure (shown in Figure 3.3). The lattice parameters are a =0.266 nm, and c = 0.495 nm. [11]

It has a melting point of 419.5 °C and a high latent heat storage capacity, which makes it a potential candidate for high-temperature thermal energy storage applications. However, the thermal conductivity of zinc is relatively low 116 W/m.K, which can limit its heat transfer capabilities. [11]
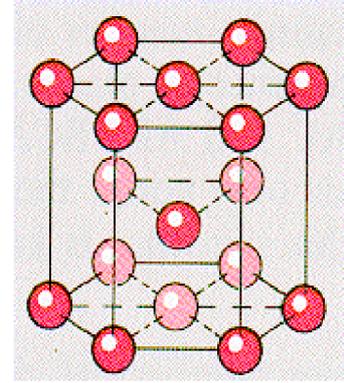
Figure 3.3: Zinc crystal lattice [11]

4) Cadmium (Cd)$_{48}$

Cadmium is a metal in group IIB of the periodic table with atomic number 48, an atomic weight of 112.40, and a density of 8.65 Mg/m3. Its melting point is 321 °C. At room temperature Cadmium has also a hexagonal close-packed crystal structure like Zinc has with a = 0.297 nm and c = 0.561 nm (shown in figure 3.4). [11]

It has a high latent heat storage capacity of 6.192 kJ/mol making it a potential candidate for low to medium-temperature thermal energy storage applications. However, cadmium is toxic and has limited availability, which can make it less practical for large-scale applications. [11]
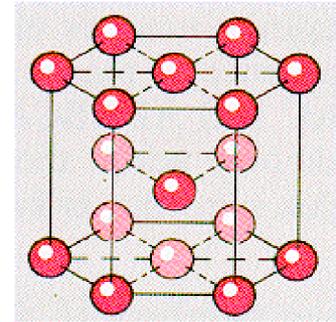

Figure 3.4: Cadmium crystal lattice [11]

5) Aluminum (Al)$_{13}$

Aluminum is a metal in group IIIA of the periodic table with atomic number 13, an atomic weight of 26.982, and a density of 2.70. It has a melting temperature of 660 °C. At room temperature, Aluminum has FCC (Face-Centered-Cubic) crystal structure with a basis of one Ni atom a=0.404 nm (shown in Figure 3.5). [11]

It has a high thermal conductivity of 273 W/m.K, which makes it a suitable candidate for PCM applications. Aluminum is used as a PCM in thermal energy storage systems for industrial applications, such as in the production of cement and steel. [11]
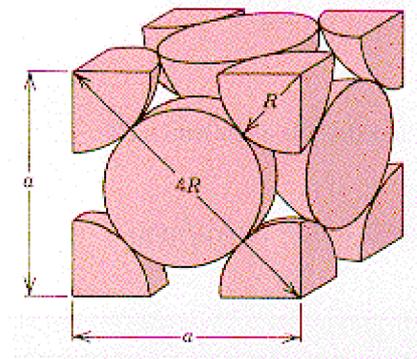

Figure 3.5: Aluminum crystal structure [11]

Chapter 4

Experiment

From Table 3.1, the metals that will be chosen as a binary system that is capable of accumulating thermal energy to be used as a thermal Energy storage system is an Al-Sn mixture by CIP (Cold Isostatic Pressing) this process will be briefly mentioned in the following context.

Cold Isostatic Pressing (CIP)

Cold Isostatic Pressing (CIP) is a manufacturing process that involves the application of pressure on a powder material using a liquid medium. The material is placed in a sealed container filled with a liquid, usually water or oil, and subjected to high pressure from all directions using an isostatic press. The pressure applied compresses the material and removes any voids, resulting in a more uniform and dense material.

CIP is commonly used in material science studies to produce samples with improved mechanical properties, such as strength, toughness, and wear resistance. The process can be used to create a wide range of materials, including ceramics, metals, and polymers.

This technology is specifically chosen because of the several advantages that fulfill the desired properties that are needed for the experiment.

There are several advantages of making the Al-Sn mixture by CIP (Cold Isostatic Pressing) and traditional alloy manufacturing methods:

Advantages of Cold Isostatic Pressing (CIP)

1) Uniformity:

The CIP process can produce a uniform mixture of the Al-Sn powder, resulting in a homogeneous microstructure and improved material properties, such as strength and wear resistance.

2) Near-Net Shape:

The CIP process allows to produce near-net-shaped parts, which reduces the need for additional machining or processing.

3) Improved Density:

The CIP process can achieve higher, resulting in improved mechanical properties, such as strength and wear resistance.

Instrumentation

1) **Microscopes**:

SEM (Scanning Electron Microscopy)

Samples were investigated by scanning electron microscopy (Carl Zeiss NTS GmbH, Oberkochen, Germany) shown in figure (4.3), equipped with an Analytical systems EDX system X-Max for analyzing the chemical composition (Oxford Instruments, High Wycombe, UK). EDS data were obtained at an acceleration voltage of 20 kV with a 60 μm aperture in high current mode using the AZtech software (Oxford Instruments, High Wycombe, UK). For material analysis was used point analysis was for local chemical composition measurement and area analysis for measuring the average chemical composition of the mixture. Further, EDS mapping was used.
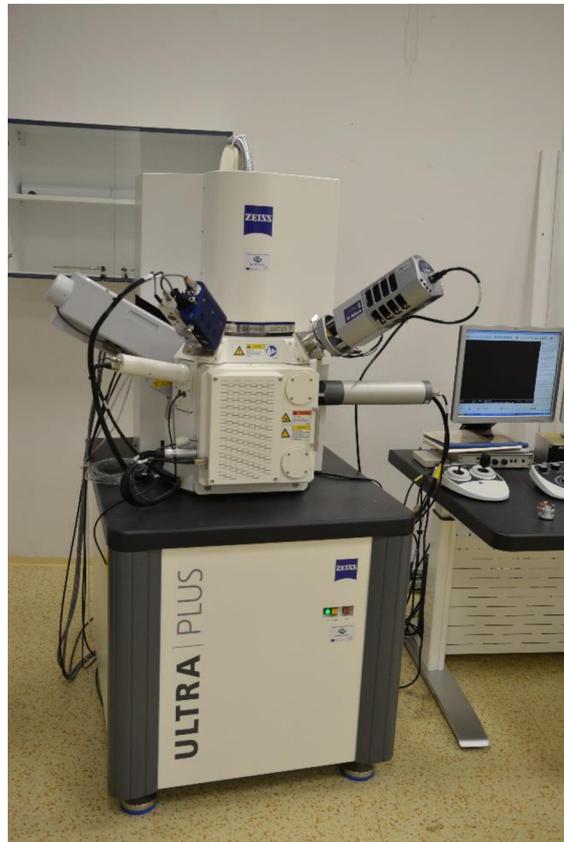
Figure 4.3: FSI, Material Science department lab, FEG-SEM ZEISS Ultra Plus

Differential Scanning Calorimetry (DSC)

The DSC analysis device principle is based on maintaining the same temperature of the standard and the reference sample (The device is shown in Figure 4.4). The samples are placed in separate forages. They are maintained by means of regulation of the input power of the fodder temperatures of both samples at the same level. The DSC curve is created using the difference in furnace power, which is then converted to heat. The record shows the change of supplied heat as a function of time.

DSC is a commonly employed method for studying the thermal behavior of materials by measuring the heat flow associated with physical and chemical processes during heating or cooling.

A differential scanning heat flux calorimeter was used for thermal analysis. It is a French device brand Setaram Instrumentation, model Setsys Evolution. The weighing sample was placed in a crucible made of corundum. The same cup, but it was empty used as a reference sample. After turning on the furnace, both cups were placed on thermocouples and then lowered into the furnace. It was then evacuated. After reaching a pressure of the order of 0.01 mbar, the argon supply to the chamber was opened. Unlike air, it does not react with metal and is not dangerous i.e., surface oxidation.
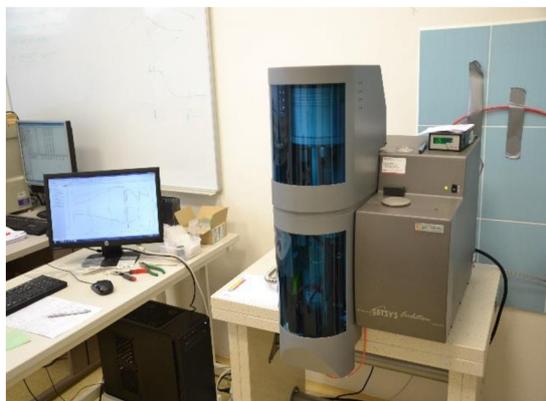

Figure 4.4: FSI, Material Science department lab, DSC analysis

Analysis of Al-Sn mixture

The mixture of Aluminum and Tin for the experiment consists of 80 wt% of Aluminum powder and 20 wt% of Tin powder. The images of powders that have been taken by SEM are shown in the following Figures (4.5 and 4.6) for Aluminum and Tin, respectively.


| 20 µm* | 10.1 mm | 20.00 kV | Signal A = SE2 | | 4 May 2023 | Photo No. = 7459 |
| | 200 X | | Width = 572.4 µm | Pixel Size = 558.9 nm | Al powder_05.tif | |
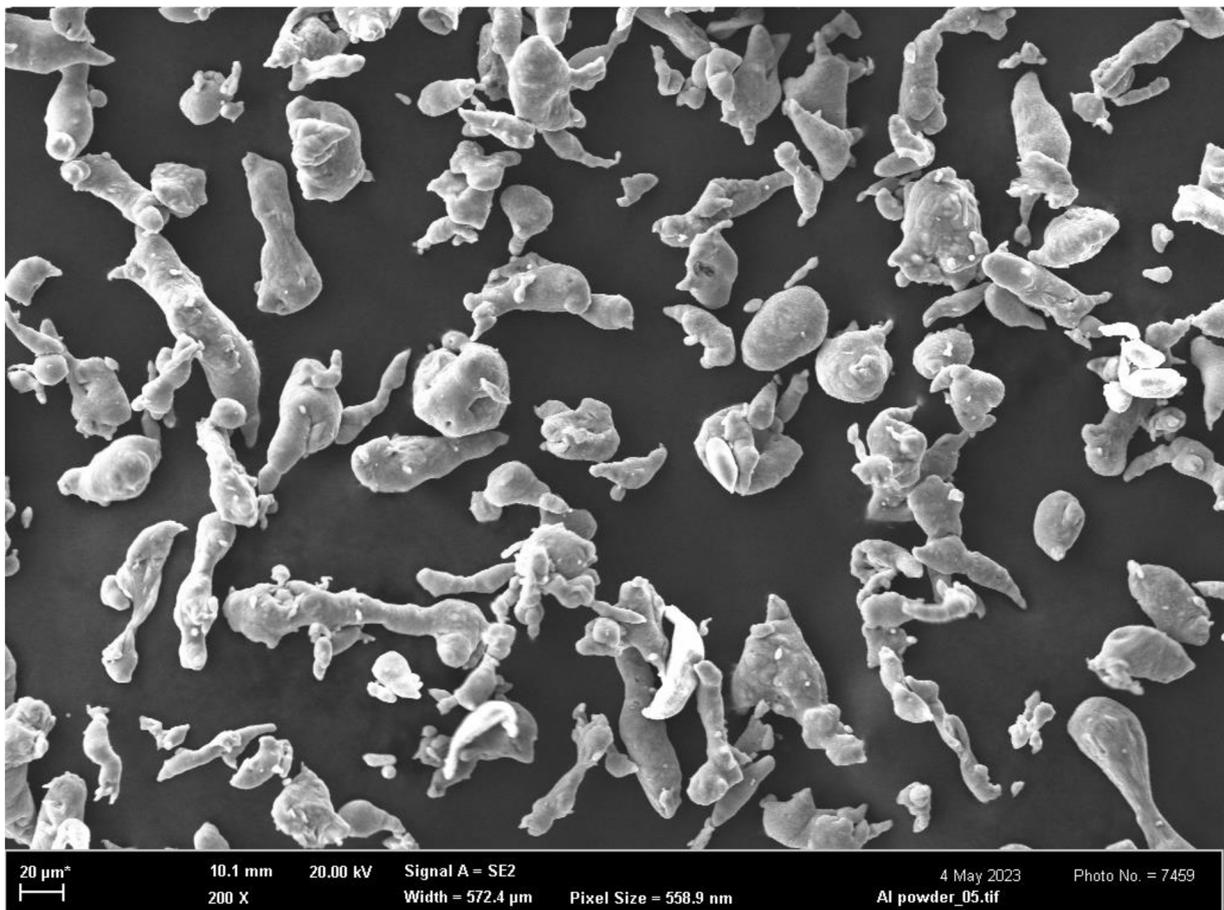
Figure 4.5: SEM image of Aluminum powder, magnification 200x, SE detector.
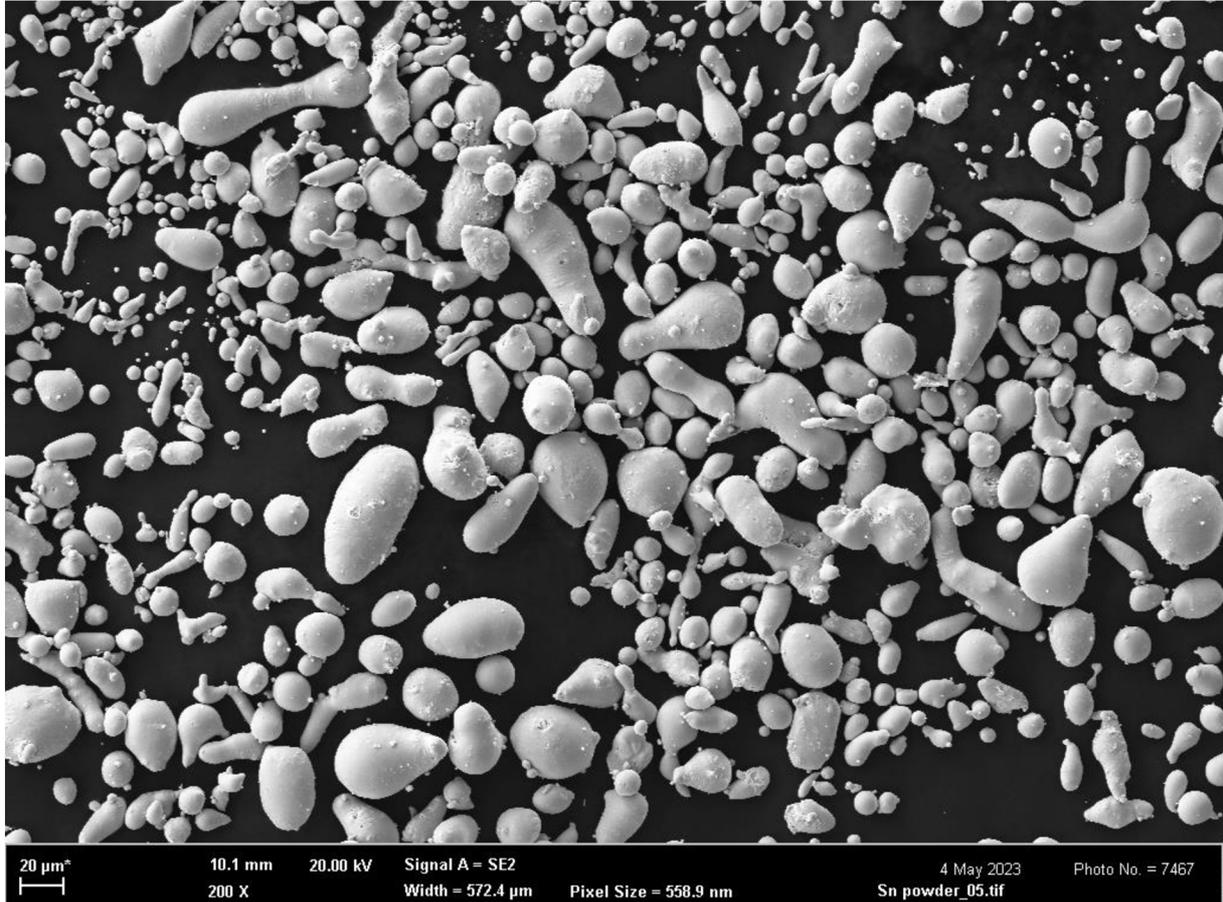
Figure 4.6: SEM image of Tin powder, magnification 200x, SE detector.

The given quantity of powder was placed in a plastic container and mixed in a rotary mixer for 24 hours. For better mixing of the powders, steel balls with a diameter of 10 mm were added to the mixture, which was subsequently removed from the mixture. A compact piece of material was created from the powders using the CIP method. The powders were pressed at a pressure of 500 MPa for 5 minutes.

Three types of samples were produced. The first type was the sample after CIP shown in Figure (4.7), the second type was the sample after one annealing cycle shown in Figure (4.8), and the last type was the sample that underwent three annealing cycles shown in Figure (4.9). The annealing cycle consists of heating to a temperature of 400°C with a duration of one hour and subsequent cooling in air. The effect of heating on the microstructure of the mixture will be observed.

To obtain the microstructure, the samples were ground on 500-2000 grit sandpaper, then polished using diamond polishing pastes with a maximum grain size of 3 µm and 1 µm. Chemical-mechanical polishing using a colloidal SiO_2 solution was chosen as the final treatment. With this procedure, surfaces suitable for imaging were obtained.

Figure 4.7: The sample after the CIP underwent 500 MPa for 5 minutes.

Figure 4.8: The sample after the grinding and polishing

Al-Sn Powders mixture after the CIP

After grinding, double polishing and preparing the sample shown in Figure 4.8 for scanning by the SEM we got the following results showing the Aluminum and Tin percentage in the sample (Light particles are Tin and darker particles are Aluminum). Figure 4.9 shows the SEM Image of the sample taken by BSE (Back Scatter Electron Detector) and Graph 4.1 shows EDS spectrum aquired from the grinded sample at low magnification. Table 4.1 shows average chemical composition of the Al-Sn mixture after CIP.the percentage of the mixture we obtain from the grinded sample.
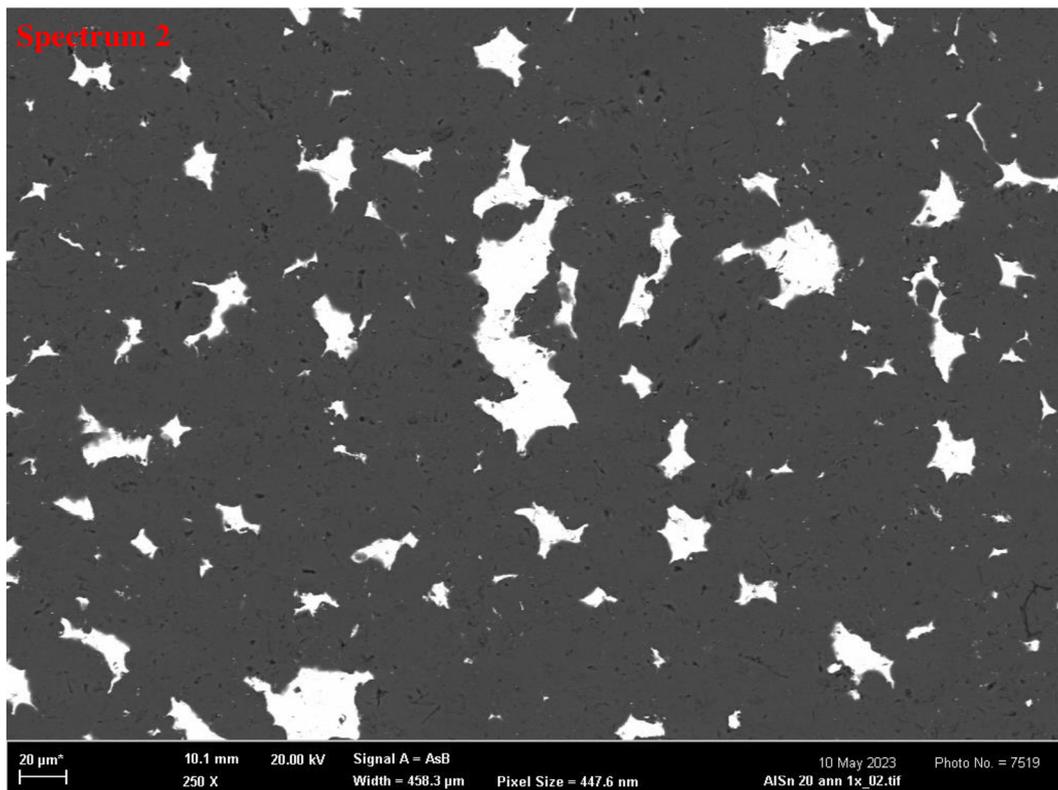
Figure 4.9: Sample after the grinding and double polishing, magnification 250x, BSE detector
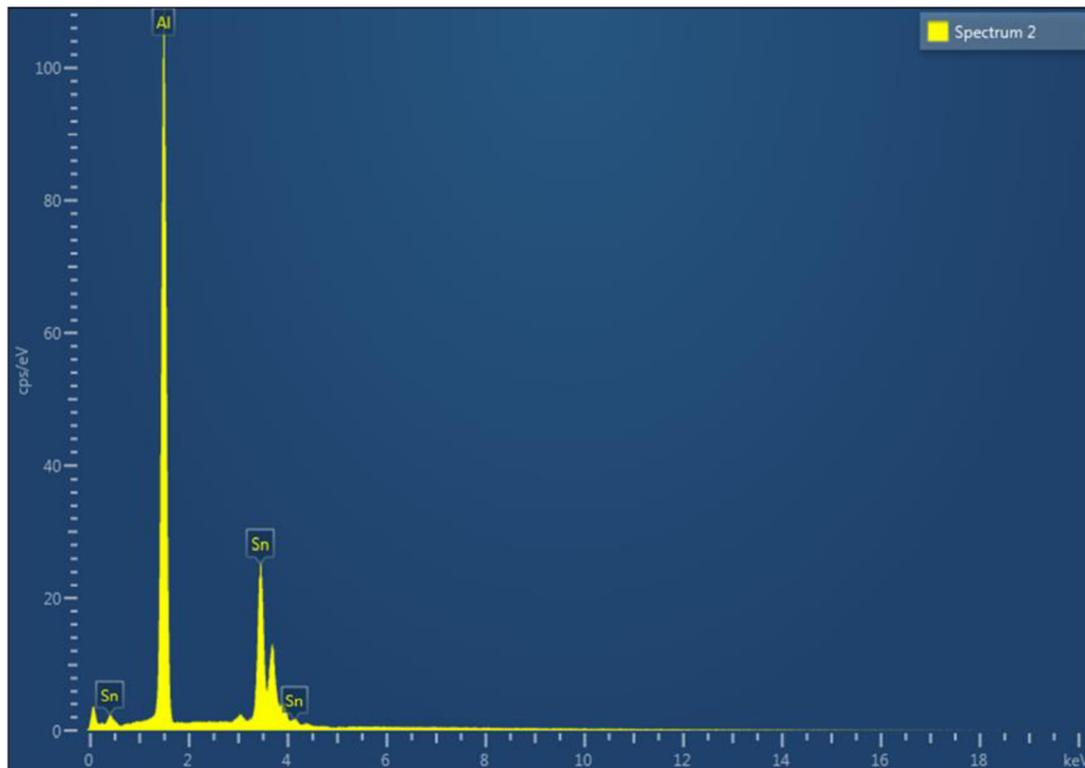
Graph 4.1:EDS Spectrum 2 after the CIP

Element	Wt%	Wt% Sigma	Atomic %
Aluminum	83.7	0.21	95.8
Tin	16.3	0.21	4.2
Total	100		100

Table 2 4.1: Average chemical composition of the Al-Sn mixture after CIP

The average composition of an Al-Sn alloy sample after the Cold Isostatic Pressing (CIP) process was determined using EDS analysis. The study was carried out on a sample area of about 4 mm2. The findings showed that the sample included 83.7 Wt% aluminum and 16.3 Wt% tin. These results were illustrated graphically in Graph (4.1), which indicated the average composition percentages of Aluminum and Tin in the sample

EDS Mapping of the Al-Sn sample

EDS mapping displays the spatial distribution of the chosen elements as we see in Figure (4.2), Aluminum is shown in blue on the left side of the mapping Image while Tin is shown in red on the right side of the mapping image.

Electron Image 12

Al Kα1

Sn Lα1
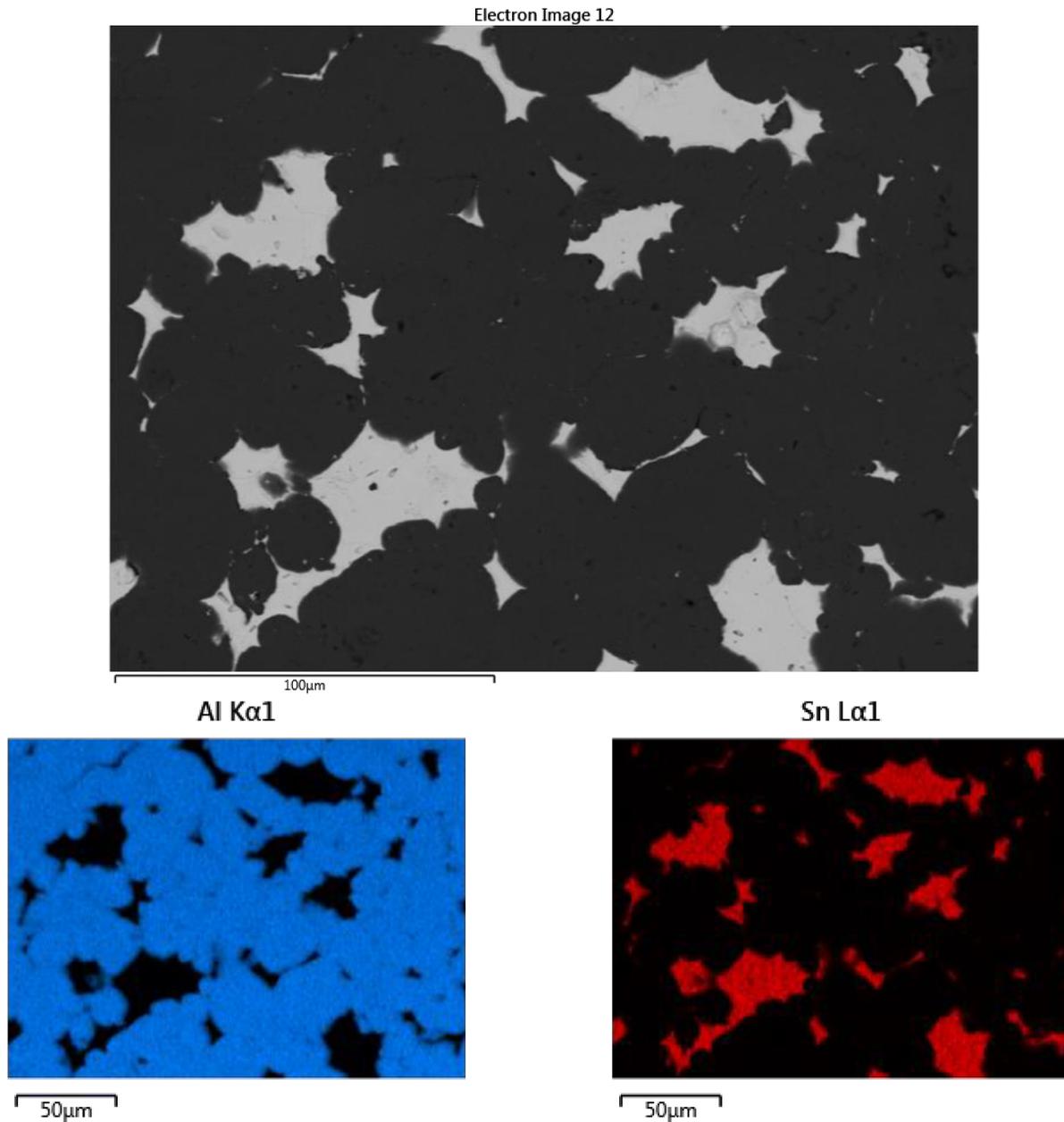
Figure 4.2: Mapping of the sample after the CIP

Second sample after annealing for one time.

After annealing the second sample for one time at temperature of 400 °C and allowing the sample to rest in the room temperature and observing the Tin grain shown in Figure (4.10) of the sample, we noted that some spectrums of the Tin grain are affected. Those effects are plotted by using AZtech software shown in Graph (4.2):
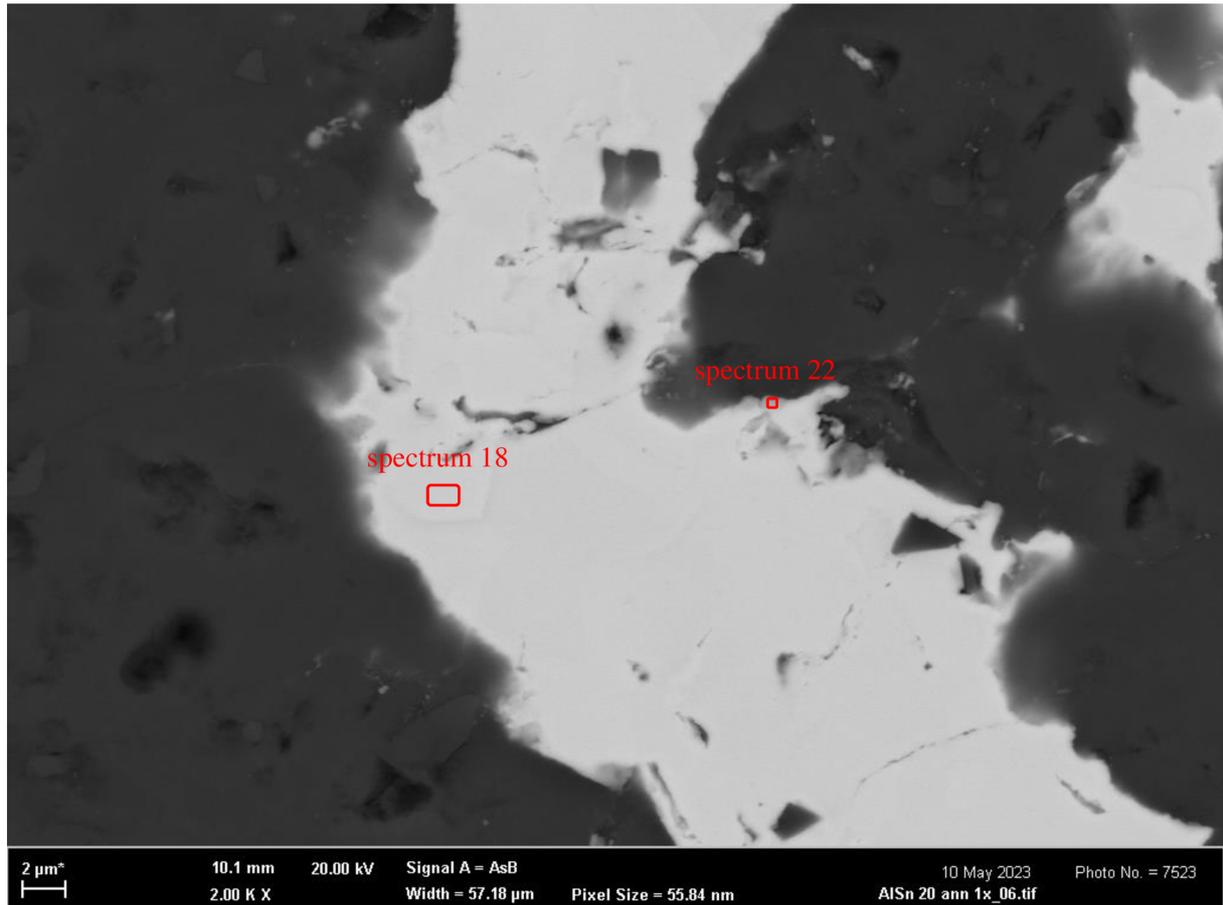
Figure 4.10: The sample after annealing of one-time at temperature of 400 °C, magnification 2000x, SE detector.

During the annealing at temperature 400 ℃, Tin particles melted. Some of the Aluminum particles which were in a direct contact with the Tin grain dissolved into the liquid Tin. After re-cooling, Initially Tin particles are not present in the mixture, but the solid solution of the Aluminum in the Tin is observed. From Table (4.2) the Aluminum measured content in the solid solution is 0.3 Wt%, and the remaining percentage is Tin.
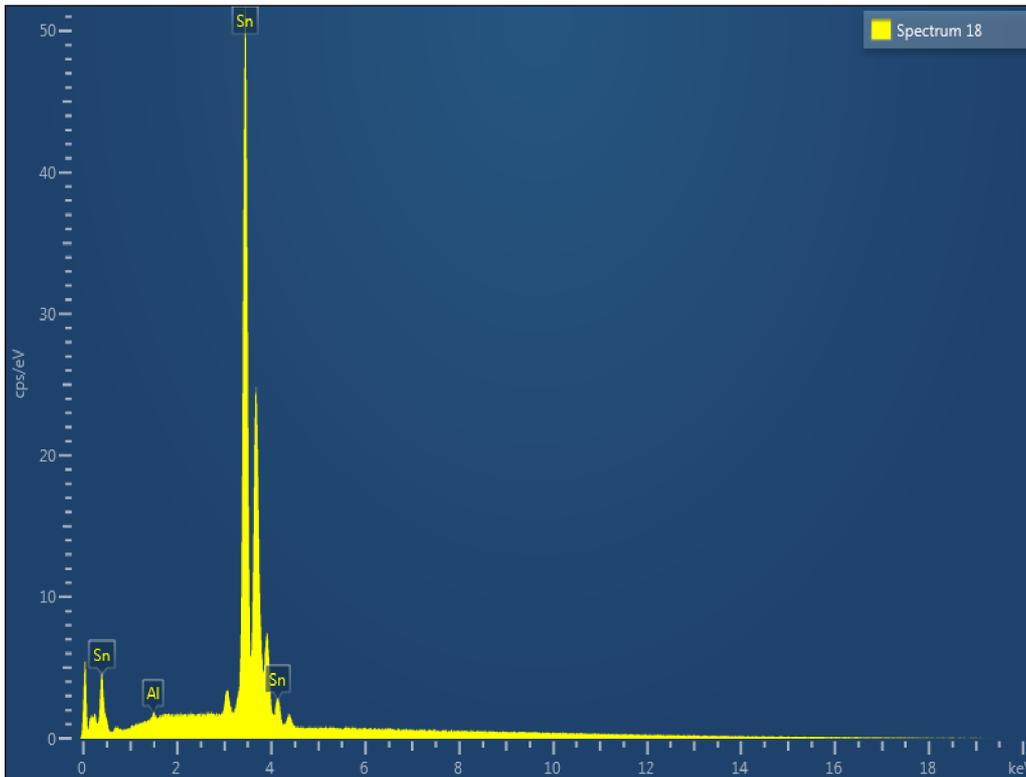

Graph (4.2): Spectrum 18 after annealing for one time up to 400 ℃

Element	Wt%	Wt% Sigma	Atomic %
Aluminum	0.3	0.06	1.5
Tin	99.7	0.06	98.5
Total	100		100

Table 3 4.2: Average chemical composition of Spectrum 18 after Annealing for one time.

During the annealing process of the sample in an oxygen-rich atmosphere, a solid solution comprising Tin and Aluminum undergoes partial oxidation. Subsequent spectral analysis confirms a heightened oxygen concentration at the examined location. From Table 4.3 the resulting oxide species is characterized by a composition consisting of 63.5 Wt% tin, 23 Wt% aluminum, and 13.5 Wt% oxygen.
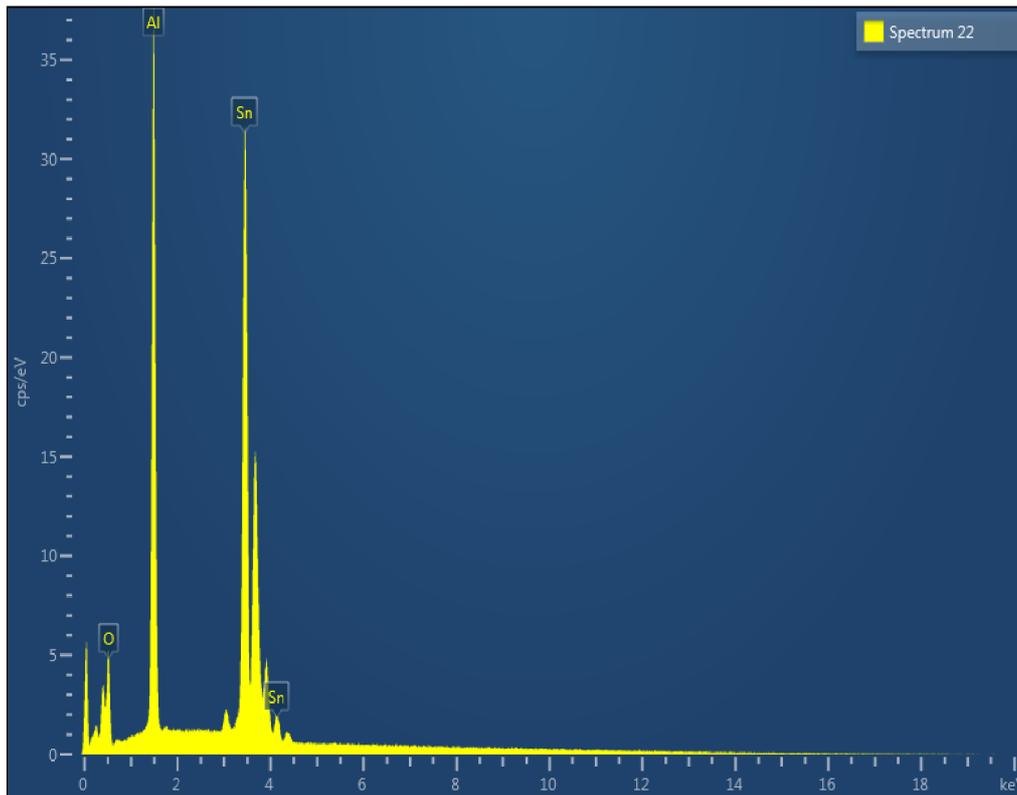
Graph (4.3): Spectrum 22 after annealing for one time up to 400 ℃

Element	Wt%	Wt% Sigma	Atomic %
Aluminum	23.0	0.15	38.2
Tin	63.5	0.26	24.0
Oxygen	13.5	0.29	37.8
Total	100		100

Table 4 4.3: Average chemical composition of Sprctrum 22 after Annealing for one time.

Following a single annealing cycle, the presence of oxide particles is observed to be minimal. However, it is postulated that with successive annealing cycles, the extent of oxide formation will progressively increase. The gradual accumulation of oxide particles over repeated cycles may have consequential implications for the material's intrinsic properties and subsequent behavior.

Third sample after annealing for three times.

After annealing the third sample for three times at temperature 400 °C and allowing the sample to rest in the room temperature and observing the Tin grain shown in Figure (4.11) After completing three annealing cycles, an increase in oxide contents is clearly visible in the sample, as can be seen in Figure (4.11). This is a BSE SEM image, and the oxides are visible as light gray areas. Figure (4.11) shows the EDS spectrum taken from this bright area. We noted that the chemical composition of the sample -some spectrums in the Tin grain- are affected as follows:
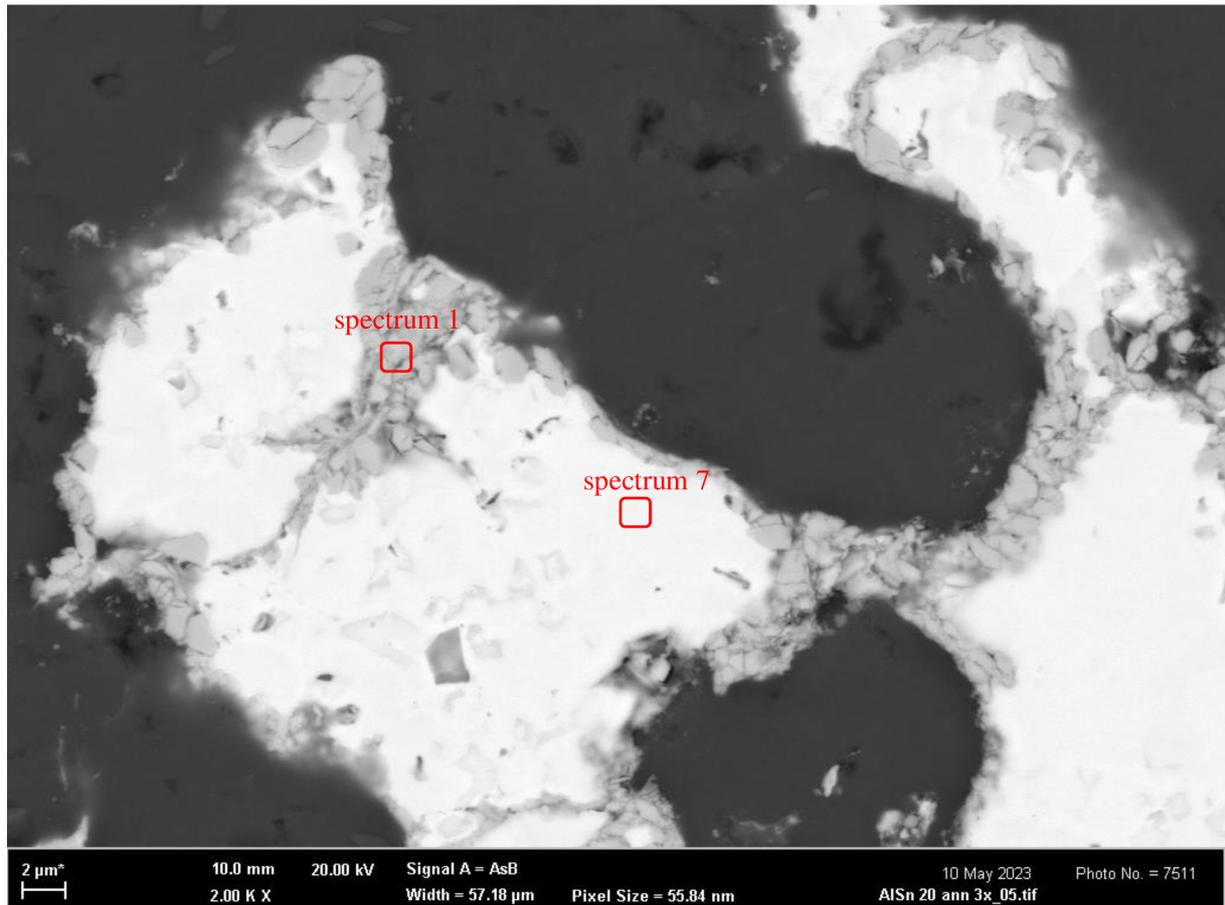
Figure 4.11: The sample after annealing of three times, 2000x, AsB detector.

Following the completion of three consecutive annealing cycles, a conspicuous augmentation in the oxide content becomes evident within the sample, as visually discerned from the BSE SEM image depicted in Figure 4.11. The image reveals distinct regions of light gray, indicative of the presence of oxides. Complementarily, Graph (4.4) presents the EDS spectrum extracted from the bright region of interest. From Table 4.4 for Spectrum 1 within the graph delineates the precise chemical composition of the analyzed domain, unveiling an aluminum concentration of 0.6 Wt%, tin concentration of 78.1 Wt%, and the remaining fraction attributed to oxidized particles. Those Oxidized Particles are from the Oxidized Tin, observing the Atomic % in Table 4.4 we noted that the ratio between the Tin atoms and the Oxygen atoms are approximately 70:30, respectively, which means that the formed Oxide is Tin Oxide Sn_7O_3, So the formed solid solution is Tin Oxide Sn_7O_3 and some Aluminum atoms that dissolved into the mixture.
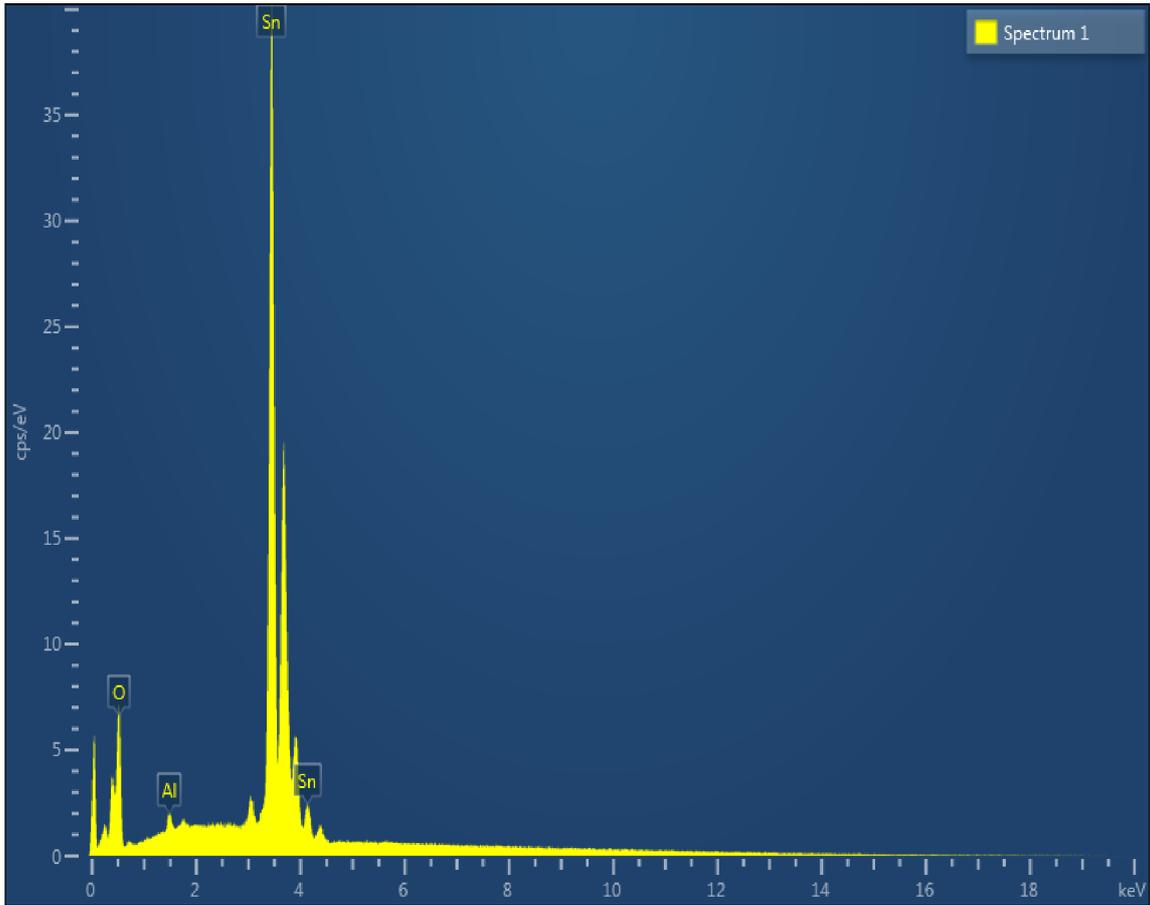
Graph (4.4): Spectrum 1 after annealing for three times up to 400 ℃

Element	Wt%	Wt% Sigma	Atomic %
Aluminum	0.6	0.05	1.2
Tin	78.1	0.31	32.8
Oxygen	21.3	0.31	66.0
Total	100		100

Table 5 4.4: Average chemical composition of Spectrum 1 after Annealing for three times.

These findings unequivocally substantiate the progressive accretion of oxides within the sample upon successive annealing cycles on the air, as affirmed by both visual observation and elemental analysis. The observed increase in oxide contents is suggestive of ongoing oxidation processes, with the accumulation of oxidized species manifesting in the form of discernible gray regions within the sample. Such observations are instrumental in comprehending the evolution of the material's composition and properties under the influence of repeated annealing treatments.

The analysis of Spectrum 7 revealed a conspicuous absence of oxygen, which can be ascribed to its location within the Tin grain. This finding implies the presence of a localized oxygen-free environment, highlighting the potential influence of the grain's microstructure on the distribution of oxygen within the sample (Far from the Tin borders).

Furthermore, a detailed examination of the composition was facilitated by Graph (4.5). The graph illustrated that Spectrum 7 exhibited a nominal content of 0.3 Wt% Aluminum, indicating a minor presence of this element in the sample. Conversely, the dominant constituent was Tin, constituting a substantial majority with a composition of 99.7 Wt%. This overwhelming Tin content underscores its significant role in shaping the material's properties and behavior within Spectrum 7.
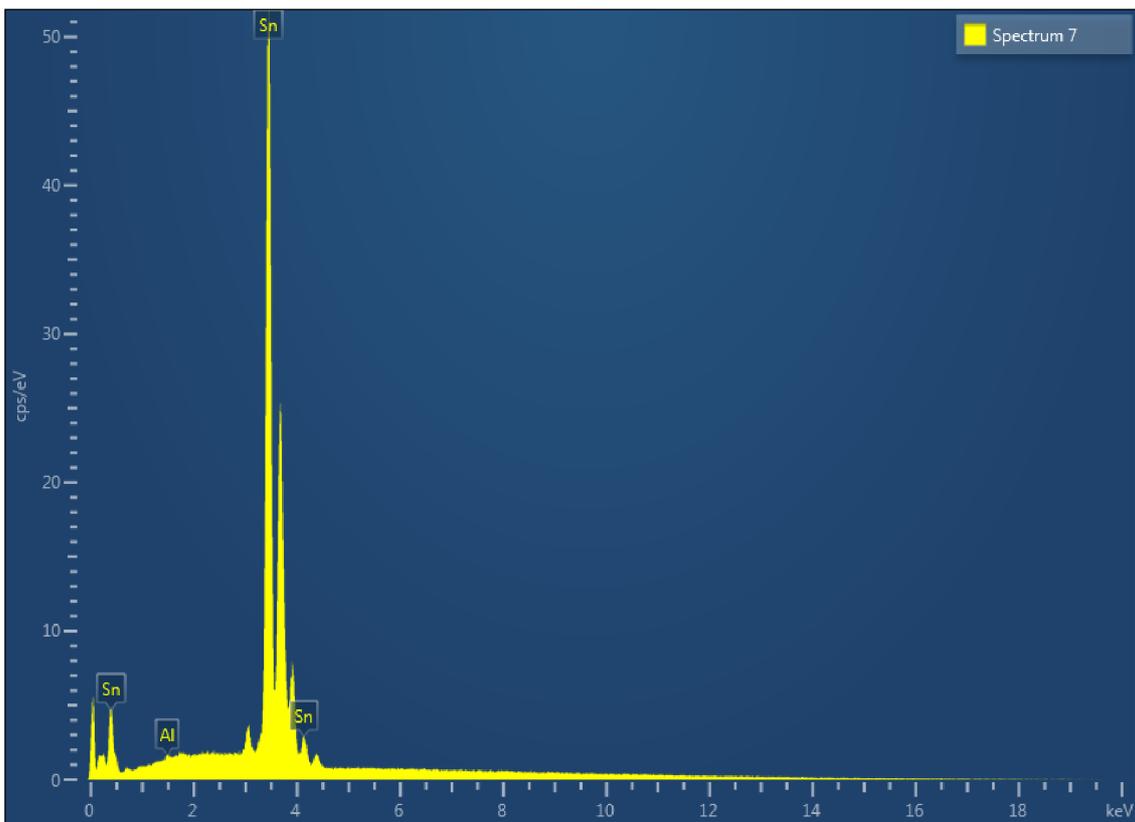
Graph (4.5): Spectrum 7 after annealing for three times up to 400 ℃

Element	Wt%	Wt% Sigma	Atomic %
Aluminum	0.3	0.06	1.0
Tin	99.7	0.06	99.0
Total	100		100

Table 6 4.5: Average chemical composition of Spectrum 7 after Annealing for three times.

DSC Diagrams Analysis

DSC Diagram for the mixture after annealing for one time.

Graph 4.6 shows the DSC diagram for the first annealing cycle of our Al-Sn sample, the X-axis represents temperature in °C, and Y-axis represents the Heat Flow in $\mu V/g$.

The red curve has several important temperatures and transitions were detected from Graph (4.6) for the melting of Tin. The onset temperature for the melting process was discovered to be approximately 231 °C, indicating the point at which the Tin begins to shift from a solid to a liquid form. The offset temperature is approximately 236 °C which indicates that the melting process has been completed. The amount of heat absorbed by the sample is 41500 $mV.s/mg$.

The blue curve also has several important temperatures and transitions were investigated from Graph (4.6) for the solidification of the Tin. The onset temperature of the solidification process was noted to be approximately 210 °C, indicating the temperature which the Tin starts to crystallize -shifting from liquid phase to the solid phase-. The offset temperature was discovered to be approximately 208 °C which denote the intersection of the peak with the base line. The amount of heat rejected from the sample due to the solidification process is -40324 $mV.s/mg$.
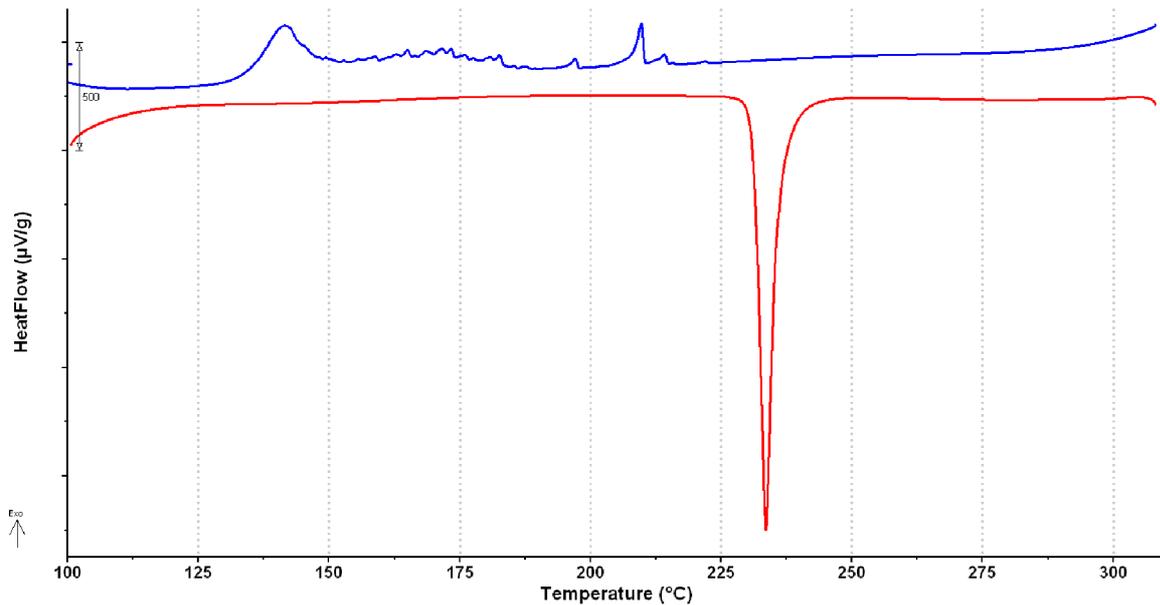
Graph 4.6: DSC diagram for the annealing process for one time

In Graph (4.6), the primary focus lies on elucidating the initial cycle of melting, specifically encompassing the Tin particle's initial phase of fusion. A comparative analysis can be conducted by comparing the melting temperature of Tin, quantified at 231.93°C, with the actual melting temperature in our mixture. Notably, within this particular mixture, the melting point of pure Tin exhibits remarkable proximity to the real melting temperature.

In the context of alloy melting, a discernible temperature deviation is anticipated. This deviation stems from the intrinsic requirement of elevating the temperature beyond the melting threshold, thereby ensuring the provision of adequate energy for instigating the melting phenomenon. Consequently, this temperature shift during alloy melting can be regarded as a normative occurrence, as it arises from the necessity to surmount the melting temperature, thus facilitating the requisite energy transfer for the melting process.

Conversely, during the process of crystallization, a discernible degree of undercooling assumes a crucial role. This undercooling phenomenon is visually discerned through the portrayal of the blue curve within the accompanying graph. The observed manifestation of undercooling substantiates the commencement of crystallization at a temperature lower than the documented value of the tabulated melting (crystallization) temperature. The indispensability of undercooling resides in its capacity to invoke and enable the formation of desired crystal structures within the alloy.

The temperature ranges and transitions give essential information for understanding the melting and solidification characteristics of Tin. The closeness of the melting start and offset temperatures shows that Tin has a small melting range. The emergence of the eutectic system is exclusively observed during the crystallization process. Initially, tin undergoes a phase of melting, accompanied by the dissolution of a fraction of aluminum within the molten tin matrix. Subsequently, as the molten mixture proceeds toward crystallization, a notable phenomenon unfolds - the formation of a distinct eutectic structure. This distinctive eutectic structure arises as a consequence of the specific composition and cooling conditions prevalent during the crystallization stage, which facilitate the organization of tin and aluminum atoms into a characteristic pattern that defines the eutectic phase. Furthermore, the solidification onset and offset temperatures indicate that solidification occurs across a greater temperature range than melting.

**DSC Diagram for the mixture after annealing for five times.**

The DSC (Differential Scanning Calorimetry) analysis was performed on an Al-Sn mixture that underwent annealing five times up to a temperature of 400 °C the following Graphs (4.7, 4.8, 4.9 and 4.10) show the phases that the mixture undergoes during the five cycles. Each curve on the Graph (4.7) shows a melting cycle and its corresponding cooling cycle in Graph (4.9) that were obtained for the cooling process of the mixture back to room temperature.
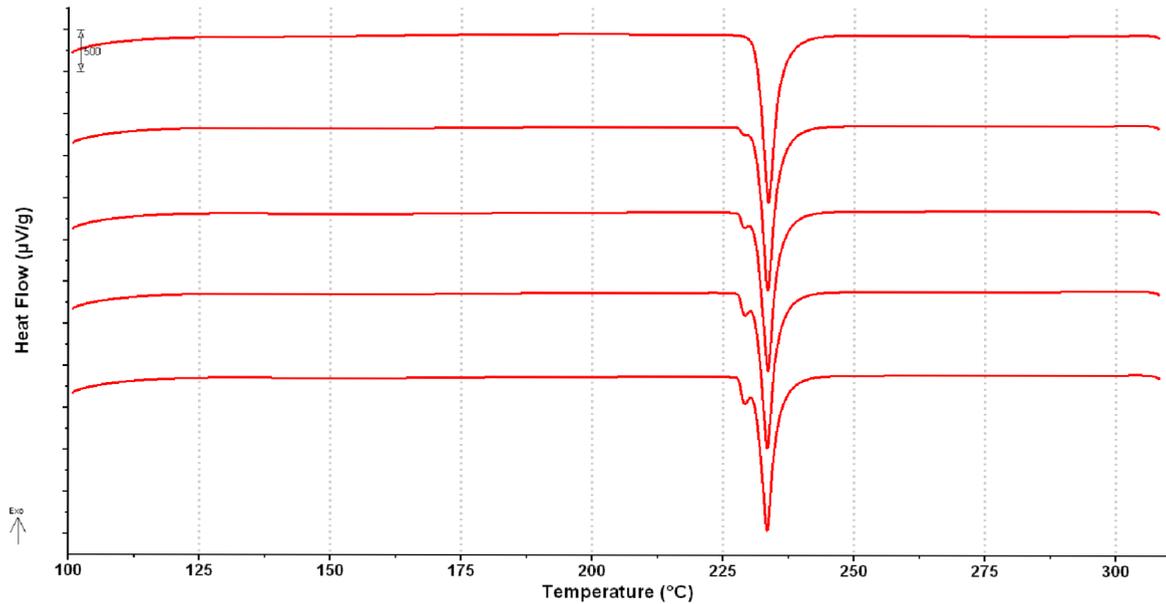

Graph 4.7: DSC diagram for the heating process of the sample undergone annealing five times.
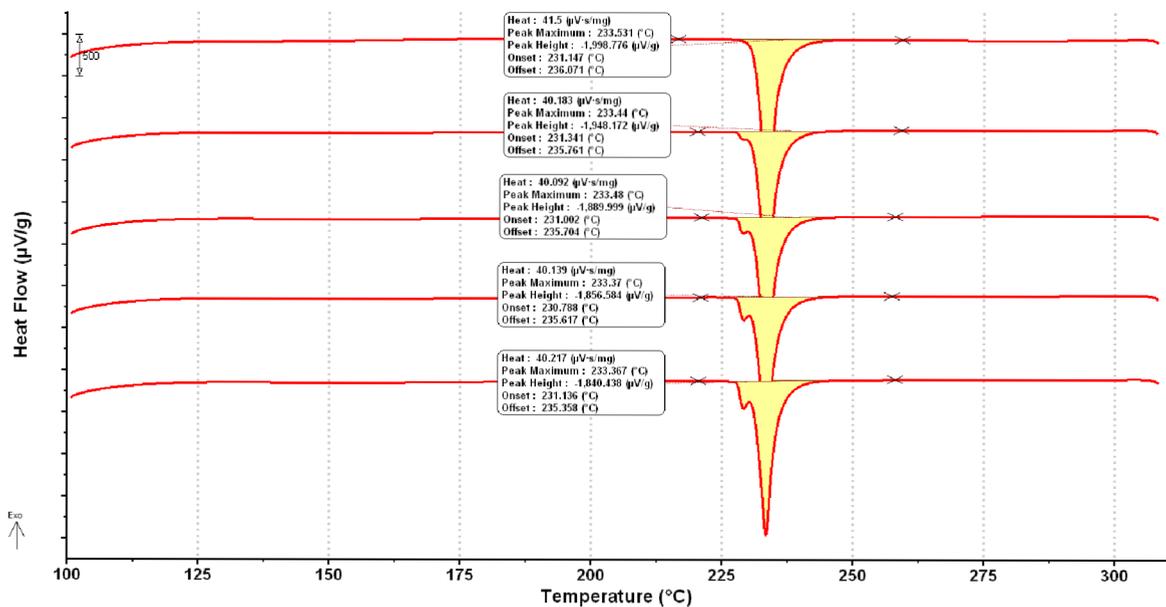
Graph 4.8: DSC diagram for the heating process of the sample undergone annealing five times, heat absorbed.
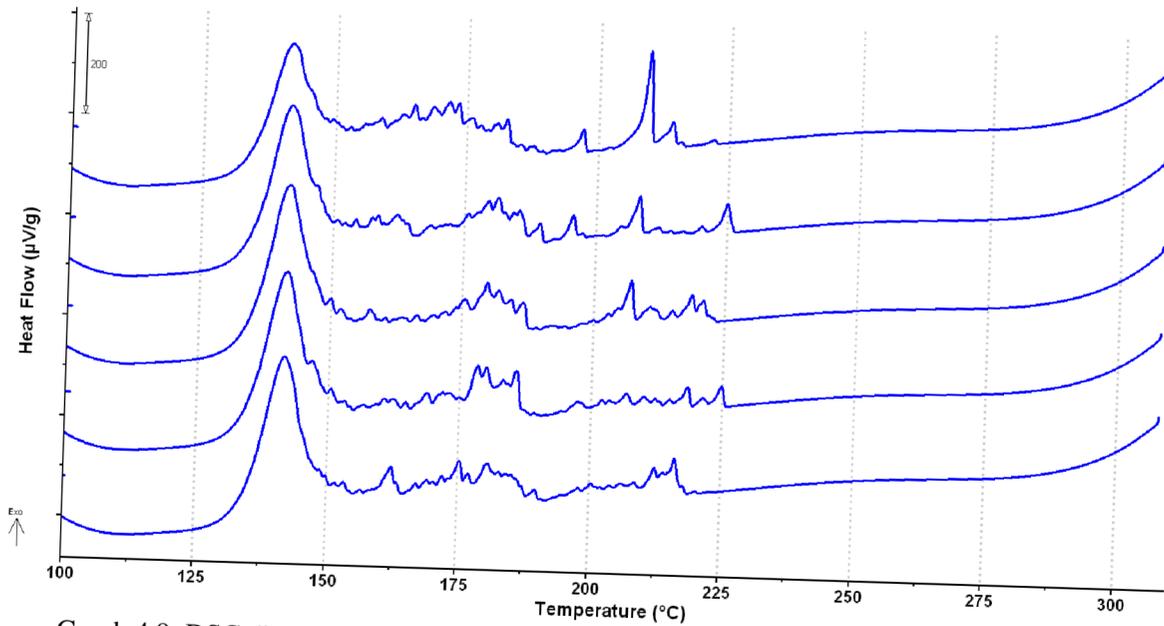
Graph 4.9: DSC diagram for the cooling process of the sample undergone annealing five times.
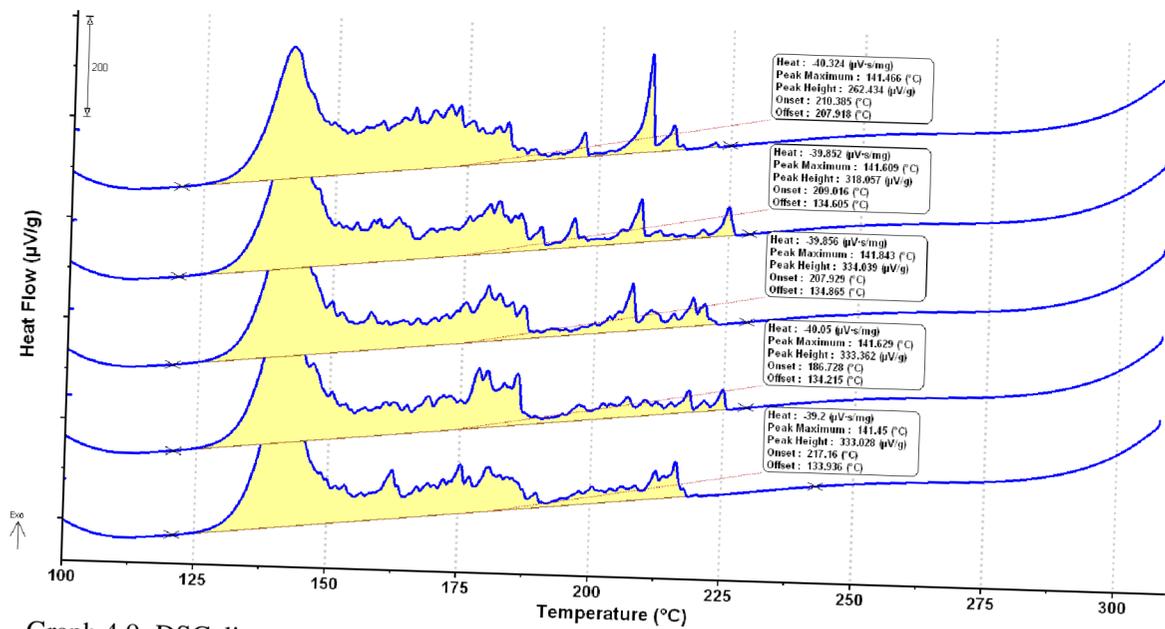
Graph 4.9: DSC diagram for the cooling process of the sample undergone annealing five times, heat rejection.

The DSC diagrams revealed consistent thermal behavior across the heating cycles. The integrated heat flow, which represents the energy absorbed per unit mass during the heating process, exhibited a relatively stable trend. The peak maximum, indicating the temperature at which the peak of the DSC curve occurs, showed minimal variation between cycles. Furthermore, the onset and offset temperatures, representing the beginning and end of endothermic reaction, respectively, demonstrated consistent values across the cycles.

The observed endothermic reactions suggest the occurrence of heat-absorbing processes within the Al-Sn mixture during heating. The magnitudes of the peak heights, which represent the extent of the endothermic reactions, varied slightly between cycles, indicating potential differences in the intensity or nature of these reactions.

Comparative analysis was performed between the cooling cycles and the previously discussed heating cycles to examine the similarities and differences in the thermal behavior of the Al-Sn mixture.

The cooling cycles exhibited negative values for the integrated heat flow, indicating heat release during the cooling process. These values were in the range of 40300 to 39200 mV.s/mg as the reaction is exothermic, similar to the heat values observed during the heating cycles which has a range of 41500 to 40200 mV.s/mg as the reaction is endothermic. This suggests consistent energy dissipation characteristics between heating and cooling and minimal heat losses between each heating cycle and its corresponding cooling cycle.

Moreover, variations were observed in the onset and offset temperatures between the cooling and heating cycles. These temperatures represent the initiation and cessation points of thermal events. The disparities in these values signify potential differences in the occurrence and duration of thermal phenomena during the cooling process compared to the heating process.

The DSC analysis of the Al-Sn mixture by CIP indicates consistent thermal behavior between the heating and cooling cycles. However, differences were observed in the nature of thermal reactions, as indicated by the positive exothermic peaks during cooling and negative endothermic peaks during heating. The variations in onset and offset temperatures further highlight distinctions in the thermal processes between heating and cooling. Further investigations are warranted to elucidate the specific mechanisms underlying these thermal phenomena and their implications for the material's properties and characteristics.

Conclusion

In this thesis we were making a Heat Storage system using PCMs (Phase Change Materials) that has the following parameters:

- Storing thermal energy in an efficient way.
- Having high storage density and isothermal nature.
- System that have the ability to absorb a large amount of thermal energy, without increasing the temperature of the system itself.

In this study, an investigation was conducted on a group of metals with melting temperatures ranging from 30 to 700 °C. The objective was to identify an appropriate binary system based on the specified parameters. Through careful analysis, it was determined that an Aluminum and Tin mixture, with a ratio of 80:20, exhibited favorable characteristics for the desired experiments.

To ensure a homogeneous distribution of the metal particles within the sample, a powder mixture of Aluminum and Tin, adhering to the aforementioned ratio, was subjected to thorough mixing in a rotary mixer for a duration of 24 hours. Following this step, a compact material specimen was prepared utilizing the Cold Isostatic Pressing (CIP) technique. The powders were compressed under a pressure of 500 MPa for a period of 5 minutes, facilitating the creation of a consolidated structure.

Subsequently, three samples were derived from the prepared specimen to enable further investigation. The initial sample was subjected to analysis using Scanning Electron Microscopy (SEM) to determine the average chemical composition. The obtained results revealed a slight disparity in the composition, with Aluminum accounting for 83.7 Wt% and Tin comprising 16.3 Wt% and that's because the mixing of the mixture was not perfect and there was probably uneven mixing of the mixture.

For the second sample, an annealing process was conducted at a temperature of 400 °C, followed by gradual cooling to room temperature. SEM imaging was then employed to examine specific regions of interest within the Tin grain. Notably, the presence of various shades of gray in the SEM images was deemed significant, as they indicated potential variations in the composition of the Tin grain. One particular spectrum captured during the analysis exhibited the presence of oxidized particles, which can be attributed to the Oxygen-rich environment employed during the annealing process. Moreover, this spectrum demonstrated a notable concentration of Aluminum, primarily originating from the boundaries of the Tin grain. Conversely, another spectrum exhibited minimal Aluminum content and was predominantly composed of pure Tin.

In the case of the third sample, a series of three annealing cycles at 400 °C, each followed by cooling to room temperature, were employed. Attention was specifically directed towards distinctive regions within the Tin grain. An intriguing observation emerged from one of the chosen spectra, revealing a relatively high percentage of oxidized particles. Further analysis of this spectrum unveiled an approximate atomic ratio of 70:30 between Tin and Oxygen, thereby indicating the presence of Tin Oxide (Sn_7O_3). On the other hand, the alternative spectrum displayed a marginal Aluminum content of 0.3 Wt% and predominantly consisted of pure Tin.

The annealing process conducted at elevated temperatures induced the formation of an oxide layer on the outer surfaces of the Al-Sn sample. This phenomenon was observed following both single and triple annealing cycles, suggesting the persistent occurrence of surface oxidation throughout successive thermal treatments or by coating the material with a special type of coating to prevent the chemical reaction between the sample and the surrounding environment.

The annealing procedure involved subjecting the Al-Sn sample to controlled thermal exposure to promote structural relaxation and induce desirable microstructural modifications. Consequently, the emergence of the oxide layer on the sample's outer surfaces signifies an oxidative reaction that transpired between the material and the atmosphere containing Oxygen during annealing.

The thermal behavior of a material was investigated using Differential Scanning Calorimetry (DSC) to analyze the solidification and melting processes of Tin. Two samples were studied; the first sample underwent a single annealing process up to 400°C, while the second sample underwent five cycles of annealing at the same temperature.

DSC diagrams were obtained to assess the heat flow characteristics as a function of temperature. The DSC diagram for the first sample revealed that approximately 41500 mV.s/mg of heat was absorbed during the Tin melting process. Notably, the onset temperature during heating was observed to be around 231°C, indicating a phase transition of Tin from the solid to the liquid state occurring approximately at the same melting temperature of pure Tin.

Furthermore, approximately 40300 mV.s/mg of heat was rejected during the solidification process of Tin, which closely exceeded the quantity of heat used during the melting process (41500 mV.s/mg). This implies that heat transfer losses during solidification were rather minor, estimated to be roughly 1,200 mV.s/mg.

DSC analysis performed on a sample subjected to repeated annealing cycles. The DSC diagram of the annealed sample, which underwent five annealing cycles, reveals slight discrepancies in thermal storage, characterized by varying heat absorption, across consecutive cycles. Specifically, the magnitude of heat absorbed for the Tin's melting process diminishes from 41500 mV.s/mg during the initial annealing cycle to approximately 40200 mV.s/mg in subsequent cycles.

Moreover, the cooling cycles demonstrate subtle divergences in the range of heat release observed after cooling the sample to ambient temperature throughout the annealing cycles. The amount of heat released during the first cycle is estimated to be around 40300 mV.s/mg, whereas in the final cycle, it approximates 39200 mV.s/mg. Consequently, this implies that the sample exhibits a relatively consistent rate of heat dissipation across successive cycles.

The aforementioned findings shed light on the variances encountered in thermal behavior, encompassing heat absorption and heat release, throughout the annealing and cooling cycles of the analyzed sample. These observations contribute to a deeper understanding of the sample's thermal characteristics and offer insights into the stability and reproducibility of its heat-related properties during the examined annealing processes.

Based on these results, the material displays excellent thermal energy storage since heat transfer losses during solidification are small. This capability is important for applications that require efficient heat management and energy storage systems.

Table of Figures:

List of Tables:

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