Performance Enhancement of Thermal Interface Materials

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Session 2018-20

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A Thesis Submitted to the US-Pakistan Center for Advanced Studies in Energy in partial fulfillment of the requirements for the degree of

MASTERS of SCIENCE in

Thermal Energy Engineering

US-Pakistan Center for Advanced Studies in Energy (USPCAS-E)

National University of Sciences and Technology (NUST)

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May 2022

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Acknowledgement

At the end of this thesis, I would like to express my gratitude towards all the individuals that without their help I would not have been able to complete this work. It is only due to their contribution of time, knowledge and moral support that helped me complete this research and I thank them for that.

My supervisor, Dr Mariam Mahmood, you helped me from the start when I only had an idea in mind, and you helped me narrow it down to a very effective research idea. And helped me understand the essence of research and to become an effective researcher. I appreciate the liberty you gave me to discover my own route, your knowledgeable advice and help when it was necessary. It was invaluable throughout all the work phases.

I would also like to take moment to thank Dr Abasin Ulasyar for guiding me to complete the electrical portion and being very helpful during all the electrical testing phase of our material. Dr Sehar Shakir for helping me navigate through the material selection process with her extensive knowledge. Dr Majid Ali for helping me with study of thermal parameters in detail. And Dr Anis ur Rehman for being so accommodating and helpful during performing the test for thermal parameters despite being from another institute. I am proud and honored that I was given an opportunity to work under the guidance of such amazing guidance committee.

Thank you

Ali Hamza

Dedication

I would like to dedicate my thesis to my beloved parents, who have been a constant source of continuous moral, spiritual and emotional support. My colleagues that helped with any obstacles I experienced during my research work. My teachers that helped me develop a sense of responsibility and guiding me throughout the research.

To my siblings, mentor and friends who shared their words of advice and encouragement to help me finish this study.

Abstract

The need of high performance miniaturized electronic devices to support the requirements of the industry 4.0 has created an increased demand for an efficient thermal management solution for smooth operations of metal oxide semiconductor field effect transistor (MOSFET). The main objective of this research is to enhance the thermal conductivity and viscosity of gallium-based thermal interface material (TIM) by adding tungsten microparticles to make it suitable for use in power electronics application. The gallium was first stirred to improve its wettability followed by addition of tungsten microparticles. After that, it was then passed through sonicator at 40 kHz to ensure equal dispersion of particles. Samples were prepared with three different configurations by concentration of tungsten. Different analytical techniques like Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) were utilized to analyze surface morphology, composition, and topography. Thermal constant analyzer and Boost circuit were used for thermal and electrical characterization respectively.

Upon oxidation the Thermal conductivity of Gallium drops from 29.3 W $m^{-1}K^{-1}$to 13.1 W $m^{-1}K^{-1}$. Our results present that 10 wt.% addition of tungsten can increase the thermal conductivity of gallium back from 13.1 to 22.82 W $m^{-1}K^{-1}$at room temperature which amounts to 74.2% overall increase in thermal conductivity. Also, when tested the material on Boost circuit, it was observed that could further push the frequency of MOSFET IRF3808 up to 20 Hz as compared to conventional TIM. A clear difference was observed between pure Gallium and Thermal interface material in terms of viscosity and fluidity. While gallium was highly fluid in its pure form, the TIM was in a semi solid state and there was no fluidity. During the electrical testing it was observed that the material did not leak out even when the temperatures raised as high as 308 Celsius. Strongly indicating that tungsten can be added to gallium to increase its thermal conductivity and viscosity and make it suitable to use as TIM for power electronics application.

Keywords: Electronics, thermal management, thermal interface material, thermal conductivity, viscosity

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1. Ali Hamza, Mariam Mahmood, Abasin Ulasyar, Sehar Shakir, Naveed Ahmed **“Thermal performance enhancement of Gallium based thermal interface materials’’**.

   1st International Conference on Engineering and Applied Natural Sciences (ICEANS 2022). Article No 494, May 12, 2022

List of abbreviations

Ga	Gallium
TIM	Thermal Interface Material
W	Tungsten
k	Thermal conductivity (W/m.K)
μ	Viscosity (N.s/m^2)
CMOS	Complementary metal-oxide Semiconductor
KHz	Kilohertz-Unit of frequency
SEM	Scanning Electron Microscopy
AFM	Atomic Force Microscopy
Wt%	Percentage by weight
MOSFET	Metal oxide semiconductor field effect transistor
R_{CO1}	Contact resistance 1
R_{CO2}	Contract resistance 2
R_{TIM}	Thermal interface material resistance
BN	Boron Nitride
AlN	Aluminum Nitride
CNT	Carbon nanotubes
LMTA	Low Melting Temperature Alloys
LED	Light Emitting Diode
CAS	CAS Registry is a collection of disclosed chemical substance information
μm	Micrometer-Unit of measurement
^{o}C	Celsius-Unit of temperature
Ga-based TIM	Gallium based Thermal interface material
Nb	Niobium
Mo	Molybdenum
Re	Rhenium

ρ	Density
C_p	Specific heat capacity
nm	Nanometer-unit of measurement
Torr	Unit of pressure
mm	Millimeter-unit of measurement
V_{in}	Input voltage
C	Capacitor
L	Inductor
R	Resistance

Chapter 1: Introduction

1.1 Overview of research

With the recent increase in performance of electronics, improved thermal management has become need of the hour to improve durability of electronics. MOSFET is an important part of any power converter circuit, to improve heat dissipation of a component we can use a thermal interface material which would improve heat transfer to ensure the component runs at lower temperatures which in turn, improves its long-term durability. In this research, a new material is developed by using a combination of two different materials to improve thermal dissipation from MOSFET. Different aspects of the topic will be discussed in the following sections of this chapter.

1.2 Power converters

The increase in performance and shrinking of size, the heat density of electronics has been increased. Which requires constant heat dissipation to ensure that the component provides us with maximum performance [1], [2] while consuming the least possible amount of energy. Depending upon the design and applications different form of electrical components ar e ,some components are very small in size, but they play a vital role in the functioning of the whole device. Hence their thermal management matters a lot. For example, in a computer system, If the heat dissipated from a CPU is not managed properly, it can cause the whole computer to slow down, hence affecting its performance.

A power converter is an electrical circuit that changes the electric energy from one form into the desired form optimized for the specific load. Power converters are being used widely in pretty much all aspects of life. One of its key components is MOSFET. It is a type of field effect transistor that is fabricated by controlled oxidation of a semiconductor, typically silicon. It is the most common power device. They have faster switching speeds, much smaller size and consume less power. MOSFETs are also cheaper and have relatively simple processing steps, resulting in high manufacturing yield. MOSFET scaling and miniaturization has been driving the rapid exponential growth of electronic semiconductor technology. The reliability of the power converter decreases significantly with every 10-15 C [3] increase in working temperature. Therefore, improving its thermal management will greatly benefit its longevity and efficiency. Thus, thermal management must be improved.

1.3 Thermal management

Electronic devices operate with their optimal performance when they are kept within the limits of their working temperatures. To ensure that they stay within their optimal limit we must ensure constant heat dissipation for regulation of the component temperature. Therefore, heat sink is attached to it, which is a heat exchanger that transfers the heat generated by the component to air passively. In computers, heat sinks are used to regulate the temperatures of all the components to make sure they perform at their highest performance levels [4]. They are also used with high power semiconductors like power transistors and Light emitting diodes. Wherever the heat dissipation ability of the component itself is insufficient to moderate its temperature. A heat sink is designed to maximize its surface area in contact with the fluid around it [2]. It can both be passively and actively cooled with the help of natural and forced air convection respectively.

1.4 Thermal interface

Whenever a heat sink is joined to the heat source, due to the roughness in the surface morphology of both the components the direct area of contact is only about 10-20% of the whole area is in direct contact. As shown in Figure 1-1 (a).

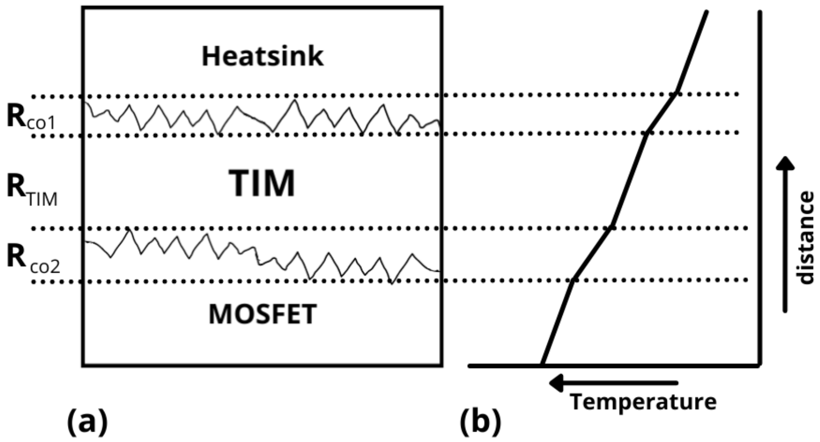

Figure 1-1 (a)Thermal interface material between MOSFET and heat sink (b) Temperature distribution

Temperature variation can be clearly seen with the help of temperature plot against distance in figure 1(b). Temperature drops extra at the interface which increases the difference in temperature of heat source and temperature at surface of heat sink. Lower the temperature at surface of heat sink means lower the amount of heat transfer from heat sink to surrounding air. Due to the roughness the air gets trapped between both surfaces. Since the thermal conductivity of air islower (k=0.024 W/ (m.K)). It resists heat transfer. Which basically provides us the exact place where we can work on to improve heat transfer. [5] The heat transfer can be greatly enhanced by reducing the thermal resistance at thermal interface. The heat resistance at thermal interface is reduced by placing a material at the thermal interface with better thermal properties that replaces the air at thermal interface of the components [6] . It helps us improve the heat transfer and ensures the constant heat dissipation from semiconductor and ensures that the component continues to work at the maximum performance.

1.5 Thermal interface material

To minimize the thermal resistance at thermal interface that is caused by air gaps trapped between both surface due to roughness in the form of concave, convex and wavy surface of both the heat sink and heat source [7]. This results in a notable temperature drop across an otherwise thin boundary of heat source and heat sink. Here, to overcome the surface asperities at thermal interface.,we use a material with a bulk thermal conductivity greater than that of air. That can conform to the surface asperities and can displace the small or larger air pockets to provide a better path of heat conduction [8]. Mechanically the material must be soft so it can create a border line thickness under reasonable pressure. But the thickness must be large enough to completely fill the air voids and surface irregularities [9]. And thermally they should exhibit low contact resistance with good thermal conductivity. They should also be reliable for long term use which means they can go through multiple cycles of heating and cooling without losing any performance.

It has been observed that one material alone would not be ideal for our use. It might be needed for us to mix some other material with it to make it ideal to use. It has been done in TIM since the beginning. The additives can serve multiple purposes such as improving thermal conductivity and decreasing its fluidity to avoid its flowing out when heated [10]

1.6 Thermal and physical properties of thermal interface materials

The material should possess certain properties to be useful as a thermal interface material.

- It should have high thermal conductivity as compared to air
- The coefficient of thermal expansion should be lower, if that is higher than it can cause stresses to develop on the heat sink which can cause cracking in the heat sink or circuit board due to stresses.
- It should be easy to handle and nontoxic in nature.
- Mounting orientation of the component with heat sink also highly affects the choice of TIM. If the thermal paste is electrically conductive and the component is mounted in vertical orientation, then there is a possibility that when heated, TIM can flow out on the circuit board and cause short circuit [11].
- Viscosity plays an important role in development of thermal interface materials.
- The material should also have good wettability characteristics so it can develop a good contact with the surfaces of the heat dye and heat sink which in turn, ensures that the heat transfer takes place in the most efficient manner.
- The application process of TIM should be simple and convenient so anyone can handle it to apply it.
- And the TIM should not degrade with time i.e reduction in moisture content and thermal conductivity over time.

1.7 Industrial significance

Using a better TIM result in improved thermal management of the component which means that the component can run at maximum performance without thermal failure. In the industry it is all about cutting down on unnecessary expenditures as much as possible without compromising that quality of product. When the company uses the TIM in their own equipment to ensure their processes run smoothly without any halt in operations due to break down of components. Also, better thermal management means that the component can run on higher performance for longer amount of time without any failure, that reduces the operations cost, saves breakdown cost as well as the time. Which means more time can be attributed to production. Which can improve efficiency and productivity of equipment which would eventually cause rise in profits.

1.8 Aims and objective

This study completely focuses on new thermal interface material. The material which has potential to improve heat transfer to achieve better thermal management for vertically mounted MOSFET. And performs better than conventional TIMs. Which would bring down losses in terms of repairing cost and reduce downtime. Other objectives are as follows

- To develop a cost-effective TIM which have potential to achieve better thermal management for vertically mounted MOSFET.
- To study thermal and morphological properties of TIM prepared
- To minimize the physical limitations of developed TIM.

1.9 Scope and limitations

The scope of this study is to develop a suitable thermal interface material for vertically mounted MOSFET. It must be cost effective and thermally conducting to improve heat transfer and reduce failure rate. The parameters that need to be improved is thermal conductivity and to reduce the fluidity of material.

The limitation of this work include gallium corrosion which limits the range of filler particles that can be used.

1.10 Thesis outline

Chapter 1 includes introduction to the need of the research, power converters and their thermal management, how it affects their operation, thermal and physical properties of a good Thermal interface material and its industrial significance

Chapter 2 includes literature review about different thermal interface materials developed. It also states numerous materials and combination of materials that were developed and tested by different researchers

Chapter 3 includes details about the material selection process, and it also includes the strategy that was used to evaluate prepared materials and the characterization techniques and Electrical circuit testing parameters that were used to verify that the prepared material was suitable for use in circuits

Chapter 4 includes the experiments that were carried out to verify hypothesis

Chapter 5 includes the results obtained and what those results represented and their significance in our research subject

Chapter 6 covers the conclusions from our experimental observations and results obtained.

Summary

For an ever-expanding use of electronics in modern world, operating these devices on high performance and avoiding failures is also a point of concern these days. To ensure smooth operation of electrical components, thermal management should be improved so the component can run smoothly at higher performance and to reduce failure rate. Chapter above includes introduction and basic details about electronics and how, by improving thermal management, one can improve reliability and durability of a circuit. Also explained what thermal interface is and how the temperature is dropped due to presence of air gaps with lower thermal conductivity at the interface. Thermal interface material that is used at thermal interface to improve thermal conductivity, and its necessary characteristics are discussed. Then the required thermal and physical properties that include better thermal conductivity, low electrical conductivity, high viscosity, nontoxic nature of material, ease of handle is explained. The importance of use of a better thermal interface material in electronics by industry is discussed and how it can help companies improve their profitability by bringing down their repair cost and downtime caused by failure of electrical components due to poor thermal management.

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Chapter 2: Literature Review

The entire study consists of developing a new thermal interface material suitable for use in vertically mounted MOSFET. The importance of electronics in modern world and their thermal management, which can be improved by using Thermal interface material is discussed in previous chapter. This chapter focuses on the research carried out to date on thermal interface materials. What and how different materials were used to improve the thermal dissipation. The combinations used and why there were combinations. Advantages and disadvantages of materials used and applications would be discussed in this chapter. All the different types of materials used at thermal interface are discussed below in section 2.1

2.1 Thermal interface materials developed so far

2.1.1 Thermal grease

Thermal grease which can also be called thermal gel or thermal paste is a thermally conductive and electrically insulation material and being most commonly used as thermal interface material in electronics and other applications, it is a little different as compared to thermal adhesive which also provides adhesive properties.

Figure 2-1 Thermal grease

In case of Thermal grease, some sort of mechanical fixation should be used such as screws to hold the heat sink in place to ensure application of constant pressure, which plays a vital role in heat transfer process. It is composed usually of a silicon or polymer-based liquid in which high thermal conductivity filler particles are incorporated to provide a thermal pathway with high thermal conductivity. Which in effect, helps lower the thermal resistivity and improves heat transfer. These high conductivity particles can consist of Oxide particles which are composed of Aluminum oxide which has a high thermal conductivity and is often used as a filler because of its low cost and high electrical resistivity. A thermal conductivity of 4.3 $W.m^{-1}.K^{-1}$ for epoxy composite containing 60 vol% Al_2O_3 (10µm) has been reported [1] Silicon dioxide (SiO_2) is one of the most commonly used substance in electronic manufacturing. Epoxy composites that contain 55 to 70 vol% fused silica are currently being used as packaging materials for electronic devices [2] beryllium oxide (BeO) has a thermal conductivity higher than any other nonmetal except diamond. It also shows corrosion resistance.

Nitride particles can also be used as filler particles specially Aluminum Nitride (AlN) its high intrinsic thermal conductivity, low coefficient of thermal expansion and high electrical resistivity has been the cause of attracting much attention recently. Thermal conductivity values of 11.5 and 11.0 $W.m^{-1}.K^{-1}$ have been reported for 60 vol% AlN based polyvinylidene and epoxy composites, respectively [3] Boron Nitride (BN) is also suitable as a filler for epoxies with high thermal conductivity. In addition to it, it also shows high electrical resistivity and low dielectric constant. Recently BN nanotubes and nanosheets have attracted research interests [4] and are a promising fillers for high thermal conductivity composites. Silicon Nitride (Si_3N_4) has a low coefficient of thermal expansion and low dielectric constant.

Graphene has attracted considerable attention because of its ultra high intrinsic thermal conductivity. However, due to its high electrical conductivity it is inappropriate to use as a filler material. It has been reported that 5.0 wt% graphene oxide epoxy composites show about a 4-fold increase in thermal conductivity and a low co efficient of thermal expansion [5]

Diamond is also a material that possesses very high thermal conductivity and has been used as a filler for preparing high conductive composites. A thermal conductivity of

4.1 $W.m^{-1}.K^{-1}$ was achieved for epoxy with 68 vol% diamond that had nominal size of 9μm [6]

2.1.2 Phase change materials

Phase change materials (PCMs) usually used are solid-liquid, have received considerable attention in recent years due to their high heat storage capacity [7]. Various forms have been utilized as thermal storage media for cooling and heating applications. These can be classified as inorganic compounds and organic compounds. Inorganic compounds are found more in abundance as compared to organic compounds.

PCMs can be much more reliable as compared to thermal pastes based on liquid and semiliquid carriers. In addition the performance can be enhanced by mixing the PCM with polymers and particulate fillers that possess high thermal conductivity. However, most PCMs with high energy storage density do possess low thermal conductivity.

A PCM with melting temperature a little above room temperature, higher heat of fusion, low viscosity in the liquid state, better thermal cycling stability and thermal conductivity is attractive for use as a thermal interface material.

2.1.3 Solders

Solder is usually used as a thermal interface material for enhancing the physical area of contact between surfaces because it can melt at low temperatures and the molten solder can flow and spread itself thinly on the adjoining surfaces, which result in improved direct contact which in turn increases the thermal contact at the interface between solder and each of the adjoining surfaces [8]. Moreover, solder in metallic state is a good thermal conductor. Inspite of high thermal conductivity of solder, the thickness of solder greatly impacts its effectiveness as thermal interface material, a small thickness does greatly improve heat transfer [9].

Solder TIMs are reflowed after application to form metallurgical bonds between substrates. They come in two forms of application, Solder paste or as a pre form foil, Since it consists of metal, its thermal conductivity is much larger as compared to polymer based thermal grease or PCMs. Together with liquid form which can fill out voids and wet to substrates during reflow a very low thermal resistance as low as 5 $mm^2.K/W$ can be achieved.

2.1.4 Carbon based materials

Carbon nanotubes (CNTs) are a honeycomb like arrangements of carbon atoms that are rolled into cylindrical tubes with diameters as small as few atoms wide and aspect ratio as high as 10^5, because of these unique features and strong carbon to carbon bonding they possess many exceptional thermal properties that have been utilized in many multiple applications [10]. CNTs can be produced from multiple processes which include Chemical vapour deposition (CVD) or plasma induced CVD processes and lasere vaporization of graphite targets. Considerable attention has been given to developing advanced TIMs that utilize the extremely high thermal conducitivity of CNTs [11]. Several studies have carried out on different materials which include dispersing CNTs in a polymer matrix composite [12]. Studies have also been carried out that include dispersing CNTs in Gallium matrix composite. Since thermal conductivity greatly depends on orientation of carbon nanotubes. Which is along its length. Several studies have been carried out in which growing vertically oriented carbon nanotubes was tried to achieve the full potential of carbon nanotubes [13]. It did impact the thermal conductivity greatly and significant increase was recorded.

Graphene has been recently gained interest in various fields which includes flexible electronics. It consists of single layer of atoms arranged in a two dimensional honey comb lattice. It has the same kind of bonding that is present in carbon nanotubes and therefore, has somewhat similar properties in the aspect of thermal conductivity [14]. Therefore, studies have been carried out to study its role as a thermal interface material.

2.1.5 Low melting temperature alloys

Low melting temperature alloys (LMTAs) containing different compositions of Gallium (Ga), Bismuth (Bi), tin (Sn) and Indium (In) have recently gained interest as their use as a TIM. They offer high thermal conductivities in the range of 26-39 $W.m^{-1}.K^{-1}$ which is much higher as compared to traditional silicon or polymer based thermal grease[15], [16]. Gallium primarily has recently been used as a TIM due to its suitable characteristics. Which include high wettability, the ability to wet and cover the surface completely and form a good mechanical bond which encourages heat transfer. Its low toxic nature is also one of the main reasons for its extensive use as a TIM. Low toxicity makes it easier to handle and apply.

It is also relatively more available, although it does not exist as pure metal but it is usually found in the ores including bauxite and sphalerite. Gallium readily forms alloy with most metals. The melting points of the alloys are different depending on the constituent and the proportion. EGaIn (78.6% Ga and 21.4% In by weight) has a melting point of 15.5 ^{o}C while Galinstan (68.5% Ga, 21.5% In and 10% Sn by weight) shows melting point of -19 ^{o}C and is commonly available. These liquid metal alloys depict high thermal conductivity and have recently started to be commercially available to be used by enthusiasts.

2.1.6 Thermal pads

Thermally conducting pads, also known as thermal pads have been used as TIM in electronics. It is a preformed rectangle of solid material which can be based on thermally conductive epoxies. It has been used extensively alongside thermal greases in electrical components such as MOSFETs, In Personal Computers it has been used to attach important components with heat sink.

At room temperature, thermal pads are relatively firm. On applying heat, they become soft and becomes able to fill gaps when exposed to heat. In comparison to thermal grease, Thermal pads are clean and relatively easier to install.

2.2 Limitations of conventional Thermal interface materials

2.2.1 Thermal grease

Thermal greases and their composition has been discussed above in section 2.1.1. It is evident from the discussion that filler particles play a significant role in deciding the quality of thermal grease. Thermal grease is the Thermal interface material that has been most widely used in electronics industry and therefore, has countless variations. In this section we will discuss some short comings and drawbacks of thermal grease with different filler particles.

In case of oxides, the most used is Aluminum oxide (Al_2O_3). Its disadvantage for insulating applications is its high dielectric constant which is around 9 at 1 MHz. While in case of Silicon Dioxide or silica (SiO_2) its low intrinsic thermal conductivity is a bottleneck for thermal conductivity of its composites. At a specific level of loading, silicon dioxide based polymer composites usually show the lowest thermal conductivity when compared with composite containing other fillers [17].

In case of nitrides, one with most interest is Aluminum Nitride (AlN). In its case, the low oxidation resistance and relatively high dielectric constant limits its application because it can oxidize, which would cause its thermal conductivity to drop drastically. Despite the excellent properties of Silicon Nitride (Si_3N_4) it is seldom used as filler for high thermal conductivity composites because of its moderate thermal conductivity [18] as can be seen in table.

Graphene oxide is easily reduced or partially reduced via low temperature thermal treatment which limits the temperature range of graphene oxide composites. And after reduction, its thermal conductivity reduces drastically.

Diamond has very high thermal conductivity but it is seldomly used because of its high cost. With its use, its thermal conductivity was raised to 4.1. In comparison with thermal epoxies available of other fillers, this clearly indicates that diamond does not show any excess superiority in development of high thermal conductivity composites.

Discussed above are some very high conductivity filler particles. But even using those could only take the thermal conductivity as high as 4-5 $W.m^{-1}.K^{-1}$. Which is consistent with the prediction by nielsen's equation where the thermal conductivity of composites is insensitive to to the intrinsic thermal conductivity of filler particles when the intrinsic thermal conductivity of filler is greater than 100 times of the polymer matrix.

2.2.2 Phase change materials

Common problems that are associated with inorganic PCMs are supercooling and the behaviour of not showing thermal stability on experiencing multiple thermal cycles. Because solidification involves nucleation. Numerous nucleating additives that have crystal structure similar to the PCM and with little solubility in the PCMs have been studied to find a suitable option to address the issue of supercooling. However, there was intuition behind the selection of nucleators for the study. And no explanation of their effectiveness was provided.

The evaluation of actual performance of PCMs as a thermal interface material still needs to be studied deeply as there are several issues with PCMs that need to be addressed, since being used a thermal interface materials was not one of the reason behind development of PCMs. They were designed to store heat for the purpose of cooling or heating. Which they already are being extensively used for. And a lot of studies have been carried out in such role.

2.2.3 Solders

As discussed in section above those solders do offer great thermal contact and thermal conductivity as compared to conventional thermal greases and other TIMs. It also offers adhesion. However, It does have some drawbacks that make it not suitable for extensive usage apart from some very specific user case scenarios.

Some of its drawbacks include difficulty in repairability, you have to apply relatively high temperature to remelt it again to rejoin it. Which can cause the component to fail if it is highly sensitive to high temperatures. It can also induce thermal stress between the heat source and heat sink and can cause cracking due to uneven cooling which can develop thermal stresses. Extreme care is needed during application process to avoid this result. High temperature also needs to be applied during application process since solder material is a metal which requires high temperature to melt so it can be applied evenly and with good contact to the surface. The surfaces need to be heated also to develop a good contact with the solder. Which can cause damage if the heat source is a component that is highly sensitive to extreme temperatures. Which would cause more damage than good.

Cost effectiveness is also a major upcoming concern in solders, since lead (Pb) was primarily used as a solder which is inexpensive and is easy to find. However, due to environmental and health concerns. New alternatives are being developed which would not be cost effective and would have issues that need to be addressed. Therefore, use of solders has been very limited as thermal interface material.

2.2.4 Carbon based materials

Carbon based materials consist of carbon nanotubes and graphene sheets. Although both materials report good thermal conductivity and exceptional wetting properties, They still are not suitable for commercial use due to their complex production processes, which require large amounts of capital in terms of complex machinery and very controlled environment. It is expensive due to its rarity and production process. It still is mainly used for research purposes.

Both of these materials properties are highly dependent on its orientation. Which means you only benefit from its high thermal conductivity when you apply it a certain way onto the substrate. Incase of vertically aligned carbon nanotubes you have to grow these nanotubes on the surface. Which require controlled environment and complex machinery. Which makes it highly unsuitable for commercial use due to complex fabrication process.

However, carbon nanotubes and graphene are still in their initial phase and extensive research has been carried out so modern world can benefit from these amazing materials in the best way possible.

2.2.5 Low melting temperature alloys

Although gallium alloys have very low thermal resistance of 1.5-6.5 mm^2.K/W and high thermal conductivity. There still are some concerns regarding wettability with some metal surfaces such as copper, silicon and stainless steel is rather poor [19] and therefore, the gallium droplets are easily extruded out from the interface which may fall on circuit board and can cause short circuit, damaging the equipment permanently. Recently some research has been carried out in which micro-oxidation reaction in which gallium oxide is equally dispersed into liquid metal uniformly by vigorous stirring. It can effectively improve wettability of low melting temperature gallium alloys with other materials. However, micro-oxidation can affect its thermal conductivity.

Fluidity is also a major concern in using LMTAs as TIM because on heating LMTAs can expand and droplets can flow out on the circuit board to cause short circuit. Causing permanent damage to the circuit and components. It also is highly corrosive to metals like copper and aluminum. Therefore, extreme care is required while using it as a TIM in any circuit.

2.2.6 Thermal pads

With the ease of installation and ease of handling in nature of thermal pads there also are some drawbacks that limit its use. First of all is its low efficiency of conducting heat. Thermal pads conduct heat less effectively as compared to thermal paste. In case of thermal pads the polymer matrix is heavily crosslinked which is the main cause of high viscosity and firmness and ease of handle. This leads to relatively thick border line thickness and requiring high pressure to properly sit on its substrates. Since high filler fractions of conducting particles increase the stiffness of the composite. Its overall performance is severely limited by this tradeoff between softness and filler fraction [30]

Table 2-1 Literature review with key findings

Title	**Key findings**	**Reference**
Gallium based liquid metal alloy incorporating oxide free copper nanoparticle clusters for high performance thermal interface materials	Copper nanoparticles were incorporated into liquid metal gallium-indium-tin (GaInSn). 80% increase in thermal conductivity was recorded while liquid metal maintained its fluidity.	[25]
Investigating on enhancing the thermal conductance of thermal interface materials using chromium coated diamond particles	Addition of chromium coated diamond particles to liquid metal gallium can increase its thermal conductivity to 112.5 W/(m.K) from 29.3 W/(m.K). Gallium forms intermetallic compound with high thermal conductivity metals like copper rapidly and dries out.	[22]
Efficient heat conducting liquid metal/CNT pads with thermal interface materials	Carbon nanotubes embedded in Liquid metal gallium results in a increase of thermal conductivity reaching 14.2 W/(m.K)as compared to commercial thermal pastes.	[13]
High thermal conductivity liquid metal pad for heat dissipation in electronic devices	Silver nanoparticles added to gallium can reduce working temperatures of cell phones by up to 5.8 degree Celsius	[23]
Thermal performance of low melting temperature alloys at the interface between dissimilar metals	Three Low melting temperature alloys (Ga-In), (Ga), (In-Bi-Sn) were tested. (Ga) showed lowest thermal resistance over thermal aging of upto 3000 hours.	[19]
Gallium based thermal interface material with high compliance and wettability	Oxidation causes thermal conductivity of gallium to drop to 13.1 from 29.3 W/(m.K), while wettability is improved.	[21]

Silver nanoparticles based thermal interface materials with ultra low thermal resistance for power electronics application	Thermal conductivity of Silver nanoparticles is very good as a Thermal interface material. It originates from its thinness, high thermal conductivity and low temperature sintering properties.	[24]
A review of dielectric polymer composites with high thermal conductivity	The thermal conductivity of a composite increases appreciably with the intrinsic thermal conductivity of the filler particles.	[26]

2.3 Novel thermal interface materials

The research interest in use of gallium as thermal interface material has been on the rise due to its superior thermophysical properties such as high thermal conductivity, fluidity, large surface tension, negligible vapor pressure, low thermal resistance, low toxic which makes it safe to handle and low melting temperature which makes it easier to handle. So far it has been reported as TIM for microprocessors in desktop computers, Light emitting diodes. However, there is a key issue in electrical safety for Liquid metal usage in electronics because it can leak out to short the circuit in motherboard during operation when the chip gets heated And different research have been carried out to improve the thermal conductivity, increase the viscosity and adhesion to surface.

In research carried out a gallium based thermal interface material was reported as a TIM that consisted of gallium oxides that were dispersed uniformly into 99% gallium metal. Wettability of the material with other materials is disclosed and compared. The thermal conductivity of such material was measured by a Thermal analyzer came out to be 13.07 W m^{-1} K^{-1} which is significantly higher as compared to the conventional thermal greases [21]. Also, an experimental study was carried out to measure the thermal resistance across the TIM under steady state condition which resulted in thermal interface resistance as low as 2.6 mm^2kW^{-1} which is also significantly lower than that of best commercially available thermal greases. It is convenient to form thermal pad out of such material due to its semi-liquid or paste like physical properties.

In another research, high heat conducting diamond particles were added to gallium liquid metal. As diamond is widely known as a material that possesses one of the highest thermal conductivities. Since diamond differs in nature with metals. To improve the contact of both the filler particle and metal matrix composite, chromium transition layer is deposited on the surface of diamond particles by magnetron sputtering method. The results present that 47 wt% addition of diamond particles metallized with chromium can increase the thermal conductivity of pure gallium from 29.3 to 112.5 W $m^{-1}.K^{-1}$. Also on thermal aging the TIM at 80 ^{o}C for 192 hours showed no signs of chromium layer degradation. [22] Which makes it very ideal for use as TIM. However, due to high cost of materials such as chromium coated diamond particles this TIM would not be commercially feasible.

A TIM using silver doped gallium based liquid metal was proposed to improve the heat dissipation for electronics. Addition of silver served several purposes which increased viscosity and reduced fluidity to prevent leakage on the other hand it also helped increase the thermal conductivity of gallium upto 46 $W.m^{-1}.K^{-1}$. Some other experiments were performed to evaluate the heat dissipation performance on the CPU of smart phone. The results demonstrated that silver doped gallium based TIM can effectively reduce the CPU temperature by upto 5.9 ^{o}C as compared to conventional commercial thermal greases [23].

Silver nanoparticles (AgNP) were employed as TIM and high conductivity and ultra-low thermal resistance was achieved. The low thermal resistance is caused by the thinness, high thermal conductivity of silver and low temperature sintering properties of AgNP [24]. Vertically aligned carbon nanotubes (VACNT) that have high thermal conductivity that were synthesized by chemical vapor deposition method were used as a TIM. Gallium based LMTA was used as a bonding material to attach VACNT films onto copper plates. Thermal parameters were measured by laser flash analysis (LFA) method. And the results showed that VACNT films do offer high thermal conductivity and is suitable for use as a TIM to enhance heat transfer performance of contact surface [13].

Summary

This chapter 2 includes detailed discussion about TIM developed so far that consists of thermal greases, phase change materials, solders, carbon-based materials, Low melting temperature alloys and thermal pads. The existing research on such materials was discussed in detail. Also, their limitations were discussed which does imply the need for a TIM with better thermal and physical properties. Later in the chapter novel material combinations that were the focus of recent research studies have been discussed where oxidation of gallium and filler particles to improve its thermal and physical properties. Those filler particles ranged from high conductivity diamond particles to silver nanoparticles added to gallium. VACNT were also used in conjunction with gallium as TIM and showed a significant increase in the thermal conductivity.

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Chapter 3: Methodology

In this chapter we will discuss the methodology and the strategy that will be used to carry out this research successfully. In previous chapters we have discussed the basics needed to understand this study and the already available materials that are being used as thermal interface materials conventionally. The TIMs that are most widely used and some TIMs were also discussed that are relatively new and does required extensive amount of research to make the TIM suitable for use commercially. In this chapter we will explain the methods through which we will achieve our goal of developing a TIM for use in power electronics, MOSFET in particular. vertically mounted thermal pads are conventionally t being used as a TIM. Which has both low thermal conductivity and requires high mounting pressure to conform to surface irregularities present at the thermal interface. In this section we will discuss material selection which includes matrix selection and filler particle selection. It also includes material preparation and the processes to carry it out in the right way. Also the properties that require characterization, their significance and the way to carry it out will be discussed. In the last section electrical circuit that was used to test and quantify the improvement in electronics that our material provides will be discussed and the data acquisition will be discussed.

3.1 Material selection

Finding a suitable material for development of a new material is of key importance since it is highly dependent on the application it is being developed for. Parameters like working temperature range among others play an important role in the material selection process for developing a TIM with a new combination of materials. Also the orientation in which the component will be mounted to heat sink does matter a lot in the selection process of the right matrix for the new proposed materials.

3.1.1 Matrix composite selection

Liquid metal would be used due to its soft nature at room temperature. It can be handled easily. However, there are certain parameters that should be kept in mind while selecting a metal matrix for development of a TIM. The matrix should be in liquid form around room temperatures. It should be easy to handle to make the application process easy and convenient. It should be nontoxic in nature therefore, should not possess any health concerns to humans. It should possess high thermal conductivity properties.

Two of the most commonly available liquid metals are Gallium and mercury. They possess thermal conductivities of 29.3 and 8.30 W $m^{-1}K^{-1}$ respectively. Melting points of 29.78 and -38.78 ^{o}C. Gallium is nontoxic in nature while mercury is toxic in nature and causes health problems among individuals.

Table 3-1 Properties comparison of liquid metals

Metal	**Melting point (^{o}C)**	**Thermal conductivity ($W.m^{-1}K^{-1}$)**	**Nature**
Gallium (Ga)	29.78	29.3	Non-toxic
Mercury (Hg)	-38.78	8.3	Toxic
Rubidium (Rb)	39.30	58.2	Toxic
Caesium (Cs)	28.5	35.9	Toxic
Francium (Fr)	27	15.9	Radioactive

Therefore, these results indicate that Gallium is the most suitable matrix for our desired TIM and would provide the best results due to its high thermal conductivity and nontoxic nature. It has the potential to replace conventional thermal greases due to its much superior thermal and physical properties. However, some of its physical properties need to be addressed in upcoming stages that include wettability with certain materials and that can be addressed by oxidizing gallium [1]

3.1.2 Filler particles

Filler particles play a vital role in improving thermal conductivity. Their thermal conductivity defines the thermal conductivity of the resulting material. Also, there are several properties that need to be kept in mind while selecting a suitable filler particle for use in a TIM that is being developed for a particular application. Which include its thermal conductivity. It is most basic and significant parameter that needs to be kept in mind while selecting filler particles. If its thermal conductivity is not high. It does not fulfill its purpose that is, improving the thermal conductivity of overall material. Another parameter that needs to be kept in mind is the nature of its contact with the matrix. It can improve thermal conductivity by providing a path of high thermally conducting particles inside the matrix. But if the contact between matrix and filler particles is not good enough, heat will not be transferred efficiently. Which would not fulfill its purpose.

Keeping the parameters in mind, we have to start looking for a material suitable to use as filler particle. For inorganic nonmetal fillers such as diamond, CNTs, graphene or boron nitride (BN) the current bottleneck is not their intrinsic heat conductivity but the poor wettability with liquid metal. Gallium has a little issue when studying its contact with other metals, the reaction rate of gallium with high thermal conducting metals such as copper, aluminum and silver is so rapid that it forms intermetallic compounds in a very short period along with liquid metal drying out. However, there is a class of metals called refractory metals that are resistant to gallium corrosion. This group consists of niobium (Nb), Molybdenum (Mo), tantalum (Ta), Tungsten (W) and Rhenium (Re). among these metals tungsten possesses the highest thermal conductivity. Therefore, tungsten will be used as a filler material for our new TIM.

Table 3-2 Thermal conductivities of refractory metals

Sr. No	**Metal**	**Thermal conductivity ($W.m^{-1}.K^{-1}$)**
1	Niobium (Nb)	53.7
2	Molybdenum (Mo)	138
3	Tantalum (Ta)	57.5
4	Tungsten (W)	173
5	Rhenium (Re)	48

Tungsten microparticles that are in the powdered form will be used because its contact with gallium is purely mechanical and there is no alloy formation as shown in a study[2], [3]. Because it will also serve one of our other goals which is to decrease the fluidity of Ga to address the flowing out and causing short circuit issue. Particle size of around 25 µm will be used for tungsten filler particles.

3.2 Material preparation

This section focuses on the methods used to prepare our thermal interface material and discusses the reasons behind different stages used in material preparation. Different stages that are discussed below consists of stirring pure gallium for oxidation, then the tungsten filler particles are added to the oxidized gallium and mixed. Then at the end sonication is carried out with the help of sonicator to ensure uniform and equal dispersion of tungsten filler particles in gallium matrix to ensure homogeneity of the whole sample. Material preparation is a key step of the whole process because if the material is not prepared with utmost care and accuracy. It will affect the results at all the upcoming stages. Three samples were prepared with different composition at wt% Therefore, parameters need to be carefully selected and adjusted to achieve uniformity in preparation of all the samples. So, at a later point in time when different characterization techniques are being used to quantify its properties. It shows a uniformity, and a right comparison can be made.

3.2.1 Stirring

Pure gallium (purity 99.99 wt%) does exhibit some undesired properties like fluidity and does express poor wettability with some surfaces. Both of issues need to be addressed in preparation of all of the three samples. Gallium does oxidized and forms gallium oxide at outer side in order to prevent from further oxidation. Oxidized gallium does express low fluidity as compared to pure gallium. It also shows improvement in wettability properties of gallium[1].

Therefore, to improve the wettability property of gallium, it was left on a hot dish at the temperature of 39 ^{o}C. for 24 hours with the stirrer on. So it keeps agitating the liquid metal and does not allow the oxide to form on the outer layer. Which in turn does not let the oxidation stop and the oxide keeps forming which improves the wettability characteristics. The viscosity of the material kept increasing gradually with the amount of gallium. Therefore, after some time of stirring gallium metal with suitable viscosity should be collected. The experimental parameters such as amount of gallium, stirring time and stirring speed should be tightly controlled throughout the 3 samples prepared to ensure uniformity.

However, this stirring and formation of oxide does also have a drawback regarding the thermal conductivity. It reduces the thermal conductivity from 29.3 to 13.1 $W.m^{-1}.K^{-1}$. Therefore, this oxidation should be carried out keeping in mind this tradeoff for thermal conductivity. Increase in viscosity does take precedent over thermal conductivity in current study. After all, filler particles with high thermal conductivity will be added later that would cause a rise in thermal conductivity of overall material. And will take us one step closer to developing a better TIM for use in power electronic components such as MOSFET. Which would help us push its performance and allow it to operate at higher performance levels to cater to always increasing electronics demand.

3.2.2 Mixing

After stirring and causing oxidation of gallium to address the issues of wettability and fluidity. Filler particles that were actually tungsten particles were added to liquid metal gallium. That would provide a conductive path with better thermal conductivity to heat and would cause heat dissipation to improve as compared to gallium matrix which has lower thermal conductivity as compared to tungsten filler particles.

While keeping the stirrer on, tungsten filler particles were added to it in small amounts to ensure good dispersion of tungsten particles and also ensures that no lumps of tungsten micro particles are formed which would cause issues with homogeneity of the material and the thermal properties might be affected by this short coming. Therefore, extreme caution needs to be carried out to ensure that the material is prepared in uniform conditions so their properties are comparable with each other without any doubt of error

Table 3-3 Composition of samples

Element	Weight% (Sample 1)	Weight% (Sample 2)	Weight% (Sample 3)
Gallium	90	55	33
Tungsten	10	45	67

Three different samples would be prepared with different wt% to study the effect of variation of tungsten micro particles on thermal and physical properties such as thermal conductivity, thermal diffusivity and viscosity and fluidity. Also, all three samples would be tested as a TIM between MOSFET and a heatsink and the effect of it on different thermal and electrical parameters will be observed.

3.2.3 Sonication

To standardize the material so its properties can be recorded and are comparable for study of their thermal and physical properties. Homogeneity of the mixture of gallium and tungsten micro particles is necessary to study the properties as a bulk material. If it does not happen. Then the properties will vary highly with the variation in sample and where the sample is taken from and what kind of irregularities are found in the sample.

Sonication is the act of applying sound energy to agitate particles in a sample. Ultrasonic frequencies (>20 kHz) are usually used. It is applied on the sample in laboratory using an ultrasonic bath called sonicator. In our situation ultrasonic bath was kept at 60 ^{o}C and the equipment operated at the frequency of 40 kHz. Using this method, we can be sure that there are no air bubbles present in the sample and that the filler particles are equally and uniformly dispersed in gallium matrix.

3.3 Material characterization

After the material preparation is discussed in detail in the section above. Extra care needs to be taken in case of remembering and maintaining the parameters throughout the preparation of all three samples so they can be compared easily. In this chapter the thermal and physical properties of the material prepared above will be discussed that matter the most. Thermal properties such as thermal conductivity, thermal diffusivity and volumetric heat capacity of material gives us a detailed idea about heat conducting attributes of any material. Viscosity tells us about the fluidity of material, surface morphology tells us about the extent of surface asperities on any surface. And morphology which is evaluated by placing the material under a microscope can tell us about the interaction of both materials on microscopic level.

3.3.1 Thermal conductivity

Thermal conductivity is a main parameter of any material that tells us about its ability to conduct heat. And it is an important parameter to quantify to study the abilities to conduct heat. It is one of our main objectives to improve thermal conductivity of gallium by adding high thermal conductivity filler particles to gallium metal matrix.

Thermal constant analyzer (Model: TPS 1500, Hot disk instrument) as shown in Figure below was used to measure thermal properties, mainly thermal conductivity of the sample. Accuracy of any equipment means how close or far off a given set of readings are to their true value. The equipment used to measure the thermal conductivity mentioned above has an accuracy better than 5% which when compared to other measuring devices is quite good. It can reproduce results with up to 1% variation in values.

Figure 3-1 Thermal constant analyzer

Due to soft nature of material, we prepared, a Teflon crucible was manufactured with the dimensions and can be seen in figure below. This was to provide support for our TIM during the thermal conductivity testing to keep it in one place. The cavity in crucible had a diameter of 15 mm and height of 4 mm. Final form of Teflon crucibles filled with TIM can also be seen in figure.

3.3.2 Thermal diffusivity

It is also a parameter that measures the ability to diffuse heat through it. In a substance with high thermal diffusivity, heat moves rapidly through it because the substance conducts heat rapidly. It measures the rate of heat transfer of a material from a hot end to the cold end. It has the SI derived unit of m^2/s Therefore, this parameter does tell a lot about an ability of any substance to allow heat to pass through it.

This parameter is also calculated by the instrument mentioned above to calculate thermal conductivity. That is, thermal constant analyzer (Model: TPS 1500, Hot disk instrument). It consists of a high precision temperature sensor sandwiched between the samples. Heat in the form of pulse is given to the sample and temperature change is detected by the temperature sensor. By making some calculations, thermal diffusivity can be easily calculated and determined by tha apparatus.

3.3.3 Volumetric heat capacity

The volumetric heat capacity of a material is the heat capacity of a sample of the substance divided by the volume of the sample. It is the amount of energy that must be added, in the form of heat, to one unit of volume of the material in order to cause an increase of one unit in its temperature. The SI unit of volumetric heat capacity is joule per kelvin per cubic meter, $J \cdot K^{-1} \cdot m^{-3}$.

The volumetric heat capacity often varies with temperature and is different for each state of matter. While the substance is undergoing a phase transition, such as melting or boiling, its volumetric heat capacity is technically infinite, because the heat goes into changing its state rather than raising its temperature.

This parameter is also evaluated by the thermal constant analyzer (Model: TPS 1500, Hot disk instrument). In which a temperature sensor is sandwiched between two samples. Which in this case, will be Teflon crucibles filled with TIM prepared. Heat energy would be provided to the samples and temperature change will be recorded with the help of temperature sensor and using some mathematical expressions, the equipment will provide us with volumetric heat capacity which would give us an idea about the ability of each sample to conduct heat.

3.3.4 Surface roughness

Surface roughness is an important factor that helps us selecting the suitable size of filler particles. An AFM generates images by scanning a small cantilever over the surface of a sample. The sharp tip on the end of the cantilever contacts the surface, bending the cantilever and changing the amount of laser light reflected into the photodiode. The height of the cantilever is then adjusted to restore the response signal, resulting in the measured cantilever height tracing the surface. Also, it tells use about microscopic surface asperities on any surface.

Surface morphology of MOSFET was evaluated using Atomic Force Microscopy (Nanosurf flex AFM) over the area of 25 µm * 25 µm. To evaluate the extent of irregularities. That would eventually help us in choosing the size of tungsten micro particles. So that the filler particles can sit in between the microscopic surface asperities on surface of MOSFET and the heat sink providing a better path for conduction of heat. Improving the overall thermal conductivity of material.

Figure 3-2 Nanosurf flex Atomic Force Microscopy (AFM)[4]

3.3.5 Viscosity

While going through all the phases of research one of the main objectives was to increase the viscosity of TIM to make it suitable for use in vertically mounted MOSFET with heat sink. Increase in viscosity would cause the fluidity to decrease. This increase in viscosity is caused by addition of tungsten micro particles that are in powdered form.

The change in viscosity would be observed and verified based on visual observations. Since increase in viscosity also helps us avoid flowing out of TIM when heated.

3.3.6 Morphology

Morphology needs to be evaluated to study the contact of matrix with filler particles on microscopic level. This would provide visual evidence that the contact of gallium matrix with tungsten filler particles is purely physical and no kind of bonds are formed which enforces the hypothesis behind selection of tungsten micro particles as a filler material that tungsten is resistant to gallium corrosion. Therefore, can be used in combination with gallium whenever needed. It has several properties such as tough surface, corrosion resistance hard in nature.

Figure 3-3 Scanning electron microscope (SEM)

Samples were placed under SEM (Tescan vega 3). Extra care was used to prepare the samples due to the low melting point of samples which could cause the material to melt when an electron beam is targeted on it. Which would cause discrepancy among results. A small amount of sample was used, and the sample was pressed and spread on the carbon tape. The sample was then coated with a layer of gold whose thickness was 15 nm. Just to prevent the rapid melting of the sample. The sample was kept under the electron beam for the lowest time possible. The pressure was around 10-7 torr.

3.4 Electrical testing

The plan next is to study the effect of using the TIM we prepared along with conventionally available TIMs by using it in the application it was designed to be used in. In this section, we study about the circuit that was used to test the performance of

TIMs prepared along with thermal grease and no TIM. So the comparison between performance of all the materials can be made. That is followed by data acquisition methods that will be used to record thermal data which consists of temperature readings and variation and electrical data that consists of recording currents and voltages at every step.

3.4.1 Circuit details

To measure the effectiveness of TIM prepared, It will be used in a circuit called boost converter. It is a DC-to-DC power converter that steps up the voltage while stepping down current. It contains a diode, MOSFET, capacitor and inductor. The circuit diagram can be seen in figure below

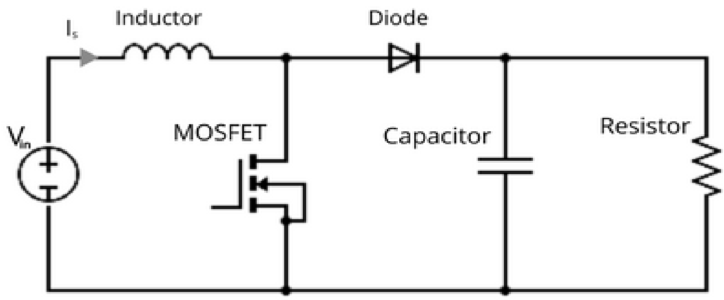

Figure 3-4 circuit diagram of Boost Circuit

There are two types of heat losses usually encountered in MOSFET. Heat is usually dissipated in the form of switching losses which means temperature rises with the rise in switching frequency. Temperature rise can affect the performance of MOSFET. Conduction losses can also be a reason for heat loss. Which is caused by the rise in drain to source resistance with the rise in temperature. Therefore, lower working temperature means lower heat losses.

3.4.2 TIM

TIM used between MOSFET and heat sink were different. 5 different runs were carried out in which frequency of MOSFET was increased from 10 kHz to as far as the MOSFET can go. In order to quantify the effect of better thermal management on the performance of MOSFETs.

Five experimental runs were performed with different TIMs, their details are given below:

- MOSFET with a heat sink without any TIM
- MOSFET with a heat sink and conventional thermal grease as TIM
- MOSFET with sample 1 as a TIM
- MOSFET with sample 2 as a TIM
- MOSFET with sample 3 as a TIM

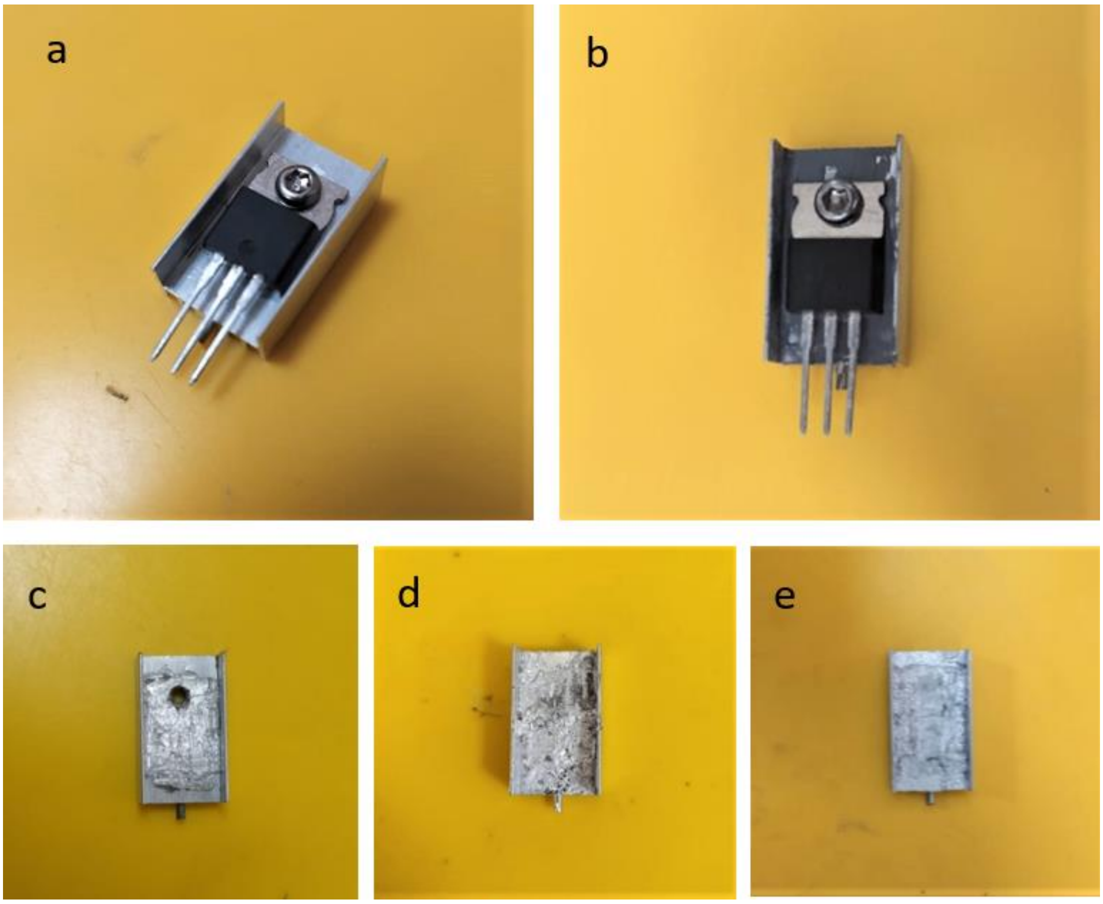

Figure 3-5 TIM applied (a) No TIM (b)Thermal grease (c) Sample 1 (d) Sample 2 (e) Sample 3

3.4.3 Electrical data acquisition

Data acquisition is a key factor in this study. It is acquired so study can be carried out on the effect of using a TIM with better thermal properties. Some parameters on which the circuit will be run are already known like input voltage and current across components.

Electrical parameters that need to be recorded are current at variable frequency. But extra care should be taken to ensure uniformity over all the samples that the conditions in which data is collected are uniform thus providing us with reliable electric data.

3.4.4 Thermal data acquisition

To study the effect of better TIMs used with MOSFET. Thermal data plays an important role in quantifying the effects of a better TIM. It consists of temperature values at the surfaces of MOSFET and heat sink. Calculating the temperature difference, we can develop an estimate about thermal conducting properties of our TIM.

Figure 3-6 Thermal imaging camera (FLUKE Ti-400)

The data was recorded with the help of Thermal imaging camera (FLUKE Ti-400). Thermal images were taken of the MOSFET and heat sink to evaluate the surface temperatures and the temperature distribution on the surfaces. It will provide with data needed to quantify heat conducting properties of TIM.

Summary

The chapter above discusses the methodology to carry out the research that suits our requirements in the best way possible. It consisted of the material selection process that included the selection of matrix material and filler particles. The analysis of basic materials showed us that gallium and tungsten were most suitable to use a matrix and filler particles respectively. Then material preparation procedure was studied which included stirring for oxidation of gallium, then it would be mixed with tungsten filler particles and at the end the mixture will be passed through sonicator to ensure uniform dispersion of filler particles in the gallium matrix. Then characterization was discussed which included thermal as well as physical properties of TIM. Electrical testing setup will be used to study the effect of a TIM with better properties directly on the circuit.

References

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[2] Kurtz, Wolfgang, and Hans Vanecek. "Gallium." W Tungsten. Springer, Berlin, Heidelberg, 1987. 12-14.

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[4] Flexible research AFM for materials and life science Nanosurf | Quantum Design.

Chapter 4: Experimental Work

Methodology or strategy that would be carried out in order to execute the proposed study on development of a TIM for use in power electronics components, such as MOSFETs. In current chapter the experimental procedure will be described. Experimental work done on preparation of TIM as discussed in methodology section above. Then to carry out characterization. The experimental setups and their working principle are already discussed in chapter above in detail. The experimental work done to characterize the samples prepared will be described.

4.1 Material preparation

Material selection and material preparation procedures were discussed in detail in previous chapter. Gallium metal was used as a liquid matrix and tungsten microparticles were employed to be used as filler particles in it to prepare new TIM that has better thermal and physical properties as compared to conventionally available TIM. Each step is discussed in detail below.

4.1.1 Stirring

Gallium liquid metal has been chosen as matrix for our TIM due to its superior thermal and physical properties such as thermal conductivity, nontoxic nature, and low melting point. However, it still faces some issues regarding its wettability with certain surfaces and high fluidity.

To address these issues, gallium needs to be oxidized and it would impact both the wettability issues due to formation of oxide that has better wetting properties as compared to pure gallium. Oxidized gallium does also increase its viscosity.

Gallium (99.9 % purity) was melted on a hot plate and was stirred vigorously to encourage to oxide formation. The agitation also helps break up to outer oxide layer that forms after oxidation which ensures uniform dispersion of gallium oxide. The temperature was kept at 39 ^{o}C and was stirred with the help of magnetic stirrer for 24 hours.

Figure 4-1 Stirring and mixing

4.1.2 Mixing

After oxidizing gallium metal at 39 ^{o}C for 24 hours. Which has improved its wettability as well as viscosity. Now the filler particles that are tungsten micro particles will be added to gallium matrix with the stirrer on. It also serves two main purposes. It improves thermal conductivity of overall material as well as increase the viscosity which would convert into a semi solid paste that is soft to touch and does not flow easily. Therefore, is easier to handle as compared to pure gallium. Its conversion to semi-solid paste like form also makes its application easier.

After stirring for 24 hours at 39 ^{o}C. Tungsten filler particles will be started being added in small amounts in liquid metal gallium to ensure uniform dispersion of tungsten particles and to avoid formation of any kind of lumps. Three samples should be prepared with varying percentage by weight amount of tungsten micro particles. Extreme care should be taken place while preparing the material.

4.1.3 Sonication

After the tungsten micro particles have been added to gallium liquid metal. All the three samples that are prepared for testing are to be placed in sonicator. Sonication of material will help disperse the tungsten microparticles uniformly throughout the material. Hence causing the homogeneity in materials that would make it suitable to treat a part of sample as the whole sample. The water temperature is kept at 60 °C to ensure that gallium stays in liquid form. The sonicator was run for 4 hours at 40 kHz. After that, three samples that had different concentrations of tungsten microparticles were obtained and those had uniform dispersion of tungsten microparticles throughout them.

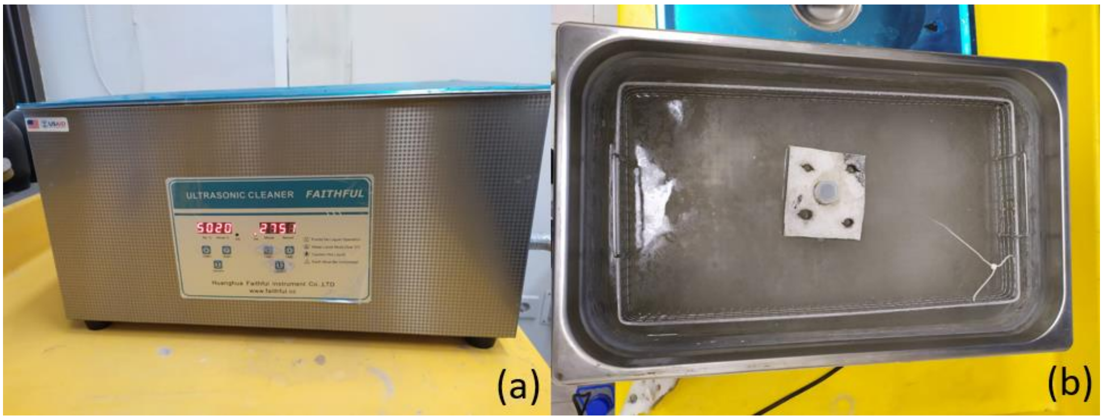

Figure 4-2 Sonicator (a) front view (b) top view

4.2 Thermal properties

After the material has been prepared using stirring, mixing and sonication. The first step in characterization techniques is testing thermal properties that would help us evaluate heat conducting ability of the TIM prepared. Thermal constant analyzer (HOT DISK instruments, TPS-1500). It provides use with three different thermal parameters which include thermal conductivity, thermal diffusivity and volumetric heat capacity. These three parameters provide an idea about heat conducting characteristics of the material. The equipment has an accuracy better than 5% and can reproduce results with upto 1% variation in them.

Due to soft nature of TIM. A Teflon crucible is used to hold material in its place and provide it with structural support during the thermal conductivity testing to keep it in one place. The crucibles filled with TIM prepared can be seen below in fig. two crucibles are needed for each sample.

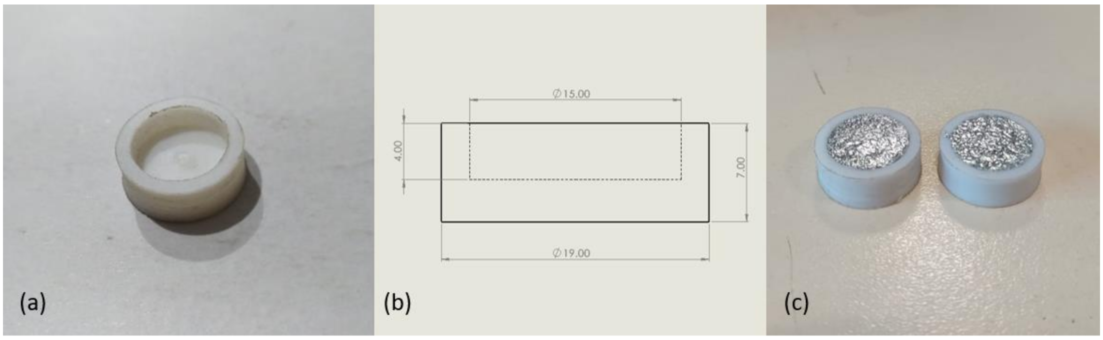

Figure 4-3 (a) Teflon crucible (b) Dimensions (c) Material filled

In thermal constant analyzer a temperature sensor is sandwiched between two discs of sample. In our case two crucibles filled with TIM. The temperature is kept at 30 ^oC. the experiment was run multiple times to ensure repeatability of results and to ensure the fact that results were close to each other.

4.3 Surface roughness

Surface roughness of MOSFET surface was evaluated using atomic force microscopy (AFM) it was done over the area of 25 µm * 25 µm. to evaluate the extent of irregularities on the surface of MOSFET.

4.4 Morphology

Morphology study was carried out by examining the three samples based on visual examination over microscope. Scanning Electron Microscopy (SEM) was used for microscopic evaluation of material. Extra care was used to prepare the samples due to its low melting point which would cause the material to melt when electron beam is targeted on it. Which could cause discrepancy among results. A small amount was taken and placed on carbon tape that was adhered to the stub. It was then coated with a layer of gold which had thickness of 15 nm. Which would help us prevent the melting of sample. The sample was kept under electron beam for the lowest time possible and the pressure was kept at 10^{-7} torr.

4.5 Electrical testing

In previous chapter the methodology to carry out electrical testing was discussed and circuit diagram is also given in the subsection in previous chapter. The MOSFET that will be used is IRF3808 along with TO-220 heat sink. They were bonded with the help of a screw. 5 different MOSFETs were prepared as discussed in the previous chapter.

The TIM in all 5 scenarios were applied on heat sink surface using silicon spatula and the uniform thickness was kept at approximately 0.21 mm. values of electrical parameters can be seen in table below

Table 4-1 Electrical parameters of circuit

Component	Symbol	Value
Voltage source	V_{in}	5 V
Capacitor	C	100µF
Inductor	L	100mH
Resistor	R	150Ω

Input voltage of 5 volts was applied throughout the process. The frequency was applied using the frequency generator and frequency was varied starting from 150 kHz in the intervals of 10 kHz to as far as it can be taken without causing the MOSFET failure. After every 10 kHz increase, the temperature would be given several minutes to let the temperature of both MOSFET and heat sink settle at a single point.

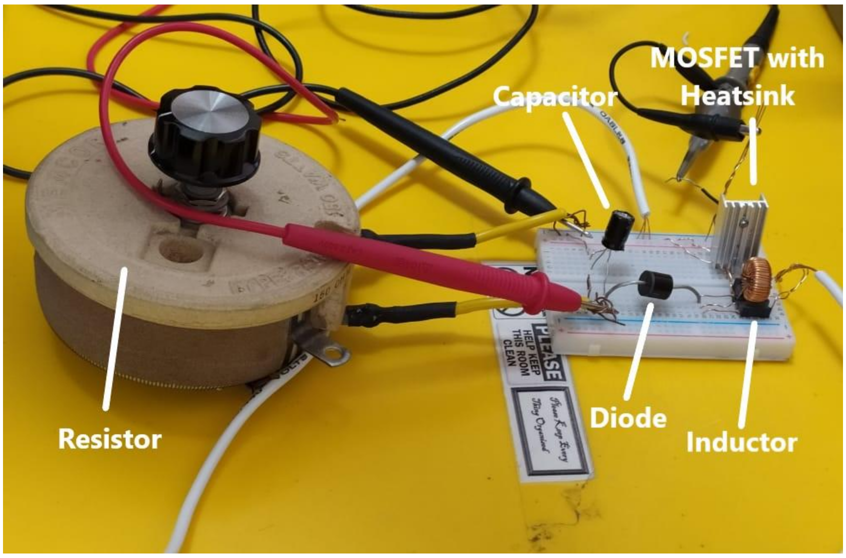

Figure 4-4 Circuit image

Thermal data was recorded with the help of thermal imaging camera (FLUKE Ti400). Two pictures were taken at every frequency interval. Which would provide us with temperatures at center point and the file would also tell us about the temperature distribution on the surfaces of both MOSFET and heat sink.

Electrical data would also be recorded manually by looking at the current readings on digital multimeter. This data will help us evaluate conduction losses and study dependency of good thermal management on minimizing conductivity heat losses.

Summary

This chapter is in continuation with the previous chapter as this chapter includes practical work that is done to fulfill the part discussed in methodology. The three samples were first prepared using stirring, mixing and sonication techniques. Then to characterize its thermal properties. Thermal constants analyzer was used which would provide us with the values of thermal conductivity, thermal diffusivity and volumetric heat capacity. Followed by different characterization techniques such as AFM to study surface roughness, use to SEM to study morphology of samples prepared on microscopic levels. And an electrical circuit based on boost converter was prepared in lab to study the effect of five types of TIM. Electrical values of all the components was known and the circuit was run, and the frequency was increased until the MOSFET failed. Until it failed thermal and electrical data was recorded using thermal imaging camera and digital multimeter.

Chapter 5: Results and Discussion

In this chapter results obtained from the experimentation mentioned above will be discussed. Which include the thermal properties, viscosity, morphology of the material the surface roughness and the most important observations and results from the electrical testing will be discussed in detail in upcoming sections of this chapter.

5.1 Thermal properties

Thermal properties were analyzed by placing the material in an apparatus called thermal constant analyzer. The experiment was run three times to ensure uniformity and reproducibility of results. The apparatus provides us with three parameters thermal conductivity, thermal diffusivity, and volumetric heat capacity all three results are discussed in detail below.

5.1.1 Thermal conductivity

Thermal conductivity is the most direct parameter that represents the ability of any material to conduct heat. Three runs were performed for direct measurement of thermal conductivity. All three runs for sample 1 are provided in table given below. Taking the mean of all the three values obtained from three runs, it comes out to be 22.82 W m^{-1} K^{-1} for sample one. Since it is provided in literature that oxidation of gallium causes its thermal conductivity to drop from 29.3 to 13.1 W $.m^{-1}.K^{-1}$ Calculating the percentage, it turns out that the thermal conductivity increase by 74.2 % as compared to thermal conductivity of starting material. One addition of 10 wt% tungsten microparticles. While for sample 2 the thermal conductivity increased from 13.1 to 16.91 $W.m^{-1}$ K^{-1} on addition of 45 wt% tungsten micro particles.

Table 5-1 increase in thermal conductivity

Sample ID	Thermal conductivity (W/m.K)		Percentage increase
	Oxidized Gallium	Sample	
1	13.1	22.82	74.20
2	13.1	16.91	29.08

Both samples discussed above show the rise in thermal conductivity with the addition of high conductivity tungsten filler particles. This change has been driven by the high thermal conductivity of tungsten (173 W m^{-1} K^{-1}). Which on addition to gallium. Form a mechanical bond with tungsten particles without reacting chemically and forming an alloy with helps in the development of a thermal path with high thermal conductivity. That provides an alternative path for heat to flow. That particular path offers low thermal resistivity as compared to the whole material and the softness of gallium also plays an important role in developing a good contact with the heat source and heat sink surfaces. Teflon was used during the testing phase because of its low thermal conductivity. Which makes it a good insulating material that does not allow the heat to escape. The accuracy of Thermal constant analyzer is <5%

5.1.2 Thermal diffusivity

Thermal diffusivity is a measure of how quickly heat diffuses through the material. Therefore, it represents heat dissipation qualities of a substance. In both of our samples, an increase in thermal diffusivity of both samples were recorded. Sample 1 thermal diffusivity increased by 44.8% while in sample 2 thermal diffusivity could only see a rise up to 31.7 percent. Which depicts that sample 1 is better at offering low thermal resistance as compared to sample 2.

5.1.3 Volumetric heat capacity

Volumetric heat capacity can be described as the amount of heat required to raise the temperature of a unit volume by 1 K. it also is a measure of heat conductivity as better thermal conducting materials possess low volumetric heat capacity. In our samples, volumetric heat capacity observed a decline of 31 % for sample 1 and 24% for sample 2 as compared to volumetric heat capacity of gallium. Which again supports the argument that sample 1 is better at conducting heat as compared to

5.2 Viscosity

Viscosity change was observed on visual basis. As adding microparticles to liquid metal gallium does increase its viscosity a

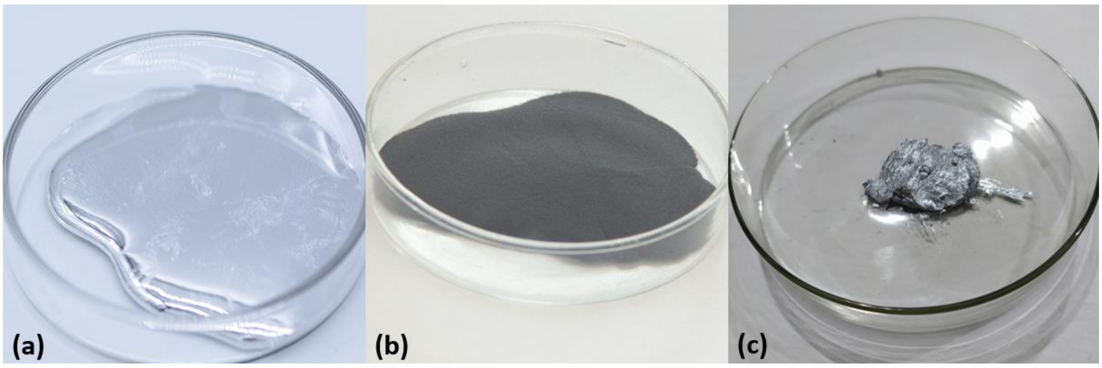

Figure 5-1 (a) Gallium (b) Tungsten microparticles (c) TIM prepared

During the electrical testing. Temperatures were raised very high and MOSFET was vertically mounted with the heat sink. And throughout the testing of all three samples prepared. The material did not flow out to cause short circuit in the circuit board. Even when temperatures were raised as high as 308 degrees Celsius. Which clearly indicates that the viscosity has increased enough to make this material suitable for use in actual electrical circuits.

5.3 Morphology

Below given are the SEM images of the 3 samples that were prepared with different compositions. the concentration of tungsten. While some of the gallium can still be seen in the background.

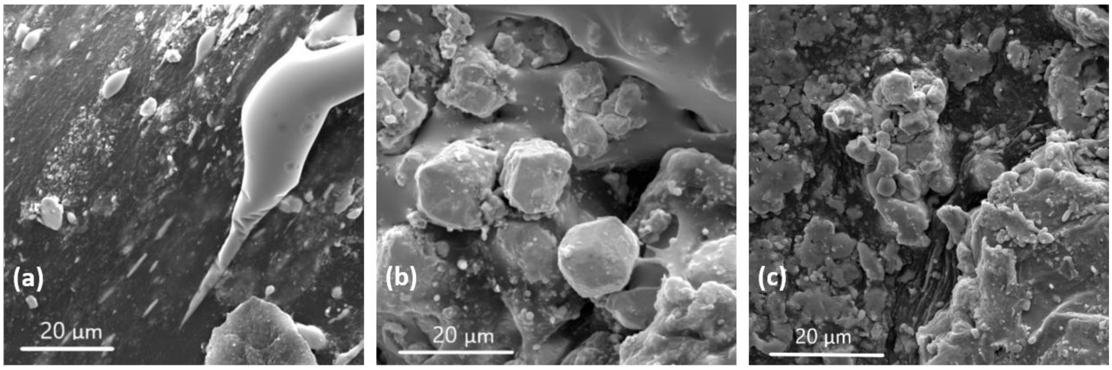

Figure 5-2 SEM images (a) Sample 1 (b) Sample 2 (c) Sample 3

The results show that the tungsten particles are fully covered with liquid metal after mixing indicating that the wettability of tungsten particles with gallium is good which may effectively improve the phonon mismatch between heterogeneous materials. The 3 samples vary in the amount of tungsten that they have in them. Fig(a) shows the sample with the lowest concentration of tungsten, Fig(b) shows an increase in the amount of tungsten. Fig (c) shows the sample with the highest concentration of tungsten

It is observed that with the increase in the amount of tungsten and reduction in the amount of gallium, the morphology of tungsten microparticles become prominent which means physical properties of tungsten also dominate.

5.4 Surface roughness

Surface morphology of MOSFET was evaluated using Atomic Force Microscopy over the area of 25 µm * 25 µm. To evaluate the extent of irregularities. That would eventually help us in choosing the size of tungsten micro particles.

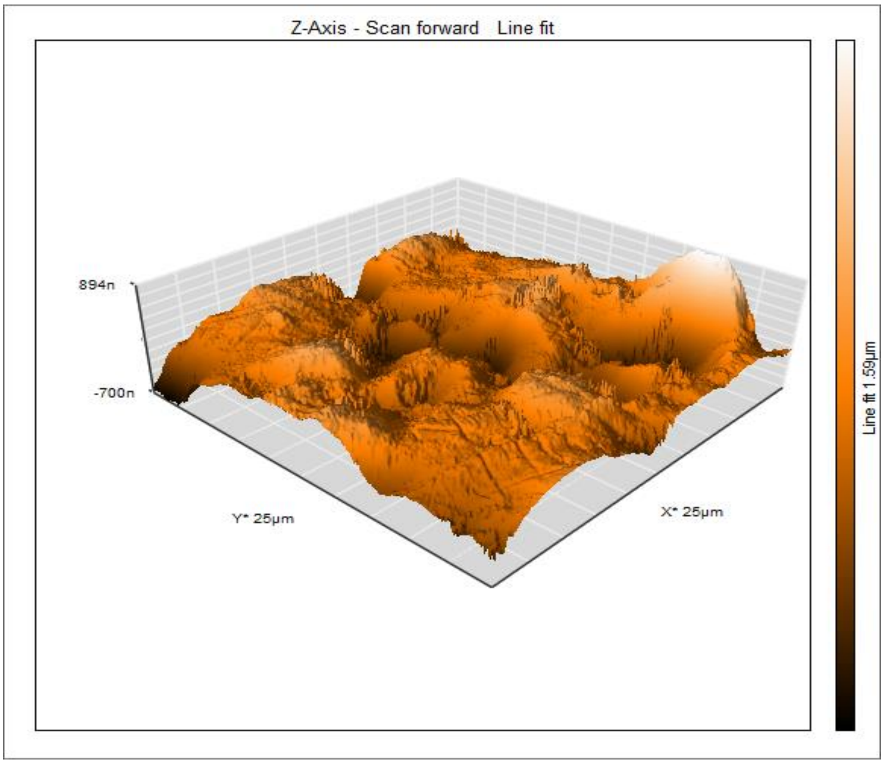

Figure 5-3 AFM image of MOSFET surface

5.5 Electrical testing

Different thermal interface materials were used to evaluate the behavior of MOSFET. Several parameters like frequency, temperatures at the surface of MOSFET and heatsink, gate current and threshold points were recorded. Temperature difference between surface of MOSFET and heat sink against frequency can be seen in graph shown below. Lower is better because lower the temperature difference between surface of MOSFET and surface of heatsink means better thermal conductivity of TIM.

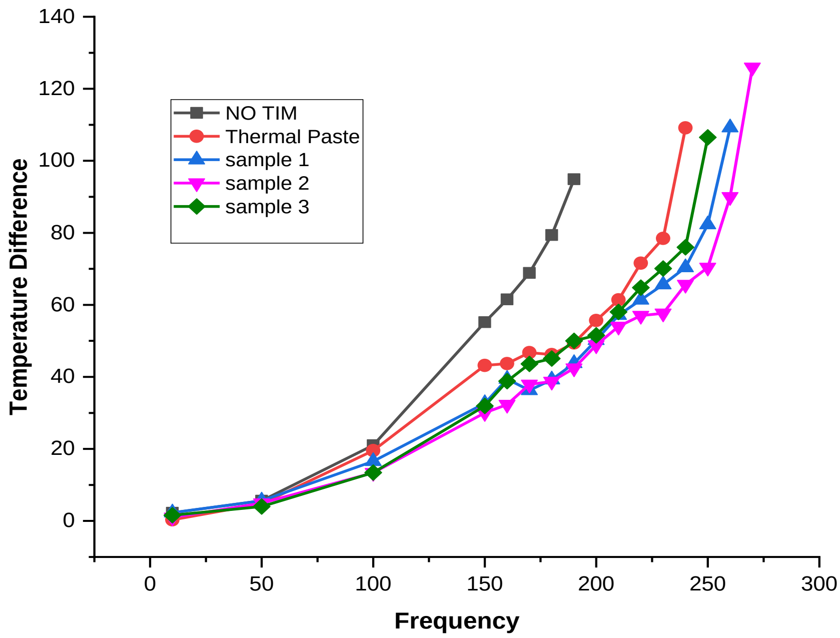

Figure 5-4 Plot between temperature difference and frequency (lower is better)

The graph shown above is based on the temperature difference between the surface of MOSFET and surface of heat sink. The lower the difference, the better the thermal interface material and its thermal conductivity. The working limits of the thermal interface materials can be clearly seen. Without any thermal interface material the MOSFET stops working at 200 KHz, while conventional thermal paste (k=0.9 W/(m.K)) manages to take the MOSFET up to 240 kHz. This clearly shows that with better thermal management switching frequency can be pushed.

Comparing our Samples that were prepared with difference concentration of tungsten. Sample 1 that had the lowest concentration of tungsten, it could only help us push the frequency upto 250 KHz, While with the help of sample 3 that had the highest concentration of Tungsten we could take the frequency up to 260 KHz, Sample 2 with the intermediate amount of tungsten helped us pushed the switching frequency oftheMOSFET upto 270 KHz which is the highest frequency that we have recorded.

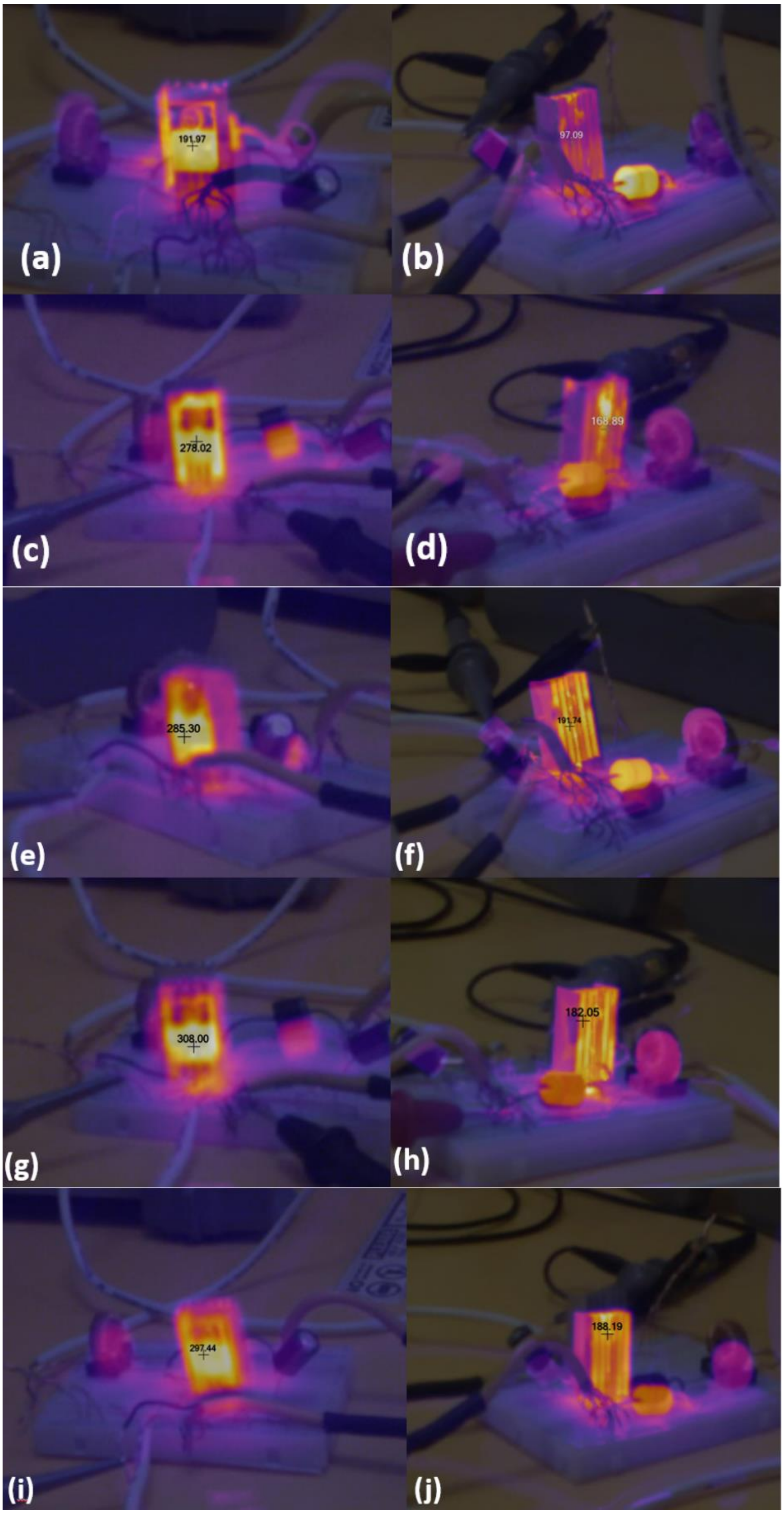

Figure 5-5 (a), (c), (e), (g), (i) Temperature at MOSFET surface (b), (d), (f), (h), (j) Surface Temperatures of Heatsink at threshold frequencies

Figure 10(a), (c), (e), (g), (i) show Temperature distribution of MOSFET surface with No TIM, Thermal grease, Sample 1, Sample 2, Sample 3 as TIM respectively. While figure 10(b), (d), (f), (h), (j) show the temperature distribution on surface of heat sink with no TIM, Thermal grease, Sample 1, Sample 2, Sample 3 as TIM respectively. Composition of each sample is given in table 4. The uncertainty associated with Thermal imaging camera is ±2 ^{o}C

Table 5-2 Threshold temperatures

	Temperature (^{o}C)	
	Surface of MOSFET	Surface of Heat sink
No TIM	191.97	97.09
Thermal paste	278.02	168.09
Sample 1	285.30	191.74
Sample 2	308.00	182.05
Sample 3	297.44	188.19

5.6 Conduction losses

To quantify the effect of better thermal management of MOSFET. A study needs to be carried out to calculate, how much difference lower working temperature makes on a MOSFET. Certain electrical parameters can be used to calculated power dissipated in the form of conduction losses. Drain source on resistance is called RDS(ON) and varies with temperature, It increases with the increase in temperature. With the help of a plot of normalized RDS(ON) against Junction temperature TJ, RDS(ON) at every temperature can be calculated. Using the formula of power dissipated

$$P=I_G^2*R_{DS(ON)}$$

While gate current IG was recorded at the time of experiment using DMM Power dissipated due to conduction losses can be calculated. Plotting the power dissipated by each thermal interface material can provide a better understanding of which sample is the most efficient to use as a Thermal interface material that minimizes the conduction losses and ensures smooth and uninterrupted operation.

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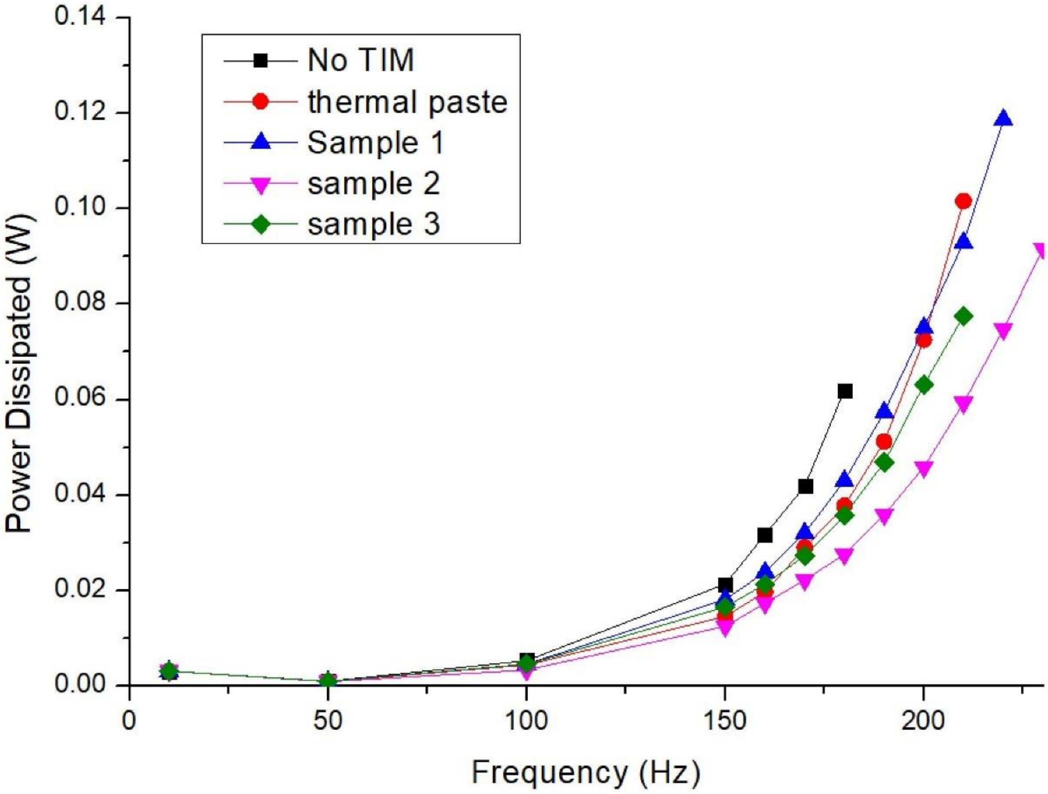

Figure 5-6 Power dissipated in conduction losses against frequency

Graph plotted of power dissipated due to conduction losses against the frequency of MOSFET. Different lines represent different types of thermal interface material we used. With no TIM the line breaks off at 180 KHz and shows the conduction loss is highest of all other TIMs used. When thermal paste is used it shows reduced power dissipated as compared when no TIM was used. And the line breaks off because the temperature was more than what was in the normalized RDS(ON) vs temperature plot. Lowest power was dissipated when sample 2 was used as TIM throughout the operation of MOSFET. Followed by sample 3 and then sample 1. All of samples showed improvement in reducing the power dissipated due to conduction losses. Lower conduction losses means improved durability and longevity of the components.

Summary

This chapter includes the details of results obtained from experimental work. These results are compulsory to validate that the proposed material has the properties that it was designed to do. The characterization included thermal properties that were obtained using thermal constant analyzer and does indicate that the material has better heat conducting properties as compared to conventional TIM and pure gallium. Morphology study showed that gallium and tungsten microparticles do form good mechanical contact which encourages heat transfer. Viscosity was evaluated on visual basic which showed that the material has higher viscosity and does not flow out from vertically mounted components even at higher temperatures. Electrical testing showed that with the help of TIM prepared with better thermal properties. It was possible to push MOSFETs frequency up to 20 kHz and on evaluation of conduction losses it was evident that using a TIM with better thermal conductivity did bring the conduction losses down.

Chapter 6: Conclusions and Recommendations

6.1 Conclusions

The suggested material was prepared and tested which showed that it has shown an increase in thermal conductivity by upto 74.2 percent as compared to the thermal conductivity of oxidized gallium and shows significantly better thermal conductivity as compared to thermal grease whose thermal conductivity is in the range of 0.8 to 1.3 W $m^{-1}K^{-1}$ while the material thermal conductivity was increased upto 22.82 W $m^{-1}K^{-1}$.

An increase in thermal diffusivity and decrease in volumetric heat capacity was recorded which represented that there is an improvement in heat conducting properties of TIM prepared. As for physical properties there was a clear increase in viscosity of TIM which addressed the fluidity issue for use in vertically mounted MOSFET. A microscopic study revealed that the contact of gallium was good with tungsten particles.

While testing the three samples prepared on the electrical circuit called boost circuit it was recorded that by using the TIM we prepared. MOSFETs frequency can be pushed upwards by 30 kHz. Which is a significant increase in performance of MOSFET. This push in frequency is made possible by improvement in heat dissipation from MOSFET.

The conduction losses were also calculated using electrical parameters. Which depicted that by better thermal management of MOSFET. We were able to achieve lower working temperatures which means conduction losses also remain lower.

In vertically mounted heat sink the material did not flow out even when the temperatures went as high as 308 ^{o}C. which means that the TIM prepared as a combination of gallium and tungsten filler particles is suitable for use in vertically mounted MOSFETs and would help improve its thermal management by improving heat dissipation. And help it achieve lower working temperatures.

6.2 Recommendations

The present study has provided a new TIM that has ability to improve heat dissipation of electronic components due to its superior thermal and physical properties. Although all the parameters are kept in mind while its development. To carry it out more efficiently there are some recommendations that are given below

1. Ceramics also possess high thermal conductivity but cannot be used due to their poor contact with gallium liquid metal. Metallization technique is use to deposit a metal on surface of ceramics that both form good bond with ceramics and with gallium due to their metallic nature.
2. Its viscosity needs to be quantified. Which would be done with the help of a rheometer. It would also help use study the viscosity behavior with the change in temperature.
3. Due to high corrosive nature of gallium, heat sink material should be selected carefully. Because of its corrosive nature it can degrade the heat sinks that are usually made of metals that can be easily corroded by gallium which can cause cracking issue and might affect the structural integrity of the heat sink over long-term use.
4. Reliability of electronic components matters a lot such as MOSFET in such case. So, use of quality components should be ensured. So, the results can be uniformly assessed.
5. A thermocouple of special type should be used at the time of electrical testing, so we have more datapoints of temperature variation with the variation in frequency.

Appendix A: Publications

Title: Thermal performance enhancement of gallium based thermal interface materials

Authors: Ali Hamza, Mariam Mahmood, Naveed Ahmed, Sehar Shakir, Abasin Ulasyar

Status: Published

Abstract: With the recent industrial boom, a sudden increase in the use of electronic components has been observed. The electronics field has seen countless inventions and improvements in recent years which resulted in significant size reductions. However, due to the increase in performance, the heat dissipated has increased. Increasing heat density of electrical components. Therefore, better thermal management is required to ensure the durability and longevity of the system. Heat management can be significantly improved by using a thermal interface material with better thermal properties which helps improve heat dissipation. In our research, We add tungsten microparticles to liquid gallium to prepare a thermal interface material that is suitable for use in electronics. The concentration of tungsten varies from 10-45% by wt in our samples. Tungsten microparticles were mixed in liquid gallium in a mortar and uniform dispersion was ensured with the help of a sonicator. After carrying out thermal analysis with the help of a Thermal constant analyzer (HOT DISK TPS-1500) it was observed that the volumetric heat capacity of our samples was reduced by 24-31% as compared to pure gallium. While its thermal diffusivity increased by 31.7-44.8% as compared to pure gallium both of the results obtained above clearly indicate that the rate of thermal energy dissipation is better than that of pure gallium.