Fabrication and Characterization of Thin Film Bridge Resistor

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2019

Fabrication and Characterization of Thin Film Bridge Resistor

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This thesis is submitted as a partial fulfillment of the requirements for the degree of

MS in Energetic Materials Engineering

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August, 2019

Dedication

I dedicate this thesis to my elder brother Muhammad Mudassir 'late' who died of stomach cancer 3 weeks before completion of this research work. He fought bravely against the disease for 10 months but ultimately had to surrender infront of Allah's Will. May Allah forgive him and bless him with highest place in Jannah. Aameen

Acknowledgements

All admirations to Allah Almighty who is the creator, maintainer and the Regulator of the world. He is the One, who bestows and gives power to us to think, utilize our expertise in knowledge in achieving remarkable solutions for the mankind in every field. Therefore, I express my greatest thanks to Almighty Allah, the universal and the architect of the world, who has gifted me the wisdom and strength to complete this research work and thesis report. As Allah Almighty says in Quran:

"Read! In the name of your lord" (Alaq; 1st revealed ayah)

This Quranic verse sums up the entire importance of the education in the lives of humans.

I would like to express my utmost gratitude to my supervisor Dr. Sarah Farrukh, who had been extremely kind and supportive throughout my research. She remained very persistent with me during my work. At many stages in the course of this research project, I benefited from her advices and recommendations, particularly when confronting new ideas. Her positive outlook and confidence in me and my research work kept on inspiring me to continue working hard till last moment. She kept on motivating me towards accomplishing my work and had been a source of constant support when I was going through multiple grave domestic problems. It was her inspirational attitude that helped me to complete my research work. Without her support, I would have not been able to do it. I am extremely lucky to have a supervisor who showed her full dedication and keenness towards my work and always responded to my questions and queries in a prompt way. It was a matter of great privilege and honor for me to work with her and I proudly say that she was not just a supervisor for me but also a mentor, an elder sister and a beacon of hope who always listened to my concerns patiently, guided me honestly and never pressurized me in undue manner. I must say that I have learnt many positive things from her way of working and her extraordinary humanitarian qualities.

I would also like to thank Dr. Abdul Qadeer Malik and Dr. Muhammad Aftab Akram for their sincere and humble guidance that led me to complete my project. Their valuable suggestions and feedback contributed greatly to this thesis. I am very grateful to all my teachers of MS degree program for making me a more knowledgeable person than what I was before.

The support and technical contribution of NINVAST team for achieving the desired results of this research work shall always be remembered. I am extremely thankful to all Dr. Khalid, Dr. Zaka and Miss Irum of NINVAST for assisting me in every possible way in synthesis of required samples.

I am also grateful to Dr. Zulfiqar Lodhi and Dr. Aziz Ullah from Air Weapons Complex, Wah Cantt for providing technical assistance as well as for timely provisioning of materials and equipment required for completion of this research.

I deeply acknowledge the efforts of SEM Lab Technician "Mr Abbas" for extending his support and help in conducting characterizations of my project samples.

I am highly indebted to Mr. Arman Liaqat for assisting me in carrying out characterization of samples, analyzing the results and preparation of power point presentation for final defence of thesis.

Last but not the least; I am extremely grateful to my family (parents, wife and siblings) for their unprecedented support. Without their prayers, I wouldn't have been able to complete this task in a befitting manner.

Abstract

Bridge-wire electric detonators are prone to malfunctioning under very high gravitational and rotational and impact forces due to breakage of resistive wire soldered between the electrodes. Globally, the resistive bridge-wire technology of electric detonators is being replaced with alternate and reliable systems. Thin film bridge resistors are known to be more rugged and reliable than bridge-wire technology. This study is aimed at exploring and fabricating thin film bridge resistor to be used in electric detonator to enhance their reliability and performance. Nickel Chromium alloy with 80/20 composition, Gold and Silver have been used individually for manufacturing of thin film resistors. Fabrication of thin films has been carried out through different Physical Vapor Deposition techniques on glass and Bakelite substrates. Scanning Electron Microscopy, X-Ray Diffraction and Atomic Force Microscopy has been carried out for characterization of thin films The electrical resistance of thin films has been determined using Two Probe Method.. A comparison of characterization and resistances of all films has been made with existing literature and also with each other to identify most suitable material for fabrication of thin film bridge resistor for electric detonators.

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Chapter 1
Introduction

“A detonator is an explosive device used in Fuzes to initiate the booster charge and then the main explosive charge” [1]. Generally, a detonator is filled with an explosive Service Lead Azide (SLA) [2] which is extremely sensitive and requires very small energy for ignition. In an electric detonator, the ignition stimulus or energy is provided to the explosive through a capacitor discharge. In between the capacitor, there exists a bridge-wire resistor. When the capacitor discharges, the current flows through the bridge-wire resistor causing it to generate heat. The heat emitted by the resistor is sufficient to initiate the explosive impressed upon it [3]. The initiation of explosive therefore greatly depends on the resistor which is connected between the electrodes through spot welding forming a bridge. A more recent technology uses resistive thin film bridge instead of bridge wire resistor in the detonators to enhance the reliability of detonators. In order to understand the utilization of thin film resistors in electric detonators, it is important to have generic understanding of explosives, detonators and thin film resistors.

1.1 Explosives

“An explosive is a material which when suitably initiated, can undergo a very fast reaction or decomposition in a self-propagating mode with the formation of more stable materials and simultaneous evolution of large amount of heat and gas to produce practical effects” [4]. Explosives can be broadly categorized as low and high explosives [5].

1.1.1 Low Explosives

These are the substances that do not undergo detonation but deflagrate i.e they burn at a relatively slow rate [6]. They generally contain their own oxidant for combustion and produce high pressures as a desired product.

1.1.2 High Explosives

"High explosives consist of materials that typically combine the reacting elements in the same molecule. This allows them to react much faster, and they detonate. Detonation involves supersonic shock waves that pass through the material, causing chemistry that happens quite faster than burning" [6].

Since the detonators contain only high explosives, therefore, further elaboration of only high explosives is presented here. Depending upon the figure of insensitivity, initiating mechanism and the output power produced, the high explosives can be sub-divided into primary and secondary explosives.

1.1.2.1 Primary Explosives

These are the explosives that are very sensitive to heat, friction, electric spark and shock wave and can be readily detonated when provided with small stimuli. However, they do not produce much power upon detonation. These explosives have the capability to readily transform burning into detonation and produce shock wave to further initiate relatively less sensitive explosives [6].

1.1.2.2 Secondary Explosives

These explosives are relatively less sensitive to heat or shock and are not readily detonated by such stimuli however, once detonated they produce much more power than the primary explosives. Secondary explosives can be initiated to detonation only by the shock wave. The shock wave required for their detonation is generated by the detonation of primary explosive [6].

1.2 Explosive Train

Since the powerful secondary explosives are relatively less sensitive and can be detonated only through shock wave, they require a primary explosive in series with them during initiation process. The primary explosive gets initiated when a small stimulus is provided to it and produces shock wave. This shock wave then initiates the secondary explosive kept next in line. This arrangement of explosives for initiation of secondary high explosives is called explosive train [7]. An explosive train comprises of three stages of high explosives. At the beginning is the primary high explosive (initiator) followed by

the booster and then the main charge. The primary explosive, being very sensitive to heat or shock, is initiated by some external stimuli. This produces a shock wave which then initiates the moderately sensitive booster charge. The booster charge magnifies the shock wave and forwards it to the least sensitive but most powerful main charge. The main charge detonates by the incoming shock wave and produces a very powerful shock accompanied with loud noise, heat and light to generate the desired destruction effect.

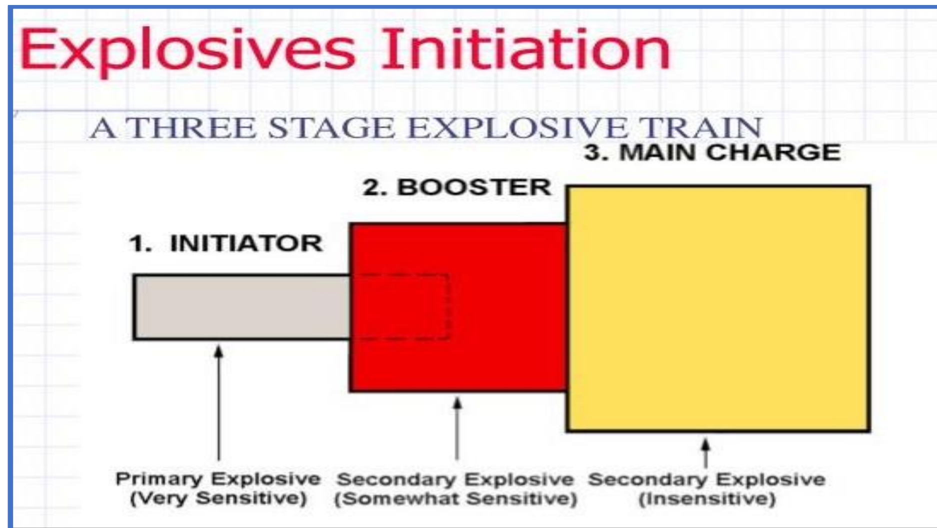

Figure 1: Schematic Diagram of Explosive Train

1.3 Detonator

A detonator is an explosive device used to initiate an explosive charge [1]. A basic electric detonator consists of a cylindrical metal case usually made of aluminum or copper filled with primary explosive (Lead Azide or Lead Styphnate) pressed onto base charge. On top of the primary explosive, a layer of priming or ignition charge is pressed. A pair of leg wires are inserted into the tube passing through an insulating header that physically and electrically isolates the two legs from each other. The insulating header also ensure that the detonator remains water or moisture free. The leads / wires are joined together with the help of a resistive bridge-wire. The priming charge is pressed onto the resistor or the resistive bridge-wire. Once the entire set is inserted into the metal case, it is crimped near the opened end [8]. A cross sectional view showing construction of an electric detonator is given in figure below.

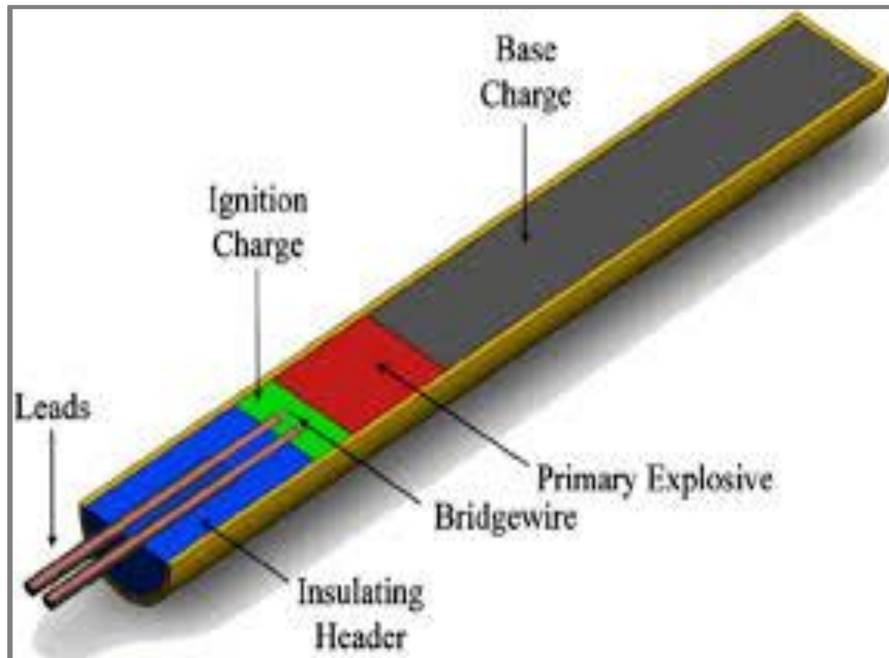

Figure 2: Cross Sectional View of Electric Detonator

1.3.1 Working Principle of Detonator

In order to initiate an explosive charge, the detonator is fitted into an explosive charge. The detonator is connected to power supply or to non-electric input source that provides the required amount of current/ non-electric stimulus to initiate the priming charge within the detonator. The external stimulus heats up the priming charge up to its ignition temperature thus causing the explosive to initiate. The ignition/priming charge in-turn initiates the primary explosive and the detonation train continues until base charge is detonated [8].

1.3.2 Features of Detonators

Since the detonator plays a significant role in the detonation train, the detonator must meet certain design features. Some of the characteristics are generic which must be fulfilled by all detonators however, few of them are specific that are dependent upon application of detonator. The standard requirements / characteristics of an electric detonator are [9]:

1.3.2.1 Correct Functioning

The most significant requirement of a detonator is the correct functioning. This includes sensitivity of the detonator only to the correct external stimulus to initiate the primary explosive and generate shock wave of sufficient magnitude to initiate the next in line high explosive.

1.3.2.2 Small Size

With the development of sophisticated weapons, the sizes of sub-assemblies play vital role in defining the weight of the main assembly and in enhancing / reducing the range of the propelled weapons. The detonator must be small in size and compact in design to easily fit into the designed cavity in weapons or fuzes.

1.3.2.3 Safe Handling & Transportation

Safety of the equipment and operator is of paramount importance while handling with explosives. The detonator must fulfill the standard safety requirements and must be safe to handle, store and transport from one place to another.

1.3.2.4 Fast Response

The response time of a detonator's initiation upon receiving the input signal is extremely critical especially during application in missiles and in other weapons delivered at long ranges. Undesired initiation delay of detonator by even few milliseconds can result in missing the target or failure in successful detonation. So, the detonator must respond to the input signal within microseconds to generate the required shock wave.

1.3.2.5 Long Life

The detonator must have long shelf and considerable service life to avoid wastage due to low life span.

1.3.2.6 Low Cost

Since the detonators have many military and commercial applications, its high consumption demands that it must be cheap to purchase.

Besides the above stated standard requirements, the electric detonators also have certain special features that are dependent upon the mission specifications. Some of the special characteristics of an electric detonator used in hypersonic velocity missiles are [10]:

1.3.2.7 Low Input Energy

One of the special requirements of a detonator used in high velocity missiles is low input energy for its initiation. This allows smaller power units to be used for said purpose and hence reduces the payload and helps in attaining high speed and long range.

1.3.2.8 High 'G' Resistance

A hyper sonic missile experiences 'G' force upto 100,000 g. The detonator must withstand the 'G' force experienced by the missile and must not break or malfunction at such special environments.

1.3.2.9 High Spin Resistance

Similar to high G force, a high-speed projectile also experiences a very high spin rate. As a special requirement, the detonator must withhold the force experienced by the detonator due to high spin rate.

1.4 Drawbacks of Bridge-Wire Resistor in Detonators

Owing to the specified application, the detonators may be required to operate in very harsh conditions called as special environment. The harsh conditions may be of extreme temperature or exertion of extra physical pressure on the detonator. In general, temperature related issues are very rare to occur as the detonator is tightly enclosed in its bigger sub-assembly in such a way that it does not get effected by ambient temperature. However, the probability of encountering tough physical conditions, such as high impact pressure, is relatively more for the detonators once it operates in special environments. The physical connection of resistor (bridge-wire) with the electrodes of detonator is made by spot-welding the wire to the electrodes. The quality or strength of this connection depends upon the type of material used as well as on the expertise of the operator. Not all bridge-wire resistors possess same strength at junction points even if they are soldered by same person using same equipment. The bridge-wire resistors tend to have certain kinks at the electrode's junction due to welding process which can cause

them to break under special environment. Therefore, the bridge-wire resistor of the detonator is quite sensitive to shock due to extremely high intensity impact which can exert a very high gravitational force on the detonator and thus results in breakage of the wire from the connection point. Similarly, bridge-wires are also sensitive to rotational force due to high spin rate which also cause similar phenomenon of wire breakage. Since one of the major applications of detonator is in ultra-high speed missile, the bridge-wire detonator experiences all the above-mentioned phenomenon. If a bridge-wire electric detonator is used in such applications, its resistive wire gets prone to breakage thus causing failure of ignition system. This further causes failure of complete detonation train and thereby rendering the entire munition useless.

1.5 Thin Film Resistor Technology

"A thin film is a layer of metallic or semiconductor materials formed by atom to atom or molecule to molecule condensation process, with its thickness ranging from few nanometers to several micrometers depending upon the field of application" [11]. Thin films and the devices made of thin films play a vital role in the advancement of modern-day technology. Thin films and coatings offer functional or decorative characteristics to modify surface properties of substrate such as resistance against corrosion, electrical conductivity, sheet resistance, reflectance and adhesion etc. [12]

"The resistor which uses metal film to limit the flow of electric current to certain level is called metal film resistor". In modern day world, thin film resistors find wide number of applications in various fields especially in micro-electronics industry, optoelectronics, aerospace Industry and military equipment [13].

1.5.1 Construction of Basic Thin Film Resistor

A basic thin film resistor comprises a substrate material of insulating nature onto which a metallic thin film is deposited. The thin film offers resistance to the flow of current, thereby serving the purpose of resistor.

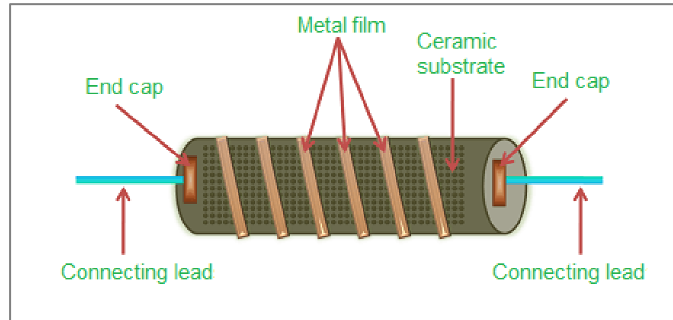

Figure 3: Metal Film Resistor

The metal film which is deposited on insulating substrate is connected at both ends with electrodes. The resistance of the film is dependent upon the length and cross sectional area of the film. In order to vary the resistance, either the thickness is altered or the width of film is changed by cutting the film in helical pattern along the length by using laser.

1.5.2 Key Electrical Parameters

Three major electrical properties of a thin film material are sheet resistance, temperature coefficient of resistance and resistance stability [14].

1.5.2.1 Sheet Resistance

"The sheet resistance, expressed as ρ/d, is the resistance offered by the thin film material which is directly proportional to its resistivity ρ, and is inversely proportional to the film thickness d."

Resistance, $R = \rho l/ dw$

since, $l/w = 1$ (for square patterns)

$Rs = R = \rho/d$

1.5.2.2 Temperature Co-efficient of Resistance

"The change in resistance per degree change in temperature is called temperature co-efficient of resistance (TCR)." The need to define this parameter aroused because resistance is a function of temperature; and a change in temperature causes an increase in the length of wire and decrease in its cross-sectional area thereby causing a change in resistance. To account this variation in resistance, TCR is define. TCR can be positive or

negative. As a general rule, TCR is positive for pure metals and negative for semi-conductors.

1.5.2.3 Resistance Stability

"It is the change in the resistance of a material kept under certain conditions for a long period of time." The lesser the variation in resistance, the more is the resistance stability or reliability.

1.6 Research Objectives

Thin films resistors are known to have relatively higher strength by virtue of their own physical characteristics than the bridge-wire resistors. Thin films, if properly spread over a surface, provide uniform physical and electrical characteristics. Unlike bridge-wire, thin films do not require any soldering junction with other surface and are rather spread over the surface in a layered manner. The absence of soldering makes them free of kinks and loose connections. The primary objective of this research work is to transform a bridge-wire electric detonator, developed by Air Weapons Complex Wah Cantt for use in hyper-sonic velocity missile, into thin film detonator. For this purpose, the basic aim is to carry out literature survey and find out the compatible materials being used internationally for manufacturing of resistive thin film bridge in detonators. The compatibility assessment would be carried out by first conducting feasibility study on choosing a material that exhibits low resistance of about 2Ω to 3Ω once deposited as thin film. Subsequently, electrically insulating material for substrate would be searched out over which thin film can be grown. Both the materials should have good physical compatibility with each other such that the substrate provides sufficient adhesion to the film deposited upon it. After the feasibility study for desired materials, thin film shall be deposited upon the substrate using suitable technique and their electrical parameters would be determined. The characterization of thin film would then be carried out to notice different features of thin film resistor. As a second phase of the project (to be conducted later), the manufactured thin film resistor would be used in an electric detonator to replace bridge-wire, followed by practical testing of the detonator to observe the firing parameters.

1.7 Thesis Outline

Chapter 1 of this thesis gives an introduction to explosives, detonators, thin films resistors technology, explains the drawbacks of bridge-wire detonators, and the research objectives. Chapter 2 encapsulates the literature survey on thin resistive films made of different materials and their application on electric detonators. Chapter 3 gives details of the selection process of materials and the techniques used for deposition and characterization of thin films. Chapter 4 covers the details of experimental work carried out for deposition of thin resistive film on different types of substrates. Chapter 5 presents the results of different characterization techniques and the resistance values achieved films followed by analysis of the films, conclusion and future recommendations.

Chapter 2
Literature Review

2.1 Frequently used Substrates

Joachim Rolke [15] in his article highlighted certain merits and de-merits of some of the most commonly used substrates. While talking about a particular thin film, he said that the output of a thin film deposition is significantly influenced by the substrate properties such as surface roughness, chemical composition and texture etc. The choice of the substrate is based on parameters like power dissipation, TCR, cost, and required stability etc. Therefore, the substrate with most suited parameters for desired application is opted. Some of the substrates with their pros and cons are appended below:

2.1.1 Borosilicate Glass

It offers high degree of uniformity and resistance reproducibility. The material has a low TCR value, provides high resistor stability and low thermal conductivity. However, it has relatively low mechanical strength but has an advantage of having low cost.

2.1.2 As Fired Thin Film Alumina

It offers high thermal conductivity and mechanical strength but suffers from low uniformity, reproducibility and stability of resistor parameters. The material has fairly high cost.

2.1.3 Glazed Alumina

Most of the properties and cost of glazed alumina lie between as-fired alumina and glass however it has reduced mechanical strength due to glaze.

2.1.4 Oxidized Silicon Wafers

Although these materials give high performance however, they have a disadvantage of being expensive.

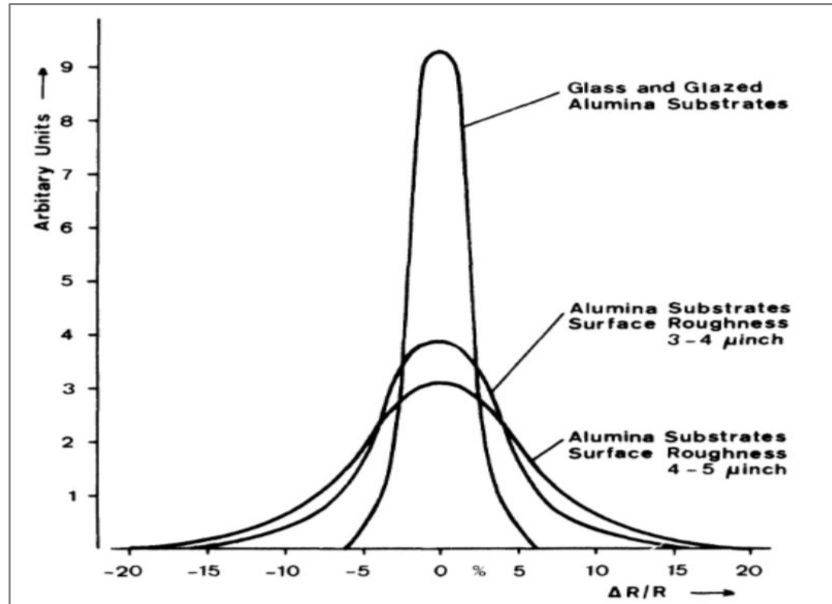

Figure 4: Effect of Surface Roughness on Resistor Stability and Reproducibility [15]

2.2 Review on Resistive Thin Metallic Films

Thin film resistors are being manufactured by a wide variety of metals, alloys and semiconductors however, only metals and metal alloys are known to give low resistance value as per our requirements. Therefore, the literature survey of only metallic thin films has been carried out.

T.H Gilani and Dian Rabchuk [16] developed thin film of gold on glass substrate and studied the variation of electrical resistivity with the thickness of film. Thin films of variable thickness were deposited under high vacuum using thermal evaporation technique and their resistivity values were monitored at room temperature using the four probe DC method. A decrease in resistivity was observed with the increase in thickness of the film which ultimately reached the bulk resistance of gold (2.20×10^{-8} Ω.m) as the thickness was continually increased. The smallest value of attained resistivity was $2.49 \pm 0.3 \times 10^{-8}$ Ω.m for which the thickness of the film was 66 ± 1 nm. The bulk value of the resistance was achieved when the film thickness was kept at about 38 ± 1 nm. The mean free path for gold electrons to conduct at 25 ^{0}C is 37.7 nm. Therefore, it was observed that, by increasing the film thickness up-to or greater than the mean free path, the bulk and the thin film resistivities become equal. The graph below shows the pattern of

decrease in resistivity with increase in thickness. The resistivity becomes almost constant as the thickness becomes equal to or greater than the mean free path of the electrons (37.7 nm).

Sebastian Bahamondes et al [17] worked on resistivity determination of gold thin films prepared on mica substrate at room temperature. Multiple films of variable thickness between 8 to 100 nm were prepared using thermal evaporation and a comparison of resistivity values at different film thicknesses was made. The values obtained were plotted on graph for ease in understanding the trend of inverse relation between the two parameters.

A. Chaloupka et al [18] conducted a study on "influence of substrate on properties of gold nanolayer". Thin Films of gold were prepared on glass and PE substrates through sputtering and the effect of substrates on various properties were determined. The effect of deposition time on film thickness was monitored and it was observed that the thickness increases by increasing the deposition time. The sheet resistances of the films, measured by two-point technique, revealed that with increase in deposition time above 30 and 45 seconds for glass and PE substrates respectively, the sheet resistance undergoes a drastic decrease. The decrease in resistance is less drastic on PE substrate than on glass due to higher initial surface roughness of PE. A higher tensile stress in observed for the film deposited on PE substrate. The grains deposited on glass are smaller in size than those on PE and exhibit relatively small micro-strain. It was concluded that the gold film deposited on glass was better and more stable than on PE substrate.

Jahanbaksh Mashaiekhy et al [19] prepared silver thin films on glass substrate using DC magnetron sputtering technique and explained the effect of film thickness and substrate temperature on surface morphology, electrical and structural properties of the film. It was observed that by increasing film thickness or the substrate temperature, the electrical resistivity of silver thin films decreases and vice versa. Multiple Thin films with variable thickness prepared at 50°C were subjected to resistivity measurement. The highest resistivity of 4$\mu\Omega$.cm was observed for a film of 100nm thickness whereas the

lowest resistivity of 2.7μΩ.cm was obtained for a film with thickness of 800 nm. The bulk resistivity of silver is 1.67μΩ.cm and the rise in resistivity from bulk to 4 μΩ.cm was due to lower thickness of the film.

W.R.Hardy et al [20] carried out reactive sputtering of nichrome alloy in O_2 using DC diode sputtering technique and measured electrical parameters of the resistive nichrome films. The resistivity of films was between 150 to 300 μΩ-cm by varying partial pressure of oxygen.

Imam H. Kazi et al [21] conducted a study and performed experimental work on the effect of film thickness on its resistance. Nichrome thin films of various thickness were prepared on glass substrate using RF Magnetron Sputtering technique using Argon as sputtering gas. The thin films were coated with films of Al_2O_3 having thickness of 0.2 μm, sputtered through RF magnetron using 90% Argon and 10% O_2. Very high sheet resistance, upto 183 Ω/sq were observed for films with very low thickness 0.025 μm. The resistance of the films decreased with increasing thickness.

Cheng-Hsien Lin et al [22] made an effort to identify materials and methods for enhancing thin film resistivity of NiCr alloy. The team prepared thin films of two different materials and determined their electrical properties. Firstly, thin film of NiCrMn was prepared using DC and RF magnetron co-sputtering technique in which NiCr film was sputtered using DC magnetron and Mn was sputtered using RF technique. A resistivity value of 400 μΩ-cm was achieved when NiCrMn was annealed to 300 ^{o}C. Secondly, NiCrMnZr thin films were prepared using same technique in which NiCrMn was sputtered using DC Magnetron through NiCrMn while Zr was sputtered through RF technique. The resistivity of NiCrMnZr films having 16.7% Zr annealed at 300°C was around 510 μΩ-cm and showed TCR of 53ppm/°C.

Ruei-Cheng Lin et al [23] studied the electrical behavior of resistive thin film of Ni-Cr-Si-Al-Ta under different sputtering powers of 100W and 200W and different annealing conditions. Thin films of Ni-Cr-Si-Al were prepared on two different substrates i.e glass and Al_2O_3 using co-sputtering technique. Thin films on glass were prepared for carrying out characterization of the films while Al_2O_3 substrate was used for observing electrical

parameters. When sputtering power of 100W and annealing temperature of 300°C was used, the films exhibited amorphous structure. The films sputtered with 100 W and annealed at 300°C displayed lowest TCR of -10 ppm/°C and a resistivity of around 2200 μΩ-cm.

Lifei Lai et al [24] conducted a study on “comparison of microstructure and electrical properties of NiCr alloy thin film deposited on different substrates”. Thin films of Nichrome alloy comprising of 80% nickel and 20% chromium were grown on three different types of substrates using DC Magnetron sputtering method. The substrates chosen for said purpose were copper foil, glass and silicon. All the films were annealed at different temperatures and their sheet resistance and resistivity values were monitored and compared. Before annealing, the sheet resistance and resistivity values of NiCr films of similar thickness grown on glass and silicon substrates had close approximation however, copper foil exhibited the resistance and resistivity values equal to three times of that on the other two substrates. A decreasing pattern in sheet resistance and resistivity was observed with the increase of annealing temperature. XRD analysis of the films on all three substrates indicated that hexagonal Ni (011) and Ni (103) and face-centered Cr (110) and Cr (200) peaks were observed on copper foil whereas only face-centered Ni (111) peak was observed on both other substrates before annealing.

Nurul Khalidah Yusop et al [25] highlighted the “Structural and Electrical Characterizations of CuNi Thin Film Resistors”. Multiple thin films of CuNi with variable thicknesses ranging from 102 to 126.8 nm were grown on FR4 PCB substrate utilizing thermal evaporation method. The composition of copper was changed while preparation of every film. It was observed that the composition of thin film remained same as that of the original material. Moreover, the electrical properties of thin films varied in accordance with the concentration of copper content in the depositing material. Sheet resistance between 1 to 6 Ω/sq and peak resistance of 50 Ω were recorded while the structure exhibited by the films was beehive-like.

2.3 Application of Thin Resistive Films in Electric Detonator

Thin film resistive materials having reasonable resistivity and low temperature coefficient of resistance (TCR) are of countless significance for manufacturing of military products. One of the applications of thin film technology is in electric detonators where it is used as a thin resistive film bridge for initiation of explosive in a detonator [26]. Research in this specific field has been carried out worldwide however, since it is a classified field, very little data is available openly in literature. Some of the publications pertinent to thin film bridge detonator are reviewed in subsequent paragraphs.

Jenq-Huey Shuy et al [27] conducted a study to prepare thin film bridge detonator and used it in place of bridge-wire detonator to enhance the rigidity and reliability of the detonator. The study was further aimed to identify the effect of parameters such as bridge shape and resistance on the firing properties of the detonator. A thin film of nickel chromium alloy, also known as Nichrome, with composition of 80% Ni and 20% Cr was prepared by DC magnetron sputtering technique on glass to metal seal (GMS). Multiple samples with different bridge area, length and thickness were prepared and the value of resistance as well as the firing time of each sample was monitored. The firing time of the detonator with bridge resistance between 1.1Ω to 1.4Ω lied in the range of 180～200μs. The shortest firing time of 120μs was observed when the bridge resistance was 3.165Ω. It was deduced that besides the resistance, the main factor influencing the firing time is the bridge area.

Chapter 3
Materials and Methodology

3.1 Material Selection

Deposition of thin film primarily requires two main ingredients; a target material and a substrate. The target material is the one that is to be deposited in the form of thin film while substrate is the material onto which the thin film is deposited [28]. After thorough survey of literature, the target materials and the substrates that were short listed for manufacturing of thin film resistors are appended in subsequent paragraphs.

3.1.1 Thin Film Material

Sequel to finalization of objectives of project, the most critical task is the selection of the target material for deposition of thin film. A number of metals and metal alloys were considered for this purpose to achieve rugged, reliable and stable thin films with relatively high resistance values. The hardness of any material is determined from Mohs scale. This scale ranks the materials out of 10 according to their hardness. The hardness of the hardest known material (diamond) is 10 while that of plastics and pencils is 1 [29]. The Mohs scale of hardness provides fair idea of comparison between hardness of different materials. Some of the important properties of metals and alloys given in table below:

Table 1: Depositing Materials' Properties

Material	Resistivity (μΩ/cm)	TCR (α/°C)	Melting Point (°C)	Hardness (Mohs)
Gold	2.44	0.00371	1064	2.5
Copper	1.68	0.00386	1085	2.5-3
Silver	1.59	0.00380	961.8	2.5
Aluminum	2.65	0.00429	1220	2.75
Tungsten	5.60	0.00450	3442	7.5

Molybdenum	5.7	0.004579	2623	5.5
Nickel	6.99	0.005866	1455	4.0
Chromium	12.9	-	1907	8.5
Tantalum	13.5	-	3017	6.5
NiCr (80/20)	110	0.0004	1400	-
Constantan	49	-0.000074	1210	-

Out of different options considered, the materials finally chosen for manufacturing of thin resistive films were 'Nichrome Alloy', 'Gold Metal' and 'Silver Metal'.

3.1.1.1 Nichrome Alloy

Nichrome (80% Ni, 20% Cr) is a transition metal alloy being used extensively in microelectronic industry, especially as thin film resistor due to its low temperature coefficient of resistance, relatively high resistivity, high thermal and electrical stability, and oxidation resistance [30]. The material also encompasses the feature of altering its resistivity by varying the concentration ratio of the two ingredient metals. Moreover, the resistivity of NiCr can be further modified by adding some other metal or semiconductor into this alloy. NiCr thin films are known to be rugged and reliable due to small TCR. It is one of the most frequently used materials in manufacturing of thin film bridge detonators. Besides the technical features, Nichrome is also readily available and a relatively cheap material.

3.1.1.2 Gold Metal

Gold also exhibits good resistive behavior in the form of thin film. Although the resistivity of gold thin film varies more sharply with thickness as compared to Nichrome but the variation is not as abrupt as of other metals [31]. Even though Gold is an expensive material but the required resistance of 2-3Ω with gold thin film can be achieved with much less thickness than Nichrome films which makes Gold a cost effective option in large scale production. Moreover, Gold is also a readily available material.

3.1.1.3 Silver Metal

Low TCR, cheap price and ease of availability of silver make this metal also a good option for utilization in thin film resistor fabrication. Although the resistivity of Silver is less as compared to Gold and Nichrome however, the resistance values of can be tailored according to requirement by varying the thickness or geometry of thin film.

3.1.2 Substrate Material

The selection of substrate is as important for effective thin film production as the target material itself. However, the choice of substrate is dependent upon the material to be deposited in the form of thin film as well as the intended application. Since the film was required to be developed for harsh and special environments where it could sustain high gravity load and high speed rotational effects, therefore the substrate was to be chosen accordingly which provides sufficient strength as well as adhesion to the film. The film to be deposited was supposed to be of conducting material and an electrical isolation between the film bridge was required to be established therefore, it was essential to choose a substrate that is of electrically insulating nature. Moreover, it was also important that the substrate does not compromise on strength, adhesion and quality of thin film. The options considered with some of their important properties are tabulated below:

Table 2: Substrate Materials' Properties

Material	Conduction Characteristics	Melting Point (°C)	Hardness (Mohs)
Quartz Glass	Insulator	1713	7
Borosilicate Glass	Insulator	1648	6-7
Soda Lime Glass	Insulator	573	5.5
Alumina	Insulator	2072	9-9.5
Bakelite	Insulator	250	1

Considering multiple options, the best choices as evident from the table above is quartz glass and alumina as these materials have high melting points and are quite hard to withstand the impact effects. However, these materials could not be arranged due to scarce resources. Borosilicate can be an equally compatible material in place of alumina and almost all lab utensils are made of Borosilicate glass however, the availability of this material in substrate form was also a question. Therefore, keeping in mind all the technical and managerial aspects, the materials that were chosen as substrates are 'Soda Lime Glass' and 'Bakelite'.

3.1.2.1 Soda Lime Glass

Soda Lime Glass, which is also known as Float Glass contains both Sodium and Calcium, and is commonly used for microscopic slides and substrates. The material has a softening point in the range of 500 to 550°C and can sustain annealing upto the temperature of 500°C. Its hardness of 5.5 on Mohs scale make it a fair competitor for use as thin film substrate.

3.1.2.2 Bakelite

Bakelite is a rigid thermosetting plastic, highly heat resistant material. It cannot be recycled or melted again once it has been cured during the compression molding process. Unlike other thermoplastic resins, when heat and pressure are applied to Bakelite resin, which is a solid powdered material, it hardens rather than becoming viscous liquid. If excessive heat is applied to Bakelite in presence of oxygen, it can burn away however, it does not support flame at its own. Moreover, if the heat source is removed, the material will not burn in atmosphere. Bakelite is the first commercially produced plastic and can be compress molded into a wide variety of shapes with high quality surface finish. The material offers high electrical, thermal and chemical resistant properties so it can be used in electric insulating applications. If no reinforcing material is added to it, Bakelite exhibits brittle nature upon impact if used in very thin layer. However, a thick layer of Bakelite can withstand impact of sufficiently high intensity. Moreover, its impact strength can be improved by laminating it with materials like fiberglass. The material has been chosen as substrate because of three main reasons. First is the above mentioned properties which make it a suitable candidate for resistor

applications. Secondly, utilization of this material was an innovation in thin film resistor technology as no previous work with Bakelite as a substrate has been reported yet. Thirdly, the material was already being used as an insulating material between electrodes of bridge-wire detonator.

3.1.3 Miscellaneous

Besides the materials for thin film and substrates, certain additional materials are also required for preparation of substrates and other purposes. Those miscellaneous materials used during the synthesis of subject thin films were:

- Ethanol
- Propanol
- Acetone
- Distilled Water
- Grinding Paper (400, 800, 1200)
- Stainless Steel Electrodes
- Lint free cloth

3.2 Thin Film Deposition Techniques

Thin Films can be grown over a chosen substrate in several ways. In order to attain the required properties and usefulness of thin film, selection of proper technique for deposition of thin film is paramount. These techniques can be broadly categorized into chemical and physical methods [32]. The main difference between the two deposition methods lie in the methodology by which film material is deposited on the substrate. Since Physical Deposition Techniques are more commonly used for thin film deposition of metals therefore only these techniques are discussed here.

In Physical Deposition method, atoms or molecules are physically deposited in the form of a film on the substrate. The target material that is deposited on the substrate relies on various parameters such as temperature, vacuum, pressure, and similar other physical conditions [33]. The particles are directed straight from the target to the substrate therefore, the films formed by physical method are directional [34]. Primarily, the physical vapor deposition can be carried out either by evaporation or through sputtering

[35]. Both these techniques have several further categories which ae explained in subsequent paragraphs.

3.2.1 Evaporation Techniques

In these techniques, the target material undergoes transformation from solid or liquid to vapor phase for deposition on the substrate and then condenses to produce a solid thin film. Evaporation techniques are most common methods of depositing thin film and occur in vacuum conditions or other controlled environment conditions. The evaporation of the target material can be carried out in a number of ways depending upon the material's characteristics as well as the required degree of purity of thin film and the pace of deposition [36]. Some of the most widely used evaporation techniques used for thin film deposition are explained below:

3.2.1.1 Thermal Evaporation

In thermal evaporation technique, the solid target material is kept in a crucible from where it is heated with the help of a filament or heat source [37]. The solid target material is transformed into vapor phase which then travels through high vacuum chamber and strikes the substrate that is held opposite to the crucible. The evaporated material, after deposition on the substrate, forms a layer or film and solidifies thus giving desired thin film [38]. The generic arrangement of a thermal evaporation apparatus is shown below: -

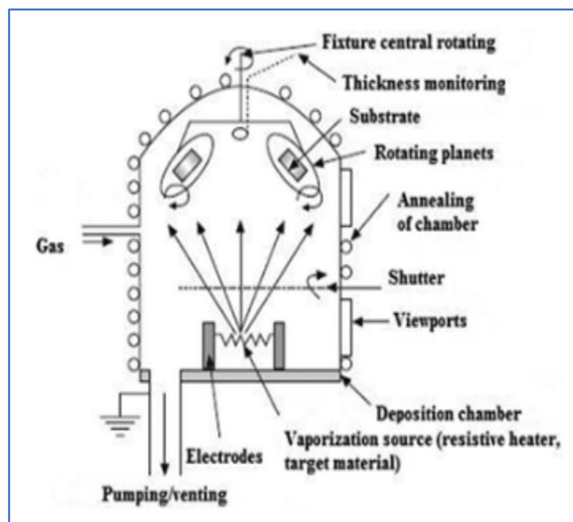

Figure 5: Schematic Representation of Thermal Evaporation Process [39]

3.2.1.2 Electron Beam Evaporation

In this technique, an electron beam is generated with the help of an electron gun or by heating a filament by passing current through it. The electrons are then directed with the help of electric and magnetic field towards the target material that is kept in a crucible. Upon reaching the crucible, electrons transfer their energy to the target material causing its atoms to evaporate. These evaporated atoms then move towards the substrate and condense on it forming thin film. This method of thin film fabrication has a number of advantages over other thermal evaporation techniques [40]. Evaporation of target material using electron beam is more efficient as it can attain high temperatures than through a crucible heater. Using electron beam evaporation technique, high deposition rate can be achieved and the materials with high melting points can also be readily deposited Electron beam evaporation has many advantages over resistive thermal evaporation.

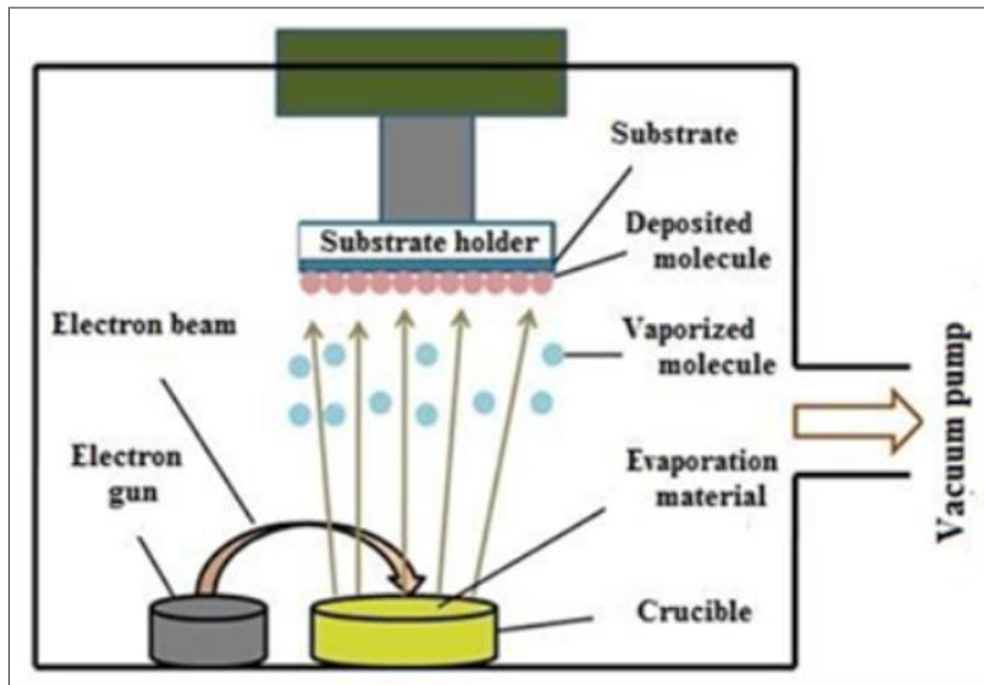

Figure 6: Schematic Representation of Electron Beam Evaporation Process [39]

3.2.1.3 Laser Beam Evaporation

This technique is also known as Pulsed Laser Deposition (PLD) in which a very high-power laser beam is used to vaporize the target material for deposition onto substrate

under high vacuum. The high-power laser beam melts, vaporizes and ionizes the target particles and forms a plasma plume. The laser produced plasma plume is basically vaporized material containing ions and electrons. The plasma plume rapidly expands away from the target and gets deposited onto the substrate and solidifies in the form of thin film [41].

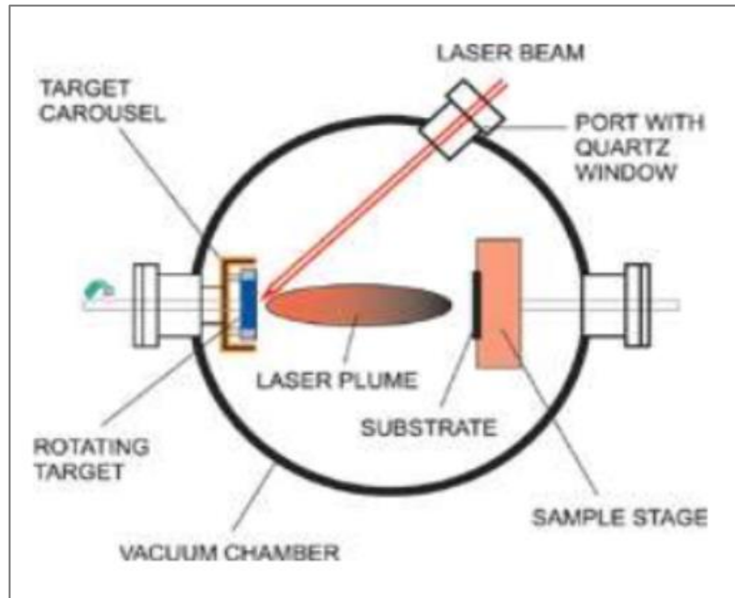

Figure 7: Schematic Representation of Laser Beam Evaporation Process [42]

3.2.1.4 Arc Evaporation

The Arc Evaporation technique, also known as Cathodic-Arc Deposition is a type of PVD technique in which the vaporization of target material for subsequent deposition on substrate is done with the help of an electric arc. The electric arc acts upon the cathode target and vaporizes it. Once the vaporized material is deposited onto the substrate, it condenses back to solid thus giving a thin solid film [43]. This technique can be used for depositing thin films of metals and composites.

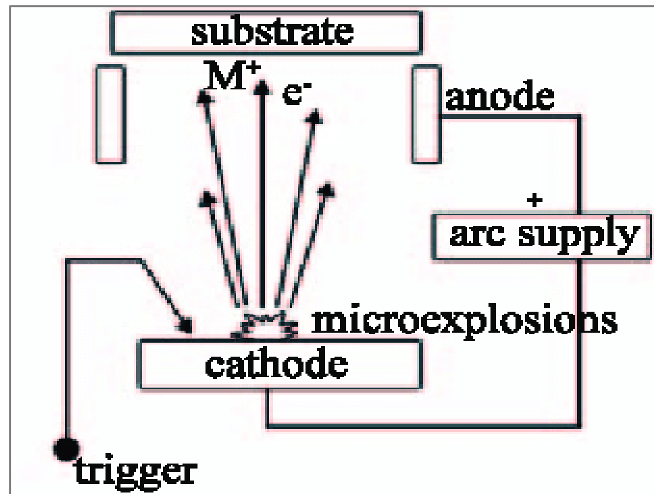

Figure 8: Schematic Representation of Arc Evaporation Process [44]

3.2.1.5 Molecular Beam Epitaxy

Developed in early 1970's by Alfred Cho and John Arthur, Molecular Beam Epitaxy is a technique used for deposition of metals, semiconductors and insulators. The basic working principle of this technique involves evaporation of the target material under ultra-high vacuum followed by deposition and solidification of the molecular or the atomic beam on the heated substrate hung infront of the target material. The target material with high purity level is kept in effusion cell where it is heated to sublimation and a molecular beam is generated that flows towards the substrate. While their journey to substrate, the molecules can react chemically with each other or with the gases that are introduced in the chamber and then condense onto the substrate in the form of thin film. In order to achieve thin film with extremely high level of purity, it is important to maintain ultra-high vacuum and use target material with high degree of purity. The crystal molecules or the atoms settle layer by layer on the substrate [45]. The crystal growth can be observed in situ by RHEED (Reflection High-Energy Electron Diffraction) and the molecular beam directed from the effusion cell can be stopped any moment thus enabling extreme control of the layer thickness and deposition process [46]. Molecular Beam Epitaxy is generally used for specific applications such as the cases where controlled temperature or sudden variation and control of layers' profile is required or where high-quality epitaxial structures are needed. The technique is not very suitable for mass production due to slow yield rate.

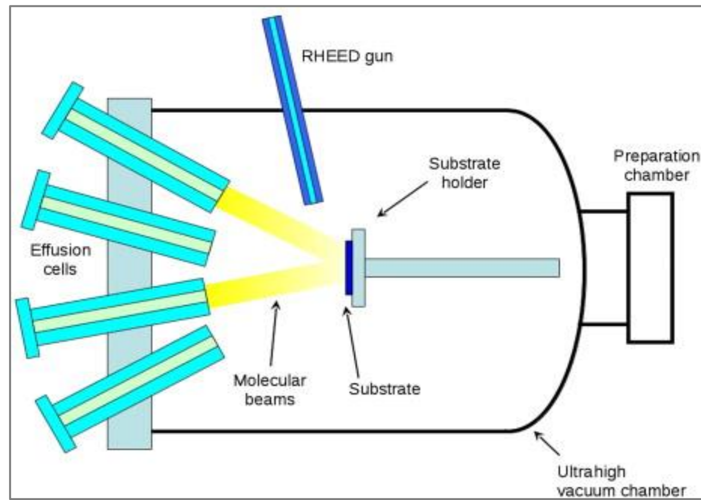

Figure 9: Schematic Representation of Molecular Beam Epitaxy Process [47]

3.2.2 Sputtering Technique

Sputtering is a type of physical vapor deposition in which the deposition takes place by the momentum transfer of energetic ions onto the target material (cathode). The bombardment is generally carried out with argon ions. The bombardment causes the atoms of target material to eject from the surface and travel towards the substrate under vacuum. The ejected atoms settle on the substrate (anode) forming a thin layer or film [48]. Sputtering can be done by a variety of methods which are explained in subsequent paragraphs.

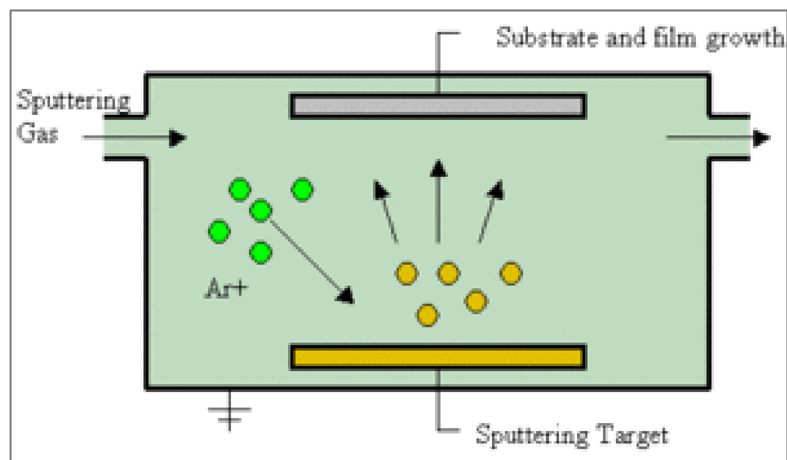

Figure 10: Schematic Representation of Sputtering Process [48]

3.2.2.1 Direct Current Sputtering

In Direct Current (DC) sputtering method, a DC voltage of several kilovolts is applied between the anode and the cathode. The negative terminal of power supply is connected with cathode and positive terminal with anode. The cathode is water cooled from one side and attached to the target material from the other side. The anode, which can be cooled, heated, grounded or in any other configuration is attached with the substrate. A non-reacting inert gas such as argon gas is introduced into the chamber at a very low pressure. The DC voltage ionizes the argon atoms to argon ions by the collision of electrons with neutral argon atoms, causing a positive charge on them. These ions rush towards the cathode and strike it at a very high speed. The high-speed bombardment extracts the particles out of the cathode which travel in different directions including the anode. The ejected particles settle on the substrate forming a thin film [49]. DC Sputtering method cannot be used if the target material is non-conducting because this would affect the conductive behavior of the anode once the particles get sputtered onto the anode. Moreover, the density of argon atoms is very low which causes very fewer particles of target to be extracted out, hence the deposition rate using DC sputtering technique is relatively low.

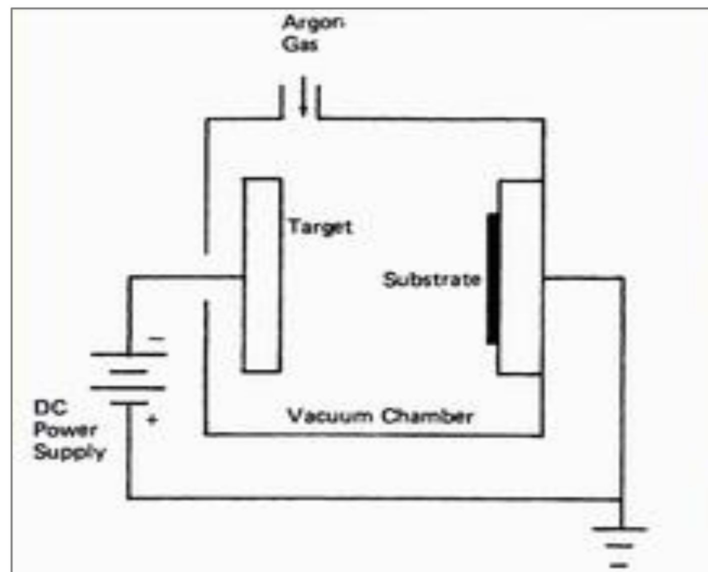

Figure 11: Schematic Representation of Direct Current Sputtering System

3.2.2.2 Radio Frequency Sputtering

The target material used in DC sputtering is generally of electrically conducting material. However, if the target material is of insulating nature, glow discharge cannot be sustained due to accumulation of positive ions close to the surface of insulating target, making the target positively charged. For sustainability of glow discharge while using an insulating material for target, high voltage RF source with frequency 13.56 MHz is used in place of DC voltage supply [50]. This technique is named as Radio Frequency (RF) sputtering. A block diagram of RF Sputtering technique is given below:

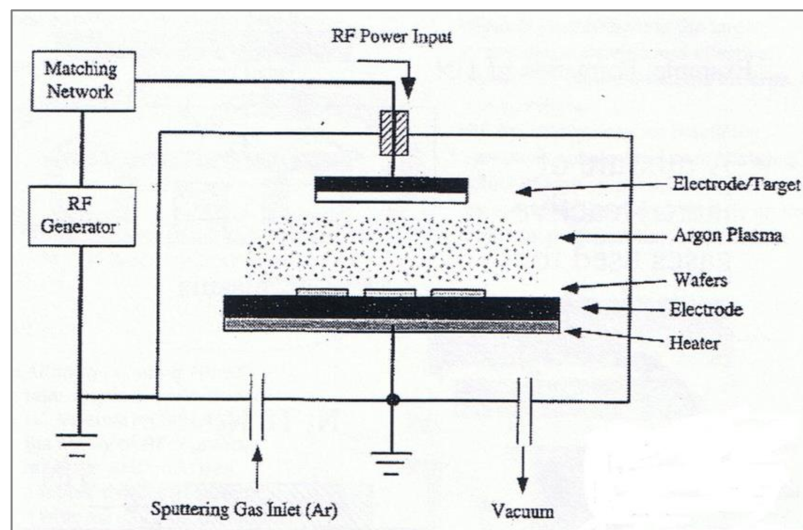

Figure 12: Schematic Representation of Radio Frequency Sputtering System

As shown in the above diagram, the RF sputtering system has an impedance matching network connected between the power source and the chamber. Moreover, there is a blocking capacitor connected in series between the impedance matching network and the cathode target. The cathode-anode configuration is asymmetric as the size or the area of the cathode is quite small as compared to that of the anode or the chamber wall. This leads to the induction of negative bias on the cathode target which causes sputtering in this technique. RF Sputtering changes the electrical charge on an electrode at Radio Frequency in order to prevent a charge buildup on the target material.

3.2.2.3 Magnetron Sputtering

In order to enhance the number of argon ions generated in DC/RF sputtering technique for bombardment on the cathode target, a magnetic field is generated close to the cathode by using permanent magnet(s) behind the cathode. In case of DC/RF sputtering, the electric field is perpendicular to the target surface however, with the induction of magnetic field parallel to the target surface, the electrons move in a spiral path close to the cathode. This causes an increase in the probability of collision between the electrons and the argon gas and hence produces more ions. These ions then accelerate towards the cathode and extract more atoms or molecules from target thereby increasing deposition rate [51].

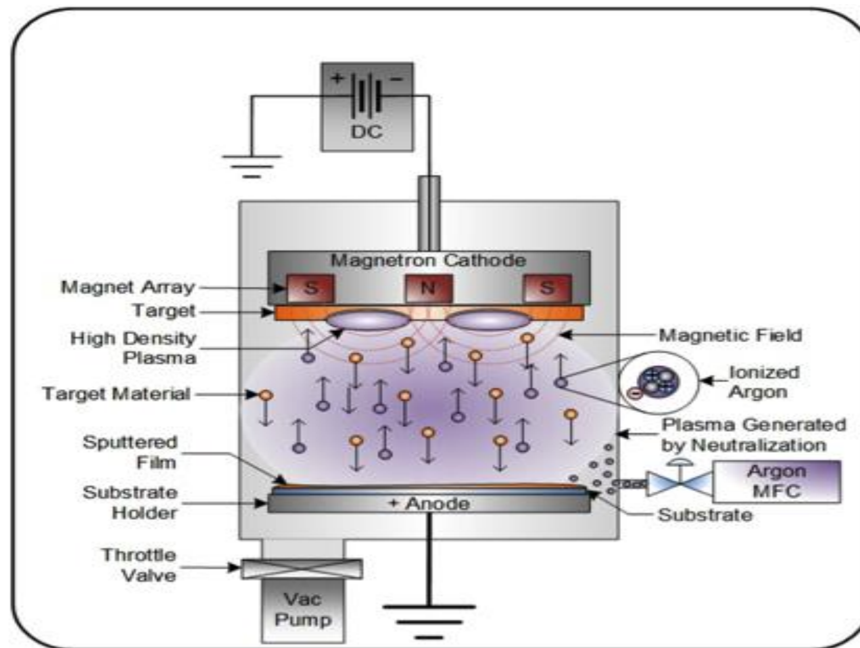

Figure 13: Schematic Representation of DC Magnetron Sputtering Process

3.3 Characterization Techniques

The term characterization refers to detailed examination of a particular object. Since thin films are in nano-scale, their properties cannot be studied with naked eye. Therefore, microscopic analysis of such objects is carried out. In order to observe various parameters of thin films, multiple characterization techniques are being used globally. Some of those techniques that would be used in this research work are explained in this chapter.

3.3.1 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy is a technique for examining the structure and morphology of thin films, coating and membranes. It is a non-destructive and easy-to-use technique for analyzing samples upto nano-scale.

3.3.1.1 Components of SEM

Primary components of Scanning Electron Microscope (SEM) include:

i. Electron source
ii. Column and electromagnetic lenses
iii. Electron detector
iv. Sample chamber
v. Display screens

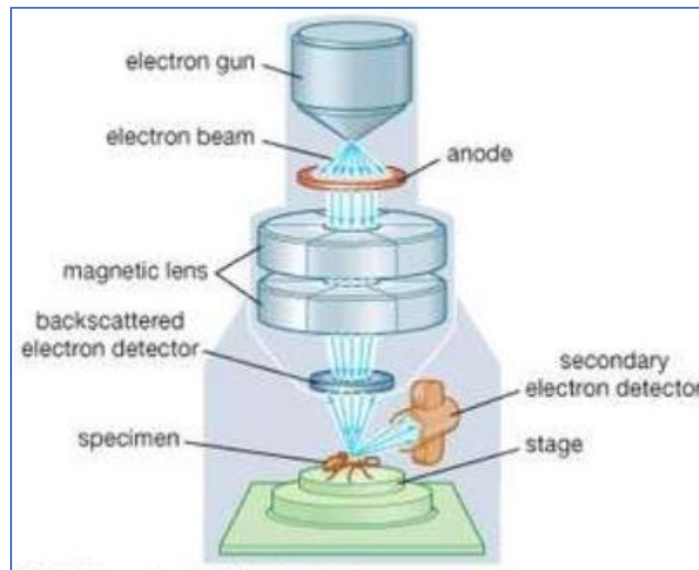

Figure 14: Schematic Diagram of Scanning Electron Microscope

3.3.1.2 Working Principle

The working principle of SEM is that an electron beam is concentrated over the surface of a sample under observation. High degree of vacuum is established inside the column of Scanning Electron Microscope through different pumps. The absence of atoms of any gas inside chamber due to vacuum lets the electron beam impact directly onto the surface of the sample. This interaction between the electron beam and the surface leads

to backscattering of incident beam as well as knocks off some particles from the surface. As a result, backscattered electrons, secondary electrons and X-rays are formed. Commonly, the detection mode of SEM relies on the secondary electrons. The secondary electrons are emanated from close to the surface of sample. This gives a distinct and a clear image of the sample under observation. Details, as small as 1nm, are also exposed through this image. The secondary electrons clarify the morphology and topography of the material. The backscattered emerge from deeper locations as compared to secondary electrons and give information about the phase. Characteristic X-rays also emit from the atoms when an inner shell electron knocks off from its shell.

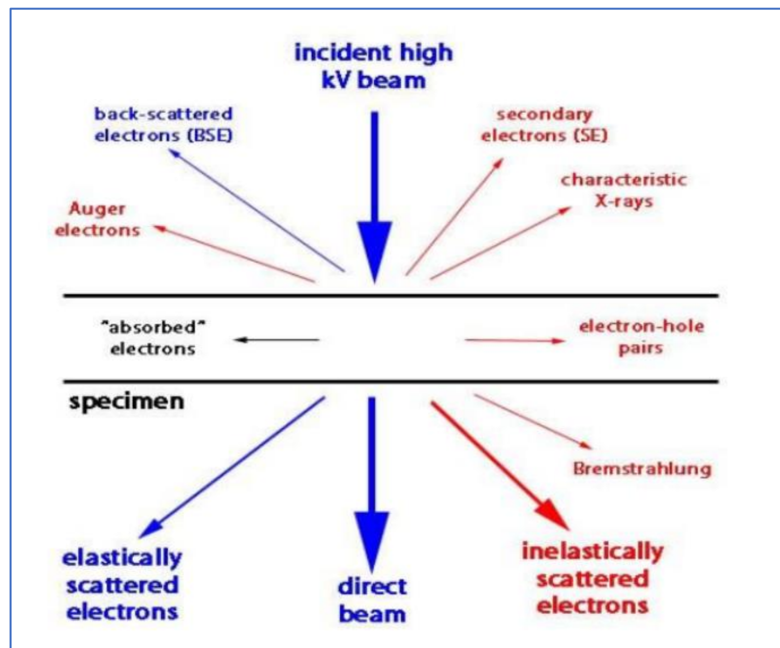

Figure 15: Scattering of Electrons on Interaction with Matter

3.3.1.3 Image Formation

Two dimensional intensity map is created by SEM and each pixel represents a point directly related to the strength of the signal. The image of SEM is displayed electronically. SEM images give two kinds of information:

i. Material's morphology.
ii. Material's topography.

3.3.2 Atomic Force Microscopy

Atomic Force Microscopy (AFM) is a versatile and powerful technique for studying compounds in nano range. The technique has the ability to provide 3D image of material's topography as well as gives information about the height and thickness of nano materials. Images with atomic level resolution can be generated with AFM.

3.3.2.1 Working Principle

In Atomic Force Microscopy, a tip attached to cantilever is used to scan a sample surface. When tip comes near to sample surface, it makes contact with sample and repulsive forces arise which deflect the cantilever from the surface. The force between the sample and probe is measured as an operation of AFM. The probe is a sharp and tall pyramid tip. The vertical resolution is mostly higher as compared to lateral resolution. It uses a piezoelectric scanner to scan the surface of sample. A feedback control system maintains a constant distance between tip and surface of sample. Optical lever monitors deflections from laser source on cantilever's back which is read by a sensitive photodiode.

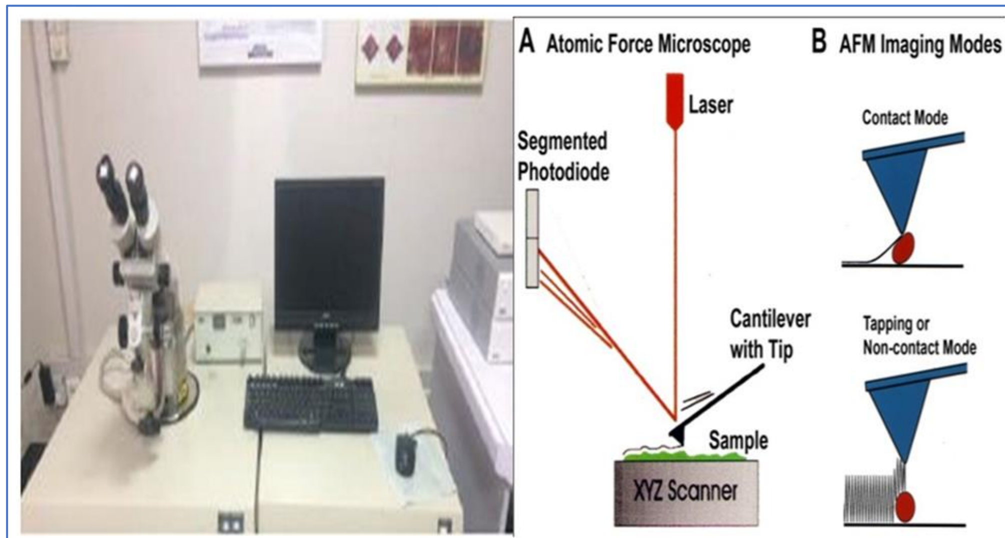

Figure 16: Schematic Diagram of Atomic Force Microscope

3.3.3 X-Ray Diffraction

X-Ray Diffraction (XRD) is a technique to determine the phase purity and crystallinity of material. X-rays have provided researchers the means to determine properties of

materials at atomic level. It can determine how the atoms are packed, the bond length and angles.

3.3.3.1 Components of XRD

X-Ray Diffractometer consists of four main components:

i. X-ray source
ii. Monochromator
iii. Goniometer
iv. Detector

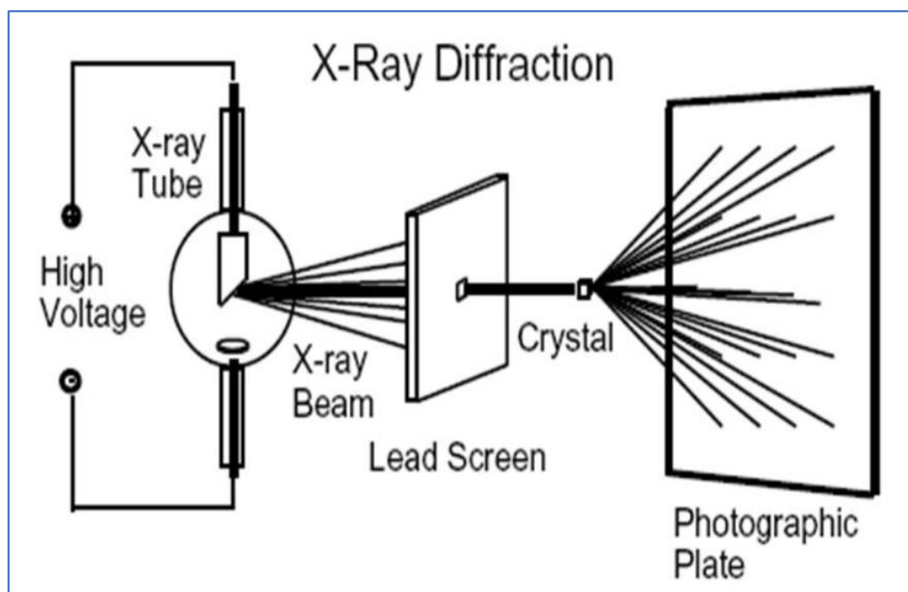

Figure 17: Principal of XRD

3.3.3.2 Working Principle

The working principle of XRD is dependent upon the interference of monochromatic rays. The X-rays generated are directed on to the sample under observation. Each material has its atoms arranged in regular planes in a crystal lattice. The X-rays after striking the material are both refracted and transmitted while some of it is also diffracted and absorbed in the sample material. The diffraction of light is different for different materials and phases depending upon the atoms arrangement in crystal lattice. The distance between the atoms is measured by Bragg's Law which is defined as

$$n\lambda = 2d\sin\theta$$

n = order of diffracted beam

λ = wavelength of incident x-ray beam

d = distance between adjacent planes of atoms

Distance between the adjacent planes of atoms generated in the x-ray scan gives the fingerprint of the sample. Material can easily be identified by comparing this scan with the reference pattern.

3.3.3.3 Applications

X-ray diffraction analysis is widely used for following applications:

i. Identification of unknown crystalline materials
ii. Determination of unit cell dimensions
iii. Checking purity of sample

3.3.4 M201 Milli-Ohmmeter

The M201 Milli-ohmmeter by Rhopoint is a small sized, portable, easy-to-use, digital meter designed to measure resistances as small as 0.001Ω upto 200Ω. The device is powered by a single 9V battery and offers high degree of purity though the use of four terminal input to remove errors induced by the length of testing leads. A very significant features of this device is low (5mA) test current. The small current guarantees that the contact under testing is not 'cleaned' by the test current which can happen if higher test currents are used. The 'cleaning' of contact under test results in inaccurate measurements.

Figure 18: Rhopoint Milli-Ohmmeter

Chapter 4
Experimental Work

Having gathered sufficient knowledge about the quantum of work globally done regarding utilization of thin film technology in fabrication of resistive films, their subsequent utilization in development of thin film based electric detonators, and the methods / techniques of manufacturing thin films along with their characterization techniques, it is now time to utilize the attained knowledge and available resources in development of thin film resistor. The experimental work carried out in development of thin film resistor is explained in detail in this chapter.

4.1 Material Preparation

After the selection of materials, the second phase was the preparation of selected materials for thin film deposition. The preparation of both types of substrates and thin film material were prepared as explained in subsequent paragraphs.

4.1.1 Bakelite Substrate Preparation

Since the basic aim of the project was to replace the bridge wire between the electrodes of an electric detonator with a thin film bridge, therefore the substrate was required to be molded in such a shape that it fits in the small cavity of the detonator. Moreover, the substrate should serve the purpose of providing insulation between the two electrodes and lastly but most importantly, the substrate should provide a flat surface for deposition of thin film. The flat surface must include the ends of both electrodes so that when the thin film is deposited over the flat surface of the substrate, the thin film forms a bridge between the electrodes thereby completing the electrical path for current to flow through it.

Bakelite in the shape of an initiator as shown in figure-24 was provided by Air Weapons Complex. Bakelite initiator was then subjected to multiple treatments for obtaining smooth and crack-free surface for tin film deposition. The sample was first grinded and polished to obtain smooth surface and was then subjected to cleaning by sonication in

distilled water and ethanol for 2 hours. The Bakelite sample was then kept in oven for 60 min at 90 ^oC to remove moisture.

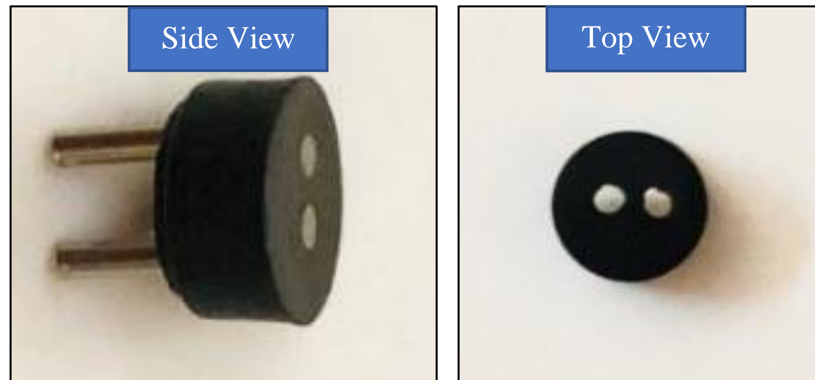

Figure 19: Bakelite Initiator

4.1.2 Glass Substrate Preparation

The glass substrate could not be prepared in the shape of an initiator because of limitations in dye manufacturing. However, since the primary scope of the project was to determine compatible thin film and substrate material that gives low resistance along with providing rigidity and strength to sustain high g shock and rotational loads in special environments therefore, any combination of such materials in any shape could be utilized to validate the required parameters. Glass is known to be one of the best materials that can be used as an insulating substrate and is strong enough to sustain in high speed special environment. Small slides of Glass having dimensions of 25.4 mm x 25.4 x 1.2 mm were prepared for thin film deposition. The Glass slides were soap cleaned followed by sonication in ethanol for 30 min and were then cleaned with acetone. The slides were then cleaned with lint free cloth and were placed in oven at 100^oC for one hour for drying and removal of moisture from its surface. The substrate was then ready for thin film deposition.

4.2 Thin Film Deposition

The deposition of thin films on the substrates was carried out using three materials individually; Gold, Nichrome and Silver. The deposition details of each material are given in proceeding paragraphs.

4.2.1 Gold Thin Film Deposition

Thin Film of Gold was deposited on glass and Bakelite substrates by Physical Vapor Deposition using 'Resistive Heating Unit'. The evaporating charge (Gold) was kept in a tungsten boat while the substrates were fitted onto the substrate holder with double-sided adhesive. The distance between boat and substrate holder was 30mm. The vacuum was of the order of 3 x 10^{-5} Torr which was created using two vacuum pumps. The rotary pump established coarse vacuum up to the order of 10^{-2} torr after which the diffusion pump was switched on which further enhanced the vacuum to 10^{-5} Torr level. Since there was no system for in-situ measurement of film thickness, therefore deposition time was monitored during the process. The evaporation process was carried out for 120 seconds after which the system was turned off. The vacuum created was gradually broken and substrates were removed and kept in moisture free environment until subsequent analysis.

4.2.2 Nichrome Thin Film Deposition

The deposition of Nichrome (Ni 80%, Cr 20%) on glass and Bakelite substrates was also carried out by PVD method using Electron Beam evaporation technique. The operation was conducted using 'E-beam Evaporation System' obtained from 'Torr International, Inc'. The evaporating material (Nichrome) was prepared by cutting a Nichrome sheet with diamond cutter into small pieces to make evaporation charge. Nichrome charge was placed in a boat of graphite. Glass and Bakelite substrates were fitted onto the water cooled substrate holder, attached straight above the crucible boat, with double-sided adhesive at a distance of 55 mm from boat. Vacuum of 2.9 x 10^{-5} Torr was created in a sequential manner. First, a rotary pump was used for creating coarse vacuum and then Turbo Molecular Pump was used to induce high vacuum inside the chamber. Input parameters were continuously updated during the process after achievement of 2.2 x 10^{-5} Torr vacuum level. The deposition was carried out for 15 min and the substrates were then removed and kept in moisture free environment until further analysis. The input and observed parameters during whole process are appended in table below:

Table 3: Applied Conditions for Nichrome Evaporation

Power	Vacuum (Torr)	Voltage (KV)	Current (Amp)	Filament Current (Amp)
0.5 %	2.2×10^{-5}	5.18	5	15.8
0.7 %	2.4×10^{-5}	5.18	8	16.5
1.1 %	2.7×10^{-5}	5.18	13	16.9
1.5 %	2.8×10^{-5}	5.18	16	17.3
2 %	2.8×10^{-5}	5.18	21	17.7
2.5 %	2.9×10^{-5}	5.18	25	17.9

4.2.3 Silver Thin Film Deposition

Thin films of Silver were deposited on glass and Bakelite substrates by PVD method using 'Resistive Heating Unit'. Same unit was used for deposition of Gold thin films. Tiny pebbles of silver were kept in Tungsten boat while substrates were attached onto the substrate holder using double-sided adhesive. Vacuum of the order of 3×10^{-5} Torr was established using rotary pump and diffusion pump. Voltage and current provided for evaporation were 38 Amp and 60 V respectively. The deposition was carried out 5 min after which the system was turned off and substrates were removed. The input and observed parameters during thermal evaporation process are tabulated below:

4.3 X-Ray Diffraction

The XRD analysis of each sample was carried out to determine the crystalline phase of the materials and their purity. X-ray diffractions were performed by STOE diffractometer at SCME-NUST. The scan angle was 10° to 85°.

4.4 Scanning Electron Microscopy

SEM of all samples was also carried out to see the surface morphology of thin films. SEM analysis was performed using 'Scanning Electron Microscope (JEOL-JSM-6490LA)' with operating voltage of 10-20 kV, spot size of 35-60, and working distance of 10mm. The images were recorded in SE mode at low and high magnifications.

4.5 Atomic Force Microscopy

Atomic Force Microscopy of all films deposited on Glass substrate was carried out to re-verify the morphological properties obtained through SEM and to monitor the surface roughness of the films. The procedure was carried out using JEOL SPM 5200 held at SCME.

4.6 Resistance Determination Procedure

The sheet resistance of all films prepared on glass as well as on Bakelite initiators was determined through 'Two Probe Method' using 'Rhopoint Milli-ohmmeter'.

4.6.1 Bakelite Initiators

The electrical resistances of Bakelite initiators that were coated with Gold, Silver and Nichrome were checked with the help of "Rhopoint milli-ohmmeter". The electrical leads of ohm-meter were connected with an attachment (Fig 5-4) into which the initiators can be fitted to check the resistance between the electrodes. The initiators, after being coated with thin films, were held one by one with the help of a tweezer from sides and were connected with milli-ohmmeter one at a time.

4.6.2 Glass Substrates

The sheet resistance of Gold, Silver and Nichrome deposited separately on glass substrates were also determined using 'Two Probe Method'. The probes of milli-ohmmeter were held at two corners of the film (substrate) and the observed values were noted down. In order to draw a comparison of resistance values of each film, their thicknesses were monitored through cross sectional image taken by SEM and was reconfirmed by Optical Profilometry.

Chapter 5

Results and Discussion

Bridge-wire resistor of an electric detonator is intended to be replaced with thin film bridge resistor in order to increase the reliability of the detonator. The fabrication and characterization of thin film bridge resistor is carried out using Physical Vapor Deposition method. Multiple substrates and depositing materials were considered to achieve thin film that offers relatively high resistance, low temperature coefficient of resistance, high strength and easy of manufacturing resistor.

5.1 Substrate Materials

Two different materials were finally selected for deposition of thin films; Bakelite and Soda Lime Glass. Both the materials were chosen on the basis of their melting points, hardness, ease of availability and past utilization. Bakelite substrate in the shape of initiator was provided by AWC while glass substrate was prepared by cutting microscopic glass slides into square shape with a dimension of 1 in x 1 inch.

5.2 Target Materials

Out of different materials considered for fabrication of thin film resistor, three materials (02 metals and 01 metal alloy) were finally shortlisted on the basis of their resistivity, hardness and low TCR. The materials were Gold, NiCr and Silver. NiCr is a transition metal alloy and the composition of the alloy was 80% Ni and 20% Cr. NiCr is most widely used alloy for resistive applications. Each metal was separately deposited on both types of substrates though Physical Vapor Deposition Techniques. The films developed from each material were characterized using different techniques to determine the surface morphology, surface roughness, film structure and resistance offered by the films.

5.3 Scanning Electron Microscopy

Scanning Electron Microscopy of all thin films deposited on chosen substrates was carried out to observe the morphology of the film. The results obtained from different films are given below:

5.3.1 SEM of Gold Thin Films

The Scanning Electron Microscopy of Gold thin films deposited on Bakelite substrate revealed that the film was uneven, non-uniform and contained multiple cracks. The deformations in the film were due to uneven and rough surface of Bakelite.

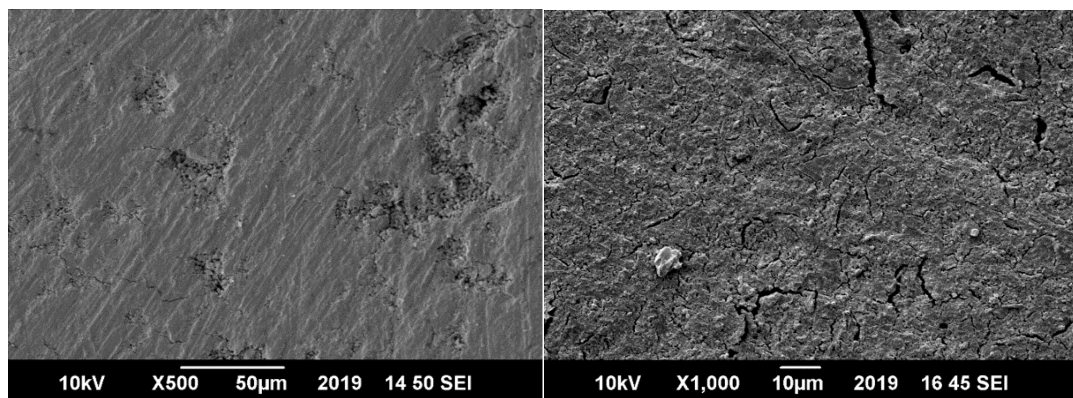

Figure 20: SEM Images of Gold Thin Film on Bakelite Substrate

However, thin film of Gold deposited on Glass substrate exhibited smooth and pinhole free images as revealed by Scanning Electron Microscope. The film was zoomed up-to 35,000 times and no crack or deformation was found in the film.

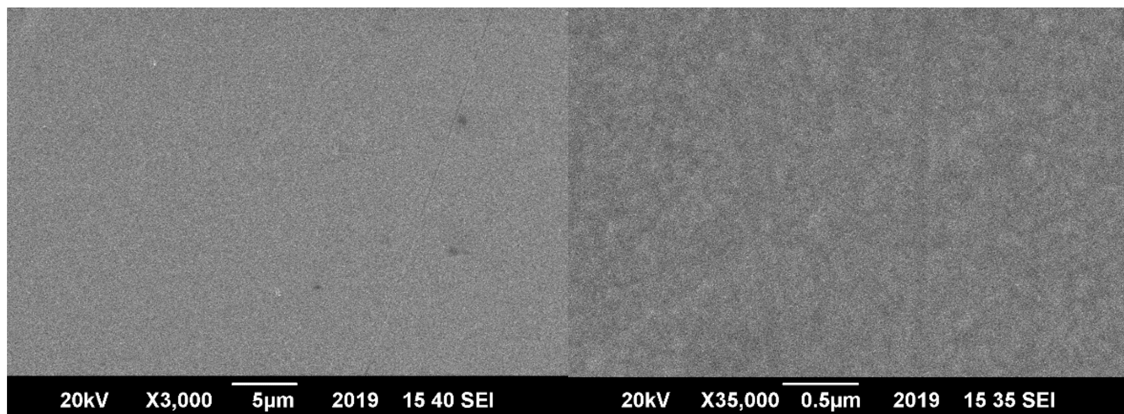

Figure 21: SEM Images of Gold Thin Film on Glass Substrate

5.3.2 SEM of Silver Thin Films

Similar to Gold thin film, the Scanning Electron Microscopy of Silver thin films deposited on Bakelite substrate also revealed uneven, non-uniform and rough surface with multiple cracks. The deformations in the film were due to high surface roughness of Bakelite.

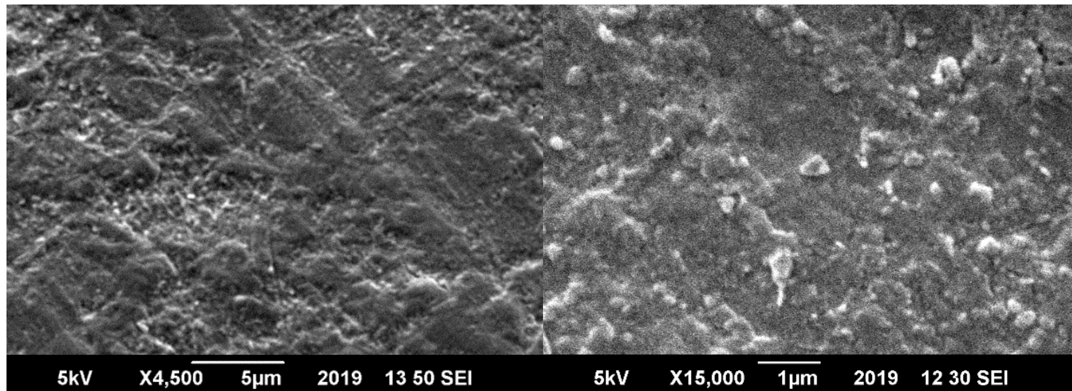

Figure 22: SEM Images of Silver Thin Film on Bakelite Substrate

On the other hand, smooth and pinhole free SEM images of silver thin films deposited on glass substrate were obtained. The film was zoomed up-to 25,000 times and no crack or deformation was found in the film.

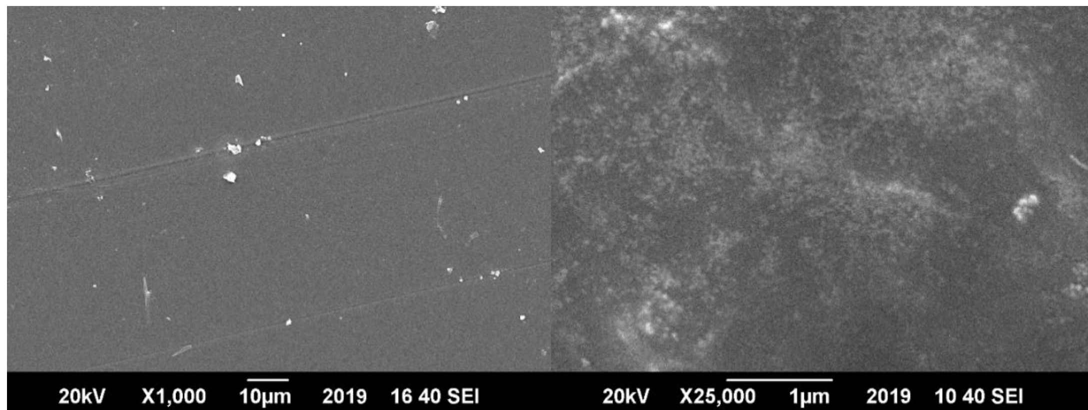

Figure 23: SEM Images of Silver Thin Film on Glass Substrate

5.3.3 SEM of NiCr Thin Films

Similar to Gold and Silver thin films, the Scanning Electron Microscopy of NiCr thin films deposited on Bakelite substrate also revealed uneven, non-uniform and rough

surface with multiple cracks. The deformations in the film were due to high surface roughness of Bakelite which led to the conclusion that Bakelite is not a suitable material for thin film deposition due to its high surface roughness.

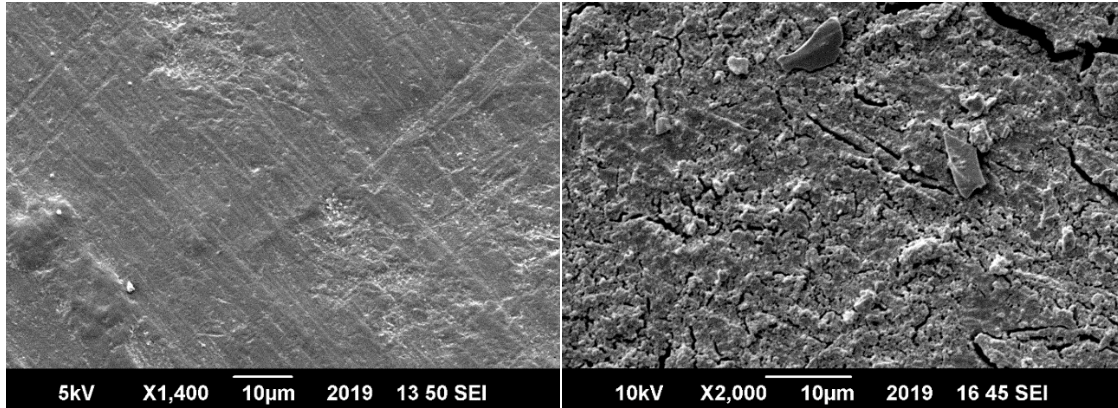

Figure 24: SEM Images of NiCr Thin Film on Bakelite Substrate

Whereas the SEM images of NiCr thin film on glass substrate revealed smooth pinhole free images of the surface. The film was zoomed up-to 50,000 times and no crack or deformation was found in the film which led to the conclusion that glass is a better substrate than Bakelite for thin film deposition due to more smoothness of glass.

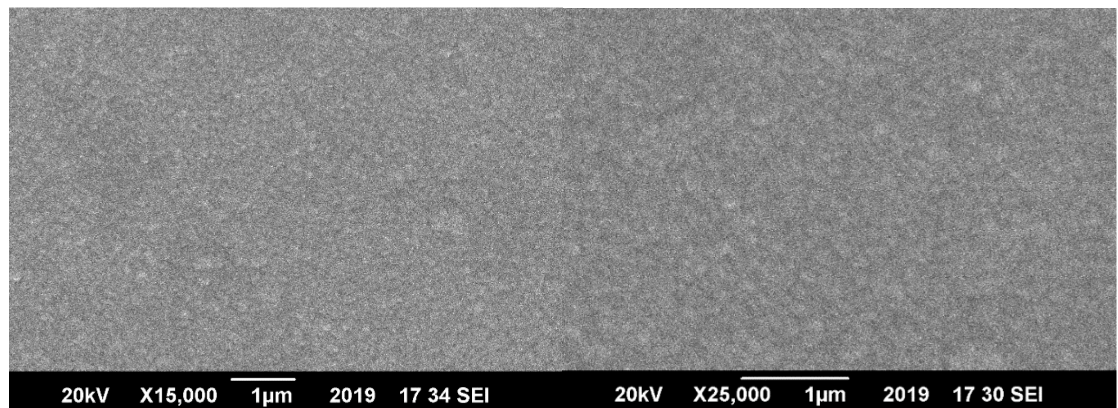

Figure 25: SEM Images of NiCr Thin Film on Glass Substrate

5.4 Atomic Force Microscopy

Thin Films of Gold, Silver and NiCr deposited on glass substrate for fabrication of resistor were analyzed through Atomic Force Microscope for observing the roughness values of the films. Since the SEM images of all films on Bakelite substrate did not

exhibit promising results therefore, Bakelite was not used in further characterizations. AFM results of each individual film deposited on glass substrate are explained below:

5.4.1 AFM of Gold Thin Film

AFM images of Gold thin films on Glass substrate were in conformance with the SEM images. The film obtained was smooth and the maximum roughness obtained in a scan area of 6µm x 6µm was 13 nm. The average roughness (Ra) of the scan area was 2.33 nm which confirms that the film obtained was smooth.

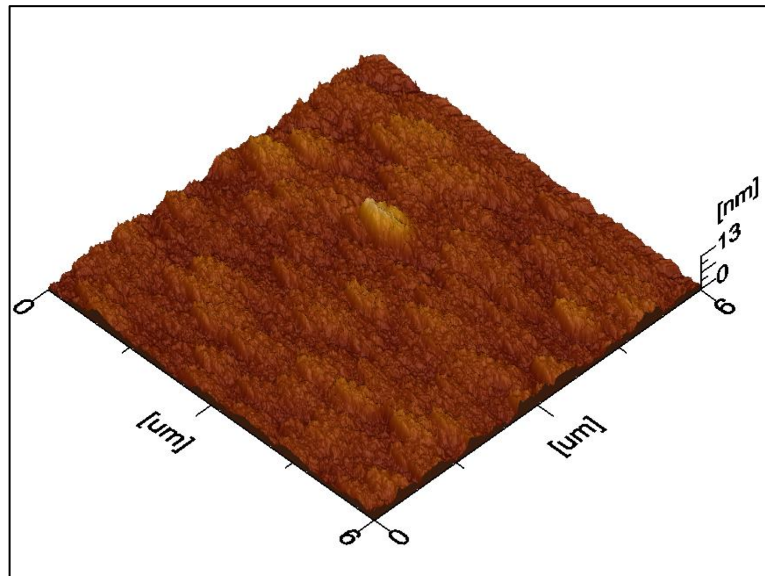

Figure 26: AFM Image of Gold Thin Film on Glass Substrate

5.4.2 AFM of Silver Thin Film

AFM images of Silver thin films on Glass substrate were also in conformance with the SEM images. The film obtained was smooth and a maximum roughness of 45 nm was obtained in a film with thickness of approx. 80 nm when an area of 2µm x 2µm was scanned. The average roughness (Ra) of the scan area was 15.2 nm which confirms that the film obtained was smooth.

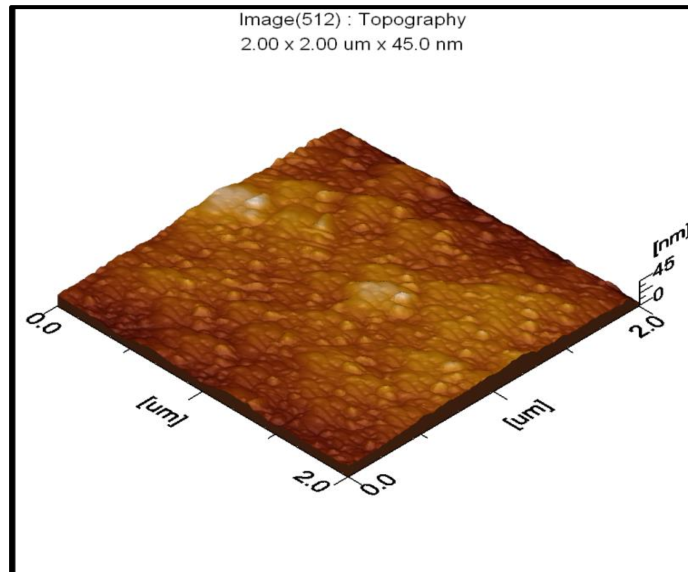

Figure 27: AFM Image of Silver Thin Film on Glass Substrate

5.4.3 AFM of NiCr Thin Film

AFM images of Nichrome thin films on Glass substrate were in conformance with the SEM images. The film obtained was smooth and the maximum roughness of 86nm was obtained in a film having thickness of 400 nm approx. when an area of 6µm x 6µm was scanned. The average roughness (Ra) of the scan area was 28.4 nm which confirms that the film obtained was smooth.

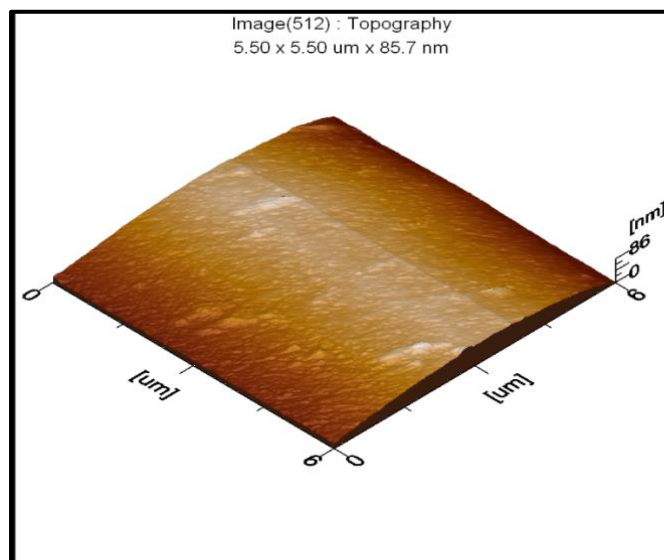

Figure 28: AFM Image of NiCr Thin Film on Glass Substrate

5.5 X-Ray Diffraction

The X-ray diffraction was carried out to determine the structure, crystallinity and the purity of the film. The XRD was carried out at scan angle range of 10 ° to 85 °.

5.5.1 XRD of Gold Thin Film

The X-ray diffraction of Gold thin film on glass substrate were matched with JCPDS Card No. 01-089-3697. Gold is characterized by four peaks, one each at 38.2°, 44.3°, 65.2 ° and 77.6°. The planes identified were (111), (200), (220) and (311) corresponding to FCC structure.

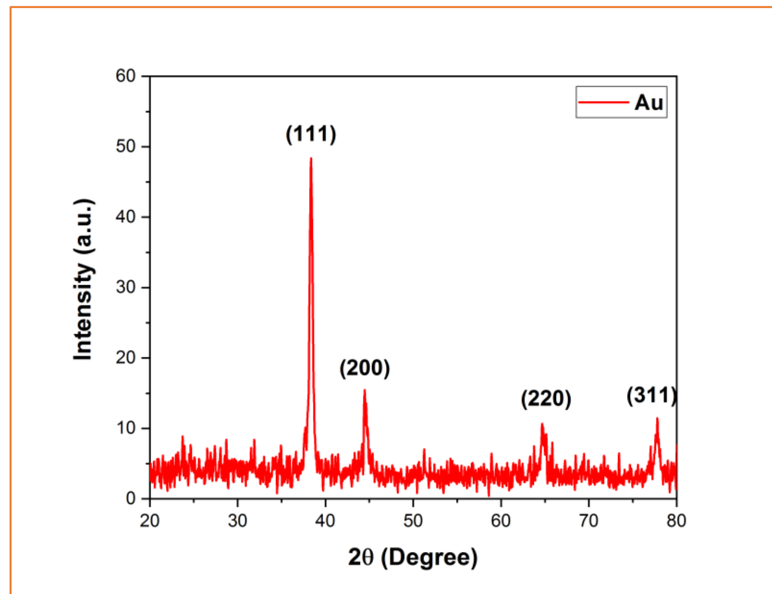

Figure 29: XRD Image of Gold Thin Film on Glass Substrate

5.5.2 XRD of Silver Thin Film

The X-ray diffraction of Gold thin film on glass substrate were matched with JCPDS Card No. 01-089-3697. Gold is characterized by four peaks, one each at 38.2°, 44.3°, 65.2 ° and 77.6°. The planes identified were (111), (200), (220) and (311) corresponding to FCC structure.

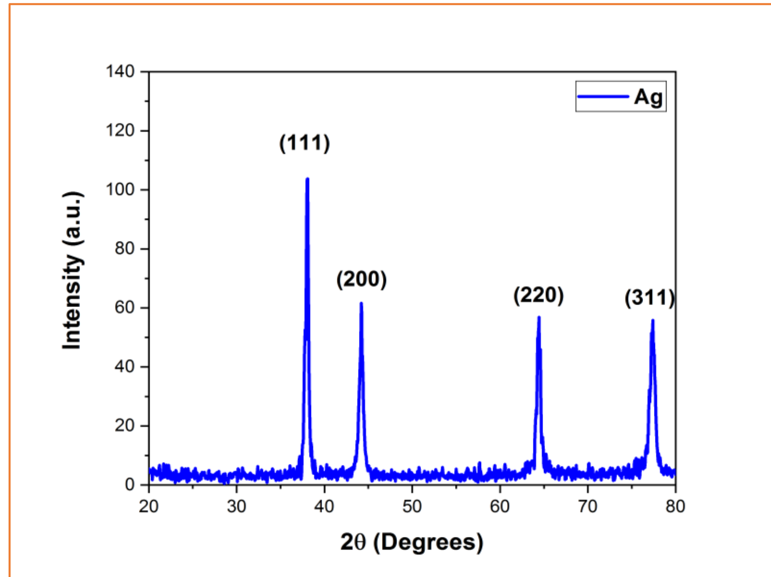

Figure 30: XRD Image of Silver Thin Film on Glass Substrate

5.5.2 XRD of NiCr Thin Film

The X-ray diffraction of NiCr thin film on glass substrate were matched with JCPDS Card No. 03-065-5559. NiCr (80/20) is characterized by three peaks, one each at 44.3°, 52.4° and 77.6°. The planes identified were (111), (200) and (220).

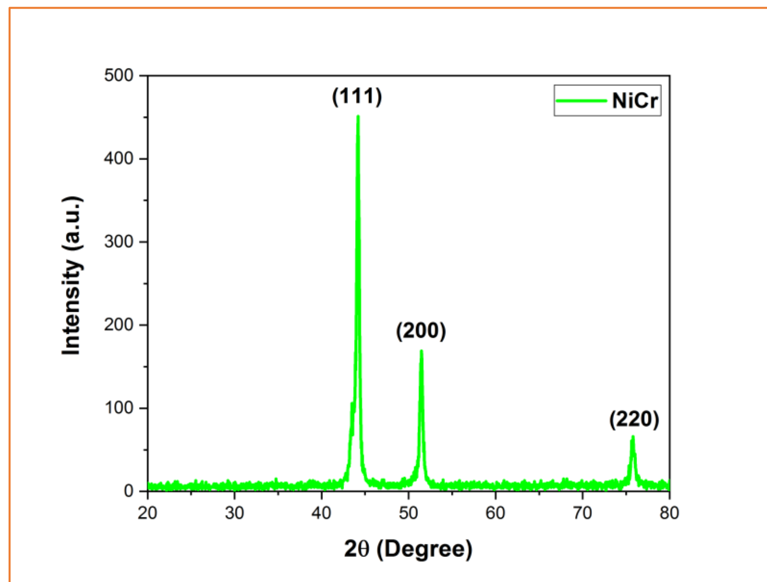

Figure 31: XRD Image of NiCr Thin Film on Glass Substrate

5.6 Sheet Resistance

After the analysis of films' morphological and structural properties, the most significant parameter of the resistor, that is the sheet resistance, was determined. The resistance values are segregated on the basis on substrate material as well as thin film material.

5.6.1 Resistance on Bakelite Initiator

Although thin films deposited on Bakelite initiator did not give promising results however, their resistance values were still measured to see if any trend can be observed in resistance variation. Given in the table below are the resistance values of Gold, Silver and Nichrome thin films deposited on Bakelite initiators.

Table 4: Resistance of Thin Films on Bakelite Substrate

Substrate	**Thin Film Material**	**Sheet Resistance Range (Ω/sq)**
Bakelite (initiator)	Gold	1.34-71.0
Bakelite (initiator)	Silver	0.03-5.09
Bakelite (initiator)	Nichrome	3.2-55.67

Film thickness on Bakelite substrate could not be measured however, it was observed that different Bakelite samples coated at the same time with same material displayed quite different results. This is due to the surface roughness and cracks in the films. Many a times, the films did not show any resistance due to discontinuous films. This shows that although Bakelite does give films with resistive properties, yet it does not follow any standard pattern of resistance variation. Due to this, repeatability of resistance cannot be ensured.

5.6.2 Resistance on Glass Substrate

The sheet resistance values of thin films deposited on glass substrate, measured by Two Probe Method' are given in table below:

Table 5: Resistance of Thin Films on Glass Substrate

Substrate	Thin Film	Film Thickness (nm)	Sheet Resistance (Ω/sq)
Glass	Gold	30	1.62
Glass	Silver	81	0.81
Glass	Nichrome	403	2.38

5.7 Analysis

Thin films of Gold, Nichrome and Silver were deposited on Glass and Bakelite substrates to fabricate thin film resistor. The analysis on results achieved from different materials is conducted here.

5.7.1 Thin Films on Bakelite

Thin films of Gold, Silver and Nichrome deposited on Bakelite substrate did not give good results due to high surface roughness of Bakelite due to which repeatability of sheet resistance cannot be ensured. However, the process can be re-verified by depositing film on Bakelite with enhanced surface smoothness and finishing.

5.7.2 Silver Film on Glass

Silver thin films were deposited easily through thermal evaporation on glass and Bakelite substrate. The sheet resistance obtained on glass substrate lied in the lower limit range of our requirement however; same can be increased by varying the film dimensions. Moreover, silver is an economical and readily available material which contributes positively towards selection of this material for thin film resistor fabrication.

5.7.3 Gold Film on Glass

Thin films of gold deposited on glass substrate also exhibited promising results and met our requirements of sheet resistance. Although gold is more expensive than silver however, the variation in resistance of gold film by changing its thickness is not as abrupt as of silver film. Therefore, gold thin film deposited on glass substrate is also a suitable candidate for thin film resistor.

5.7.4 Nichrome Film on Glass

Nichrome thin films deposited on glass showed highest sheet resistance and conforms well to our requirements. The material is also economical and can be deposited as thin film with as comfort as other two materials considered. The hardness of the bulk material is also more than that of other two materials. Therefore, Nichrome is considered to be most suitable material for fabrication of thin film resistor.

Conclusion

Drawbacks of Bridge-wire detonators restrict their usage in high speed missile application. The bridge-wire of the detonator tends to break when high gravitational loads act upon it. Thin film bridge detonators are used as an alternative to bridge-wire detonators. The bridge-wire technology is replaced with thin film bridge resistor to achieve more reliable results. Different materials have been considered for manufacturing insulating substrate and also for the resistive thin films for fabrication of thin film resistor. Bakelite and Glass were considered as substrate materials while the materials chosen for thin film were Gold, Silver and Nichrome. The materials were deposited individually on each substrate through Physical Vapor Deposition Technique by evaporating the target material and condensing onto the substrate to form film. Gold and Silver were deposited using 'Resistive Heating Unit' while Nichrome films were deposited using 'Electron Beam Evaporation' method. The fabricated films were analyzed through different characterization techniques to determine the structural, morphological and electrical properties of the films. Morphology of films was monitored through SEM and AFM which revealed that the films deposited on glass substrate were uniform and smooth with little roughness while those on Bakelite substrate were uneven with cracks in them. The structural properties were monitored using X-Ray Diffraction which was matched with relevant JCPDS. The electrical properties were determined using two probe method which revealed that highest sheet resistance is exhibited by Nichrome while lowest by Silver. Therefore, Glass is observed to be better than Bakelite as it offers high uniformity, repeatability and smoothness to the films whereas Bakelite, having high surface roughness, produces thin films with cracks and deformations. On the other hand, amongst the thin film materials, Nichrome provided best results and is considered most suitable for fabrication of thin film resistor because of its high resistivity, low price and ease of availability. Gold also produced results within our required range however, being expensive; Gold is not considered suitable material for bulk manufacturing of thin film resistors. Silver is most economical amongst the three materials and is most readily available in market however; silver shows slightly less

sheet resistance due to its high conductive nature. Yet it can also be used by modifying film dimensions to attain sheet resistance of required value.

Recommendations

It is recommended that glass substrate in the shape of initiator (similar to Bakelite) may be prepared and thin films of Nichrome may be deposited in similar manner as described in this report for fabrication of thin film resistor. Film resistor may then be used to replace bridge-wire resistor in any specific electric detonator and firing parameters may be observed by practically firing the detonator. The project being very wide in nature can also be tried with other materials. The materials can be single metals as well as alloys. It is also recommended that bi-layer and multi-layer films be developed to observe the effect on resistor and firing parameters of the detonator. It is further recommended that the thin films may be deposited on certain other substrates like quartz and alumina followed by conduction of their mechanical tests. The mechanical tests such as ‘Peal Test’ and ‘Scratch Test’ would certify the strength and reliability of thin film resistor for usage in special environment.

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