CHAPTER 1

INTRODUCTION

1.1 Background

One of the aluminothermy reactions is a reaction between 2 mol Aluminium (Al) with 1 mol of Ferum Oxide (Fe2O3). The special characterizes of aluminothermy reaction is it behaves exothermic with high calories release around 850kJ. Theoretical calculation of temperature of this reaction is possible to reach around 3000 C.

The calories released by this reaction is high enough till its been utilized in the various technical area. The common area where this reaction is used is in the Thermite Welding.

Besides this, thermite reaction is also used to obtain metal from metal oxides (extractive metallurgy). The advantage of this reaction in the metallurgy industry is that the metal obtained from thermite reaction have high

One of the most used methods of extracting metals from their ores (usually oxides) is the chemical reduction of these oxides by means of a reducing agent, often carbon or another metal. This principle has been put to industrial use basically since the Bronze Age. Among these pyrometallurgical reductions, as they are known in chemical metallurgy, reductions with aluminium, referred to as aluminothermy, occupy a prominent class. Best known among these is what is colloquially referred to as

Thermite reactions. The term *Thermite* (etymologically probably a contraction between *thermal* and *dynamite*, on account of the almost violent generation of lots and lots of heat during the process) actually originally specifically refers to the reaction of iron oxide with aluminium powder during which liquid iron and liquid alumina (aluminium oxide) are formed in a most spectacular fashion.

Iron oxide + Aluminium ---> liquid Iron + liquid Aluminium Oxide (Alumina)

(1.1)

The convenient fact that both reaction products are generated in the molten state (thus allowing to obtain lump metal or even castings) is due to the fact that the reaction is accompanied by massive heat generation, sufficient to heat the reaction products to well above their respective melting points (3,730 Fahrenheit for alumina and 2,800 Fahrenheit for iron).

Metals (understood here as *chemical elements*) as diverse as vanadium, niobium, manganese, cobalt and chromium are industrially produced using aluminothermy. Alloys (binary or complex) of these but involving also iron, molybdenum, tungsten, tantalum, osmium and others can be produced similarly. Many other elements, including copper, silicon, boron, lead, tin, scandium, nickel, zinc and a whole raft of others are not usually prepared industrially in this way but can be (and have successfully been) produced by backyard scientists.

1.2 Objective

The main objective of this project is to find alternative way to produce Metal Oxide using Thermite Reaction.. Today, iron is industrially hardly ever produced by aluminothermy but a whole array of more exotic metals and alloys is. And due to linguistic erosion, all aluminothermy processes (even those where iron plays no part) are now commonly referred to as ***thermite reactions*** **or** ***thermite reductions*** that will be used to produce Metal Oxide in this project.

1.3 Scope of Study

The Scope of Study for this Project will be: -

- Construct the Reaction Chamber for the Reactants
- Perform the experiment using the thermite reaction
- Gather the end results of the experiment (Metal Oxide)
- Perform few test to justify the characteristic and purity of the Metal
    - X-Ray Diffraction (XRD)
    - FESEM-EDS
    - Optical Spectrometry Microscope

1.3.1 X-Ray Diffraction Technique

X-ray diffraction finds the geometry or shape of a molecule using X-rays. X-ray diffraction techniques are based on the elastic scattering of X-rays from structures that have long-range order. The most comprehensive description of scattering from crystals is given by the dynamical theory of diffraction.

- Single-crystal X-ray diffraction is a technique used to solve the complete structure of crystalline materials, ranging from simple inorganic solids to complex macromolecules, such as proteins.
- Powder diffraction (XRD) is a technique used to characterize the crystallographic structure, crystallite size (grain size), and preferred orientation in polycrystalline or powdered solid samples. Powder diffraction is commonly used to identify unknown substances, by comparing diffraction data against a database maintained by the International Centre for Diffraction Data. It may also be used to characterize heterogeneous solid mixtures to

determine relative abundance of crystalline compounds and, when coupled with lattice refinement techniques, such as Rietveld refinement, can provide structural information on unknown materials. Powder diffraction is also a common method for determining strains in crystalline materials. An effect of the finite crystallite sizes is seen as a broadening of the peaks in an X-ray diffraction as is explained by the Scherrer Equation.

- Thin film diffraction and grazing incidence X-ray diffraction may be used to characterize the crystallographic structure and preferred orientation of substrate-anchored thin films.
- High-resolution X-ray diffraction is used to characterize thickness, crystallographic structure, and strain in thin epitaxial films. It employs parallel-beam optics.
- X-ray pole figure analysis enables one to analyze and determine the distribution of crystalline orientations within a crystalline thin-film sample.
- X-ray rocking curve analysis is used to quantify grain size and mosaic spread in crystalline materials.

1.3.2 FESEM-EDS

A FSEM-EDS (Field Emission Gun Scanning Electron Microscope) can be utilized for high magnification imaging of almost all materials. With FESEM in combination with EDS (Energy Dispersive X-ray spectroscopy) is it also possible to find out which elements different parts of a sample contain. The instrument is very suitable for different kinds of investigations. It is possible to investigate e.g. the fiber

structure in wood and paper, metal fracture surfaces, production defects in rubber and plastics is often used for metals, which are well suited as the samples are electrically conducting.

Examples of analysis objects: Surface coatings, segregations and casting defects. By investigating fractures you can find out why a material has broken.

1.3.3 Optical Spectrometry Microscope

The optical microscope, often referred to as the "light microscope", is a type of microscope, which uses visible light and a system of lenses to magnify images of small samples. Optical microscopes are the oldest and simplest of the microscopes. Digital microscopes are now available which use a CCD camera to examine a sample, and the image is shown directly on a computer screen without the need for optics such as eyepieces. Other microscopic methods, which do not use visible light, include scanning electron microscopy and transmission electron microscopy.

For the preparation for Titanium the metal should be cleaned properly before hand. Further the metal should be portion out to a small piece to be mounted on a block for the observation. The lens of microscope should be started by observing using the lowest magnification, 50x, 100x, 1000x, and 5000x.

CHAPTER 2
LITERATURE REVIEW

2.1 Thermite Reaction: Principles

A thermite reaction (sometimes called a "**Goldschmidt** reaction") refers to a very exothermic process occurring between a metal Oxide and a more active pure metal. The more reactive metal reduces the metal Oxide, Oxidizing itself and releasing a substantial amount of energy during the reaction.

Generally, thermite is made by mixing Iron Oxide and Aluminum powder and igniting it at very high temperatures (a few thousand degrees). The reaction releases so much energy, molten Iron metal is produced as one of the products.

The two most common types of thermite are made using either Iron(III) Oxide, Fe_2O_3 (also known as Hematite), or using Iron(II, III) Oxide, Fe_3O_4 (also known as Magnetite). The Iron Oxide is mixed with finely powdered Aluminum metal. When the thermite reacts, liquid Iron metal and Aluminum Oxide, Al_2O_3, is produced as a result.

Other, more exotic forms of thermite can also be produced. Using other metal Oxides, one can produce other, sometimes more powerful, blends of thermite. For instance, substituting Copper(II) Oxide for Iron Oxide in a thermite mixture can produce a very brightly burning reaction which yields Copper metal as a result. Although Copper Oxide thermite is probably the most common of the exotic thermites, one could also use other metal Oxides such as Tin Oxide, Lead Oxide, or

any other metal Oxide which could be reacted with a reducing metal (such as Aluminum or Magnesium). They key is that the reducing metal must be sufficiently higher on the activity series than the metal Oxide in order to support the single replacement reaction.

Thermite has found some use as a crude method of welding metals due to the intense heat and molten metals produced by the reaction. Thermite reactions can also be used on occasion to produce pure metals from their oxide counterparts as long as the reaction taking place is thermodynamically favorable

2.2 Titanium(IV) Oxide

$$3TiO_2 + 4Al \longrightarrow 3Ti + 2Al_2O_3 + \Delta H \quad (2.1)$$

According to the reaction's stoichiometry, the ratio of TiO_2 to Aluminum powder by weight is about 2.2 to 1 (2.22 to 1 to be more exact).

The change in enthalpy of this reaction is calculated to be, ΔH = -366.69 kJ assuming that both the Titanium metal and Aluminum Oxide are in the liquid state after the reaction, as they solidify, they release additional energy, bringing the total change in enthalpy to, **ΔH = -519.40 kJ** per 347.52 grams of thermite (-1.495 kJ/g).

In practice, however, the reaction does not appear to proceed as described above. The Aluminum metal does not seem to reduce the Titanium (IV) Oxide all the way down to Titanium metal but rather stops at a less-oxidized state of Titanium. A black Titanium Oxide, which is likely to be Titanium (III, IV) Oxide, is left after the reaction ceases. Upon analysis, one can further reduce the black Titanium Oxide further using Magnesium as a reducing agent. Doing so one can obtain a golden-yellow colored substance, which is presumably Titanium (II) Oxide. Titanium (II)

Oxide, TiO, is said to be golden-yellow colored, Titanium(III) Oxide, Ti_2O_3, is said to be violet colored, and Titanium(III, IV) Oxide, Ti_3O_5, is said to be black colored.

2.3 Thermite Reaction Experiment

2.3.1 Introduction

Titanium dioxide + *Aluminium* ---> *Titanium* + *Alumina* (3.1)

Does not generate enough heat for the reaction products to heat to above their respective melting points (the Melting Point of titanium is 3,034 Fahrenheit), leaving the experimenter with a sintered mass of solid alumina, with solid, powdered titanium metal locked into the alumina matrix. Only melting the whole thing to above 3,730 Fahrenheit, the Melting Point of alumina, would make the recovery of lump titanium metal possible.

There is of course a remedy to this problem and it's known in pyrometallurgical circles as *heat boosting*. Heat boosting is the technique whereby extra reaction heat is pumped into the reacting mix by running a second much hotter reaction simultaneously with the main reduction reaction, in the same reactor. In the case of aluminothermy, most usually a heat booster reaction is chosen that involves the oxidation of extra amounts of added aluminium powder with a powerful oxidizer such as Carbon.

2.3.2 Oxidizer's Role

Oxidizer + Aluminium ---> Alumina + by-product

Oxidizers capable of oxidizing aluminium with great generation of heat are a plenty (in fact, all the metal oxides suitable for thermite reductions are great oxidizers, it just so happens that titanium dioxide isn't very good on its own).

Commercially used heat booster oxidizers include chlorates, perchlorates, nitrates and sulphates (there are others, less frequently used).

The oxidizer chosen for this particular method of backyard metal production is calcium sulphate, more commonly known as plaster of Paris, gypsum or drywall.

2.3.3 Ingredient's Specifications

Titanium dioxide:

A *good quality* grade of titanium dioxide, in the form of fine flour, of good, clean white colour is advisable. Exact granulometry isn't critical. If lumpy, sieve it with a tea strainer or such like. The lumps can later be recovered by gently grinding in a mortar and pestle.

Calcium sulphate (Pottery):

Use no-frills wall filler, the cheaper the better. High-end of the market products may contain additives to regulate setting speed or wetting behavior that may (or may not) be somewhat detrimental to the thermite process.

The wall filler needs to be thoroughly dried to drive off inevitable crystal water (this would otherwise be driven off during the reaction and that could lead to spattering or a porous slag metal mix) and to ensure it's made up mostly of *anhydrous calcium sulphate*. Dry at high heat for about two hours by spreading the product in an ovenproof dish or stainless steel pan. Drying can be carried out (completely safely, wall filler isn't toxic). The wall filler will probably darken slightly in colour: this is normal. After drying and cooling, store it in a dry, hermetically closed container (e.g. a rubber sealed pickling jar), where it will keep dry indefinitely. It is not particularly hygroscopic but will, if exposed, slowly pick up moisture from the air.

Aluminum powder:

The grade isn't critical. I would advise against too finely ground grades, about 200 - 400 mesh is what advisable to use. Higher mesh (finer powder) may lead to too fast reactions and too high temperatures and hence a formulation adjustment may be required. And really coarse aluminum (shavings or turnings) would probably still work fine, provided really large (1 kg or more of mix) batches are used.

Ground Fluorite:

Fineness isn't critical either. Something of the consistency of fine sand is great. There is no such thing as "too fine" Fluorite, as this species doesn't actually take part in the reactions but merely melts and increases the slag fluidity. Its role is that of a *slag fluidizer*. Analogy of the metal coalescence from the molten slag/metal mix and the separation of oil and vinegar in a salad dressing, please note that this analogy is in fact very fair and quite accurate. To promote the separating of the metal from the molten sulphide/alumina mixture, it's beneficial to greatly improve its fluidity

(reduce its viscosity in other words), as the coalescing metal droplets will find it much easier to sink through the molten cauldron and meet up.

Fluorite (chemically *calcium fluoride*) has a much lower melting point (2,555 °F) than both alumina and calcium sulphide and is at those temperatures highly mobile, thereby lowering the viscosity of the melt considerably and keeping it fluid also somewhat longer. It's also completely chemically inert in these conditions, as aluminum is incapable of reducing this particular fluoride.

Carbon (charcoal):

Carbon is an important reduction material to be used in a reaction. An application of wood charcoal was as a constituent of gunpowder. It was also used in metallurgical operations as a reducing agent, but its application has been diminished by the introduction of coke, anthracite smalls, etc. For example, charcoal may be used to smelt a variety of metals from aluminum to copper as it burns at the necessary temperature: 1,100 °C (2,010 °F)

CHAPTER 3

METHODOLOGY/PROJECT WORK

This project will consist of few sequence method, which will be conducted to test the product purity and characteristics. The whole process of the project is shown as below: -

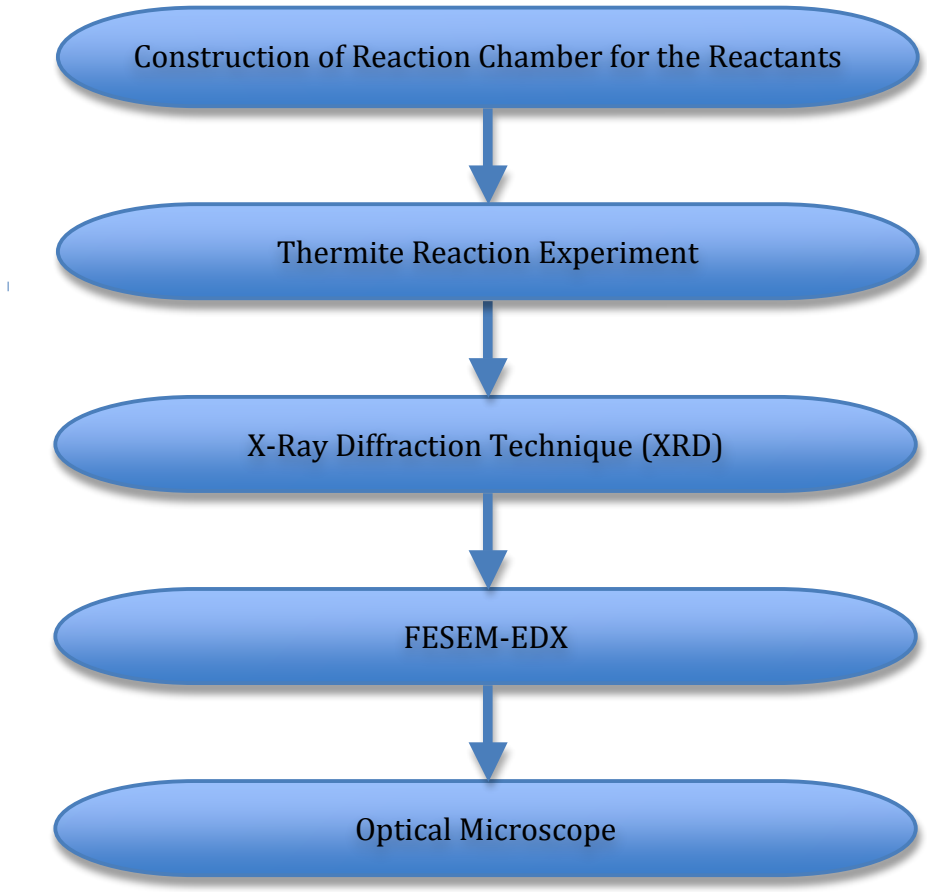


Figure 1: Flowchart of methodology

3.1 Constructing the Reaction Chamber

The Reaction Chamber constructing is the major step in this project, since the chamber behaves like a reactor for the reaction to take place and it is the first step of producing the metal oxide.

The reaction chamber is divided into 3 portions: -

Portion	Function
Reaction Portion	Area where the mixture is poured and reaction takes place
Separation Portion	Area where the molted of the reaction will be separated by charcoals (carbon)
Collection Portion	The separated molten will be stored at this area for cooling process

Table 1: Portions of Reaction Chamber

The reactor consists of few important components as below: -

No.	Component	Material Type	Function
1	Reactor Chamber House	Ceramic	1) Behaves as the container of the reaction and to accommodate all the components, mixture & end product 2) To prevent mixtures to spread out to preserve the reaction rate.

2	Gratings of the Chamber House	Steel Wire	1) To keep the Chamber House intact during the reaction
3	Top Cover of Reactor	Ceramic	1) Placed on top of chamber house to prevent the mixture from losing out from the chamber
4	Circular Mixture Mesh Gratings	Steel & Aluminum	1) Used as platform to place the mixture for the reaction in the reaction portion
5	Circular Separation Mesh Gratings	Steel & Aluminum	1) Used as platform to hold charcoals for the separation
6	Sparklers	Chemical	1) To ignite the mixture to allow reaction to happen
7	Outer Casing	Steel	1) Prevent from Reactor Chamber House from scattering
8	Sand	Silica	1) To absorb the radiation wave of the reaction to lessen the impact on the Housing ceramic 2) To keep the housing ceramic intact

Table 2:Component in Reaction Chamber

3.2 Design and Dimensions of Reactor

3.2.1 Reaction Chamber

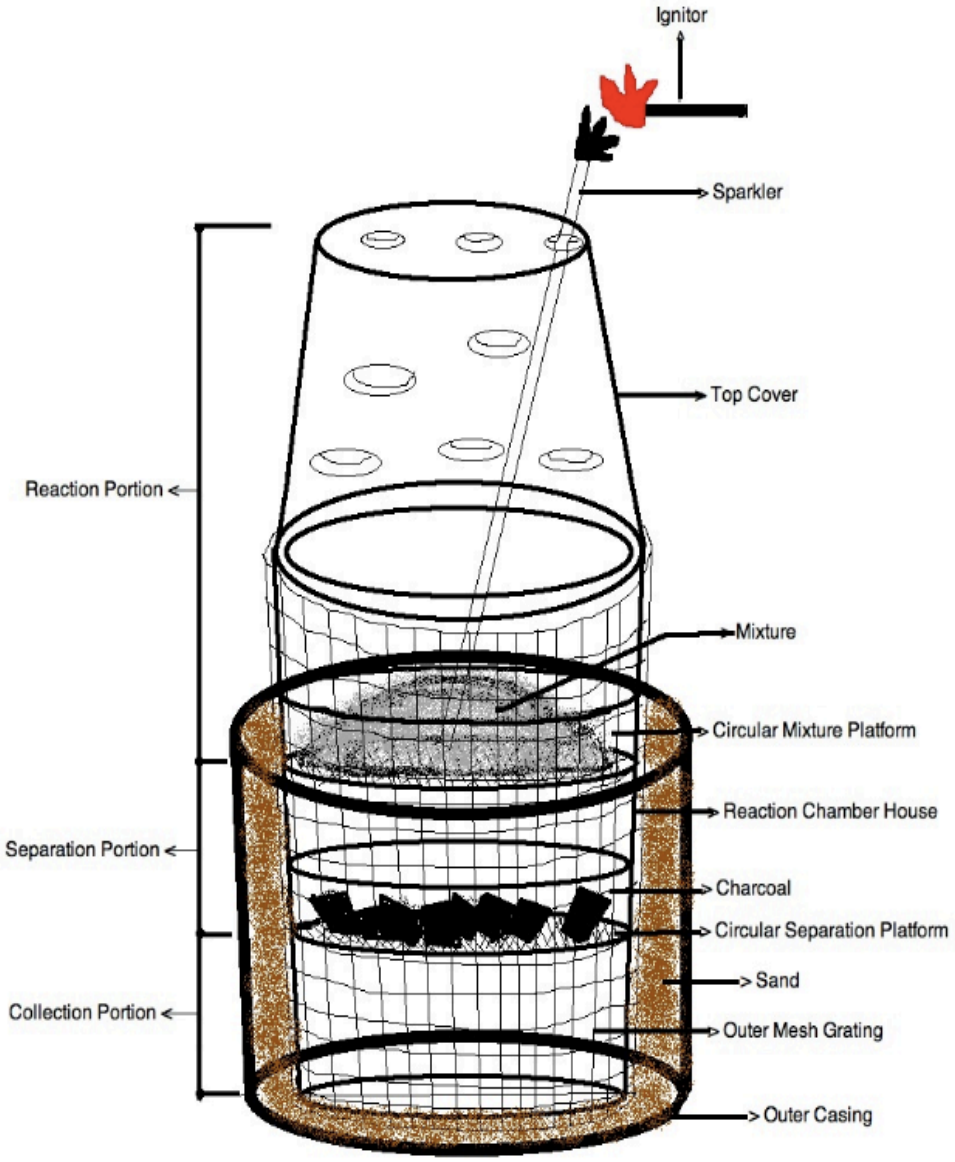


Figure 2:Reaction Chamber

3.2.2 Top Cover

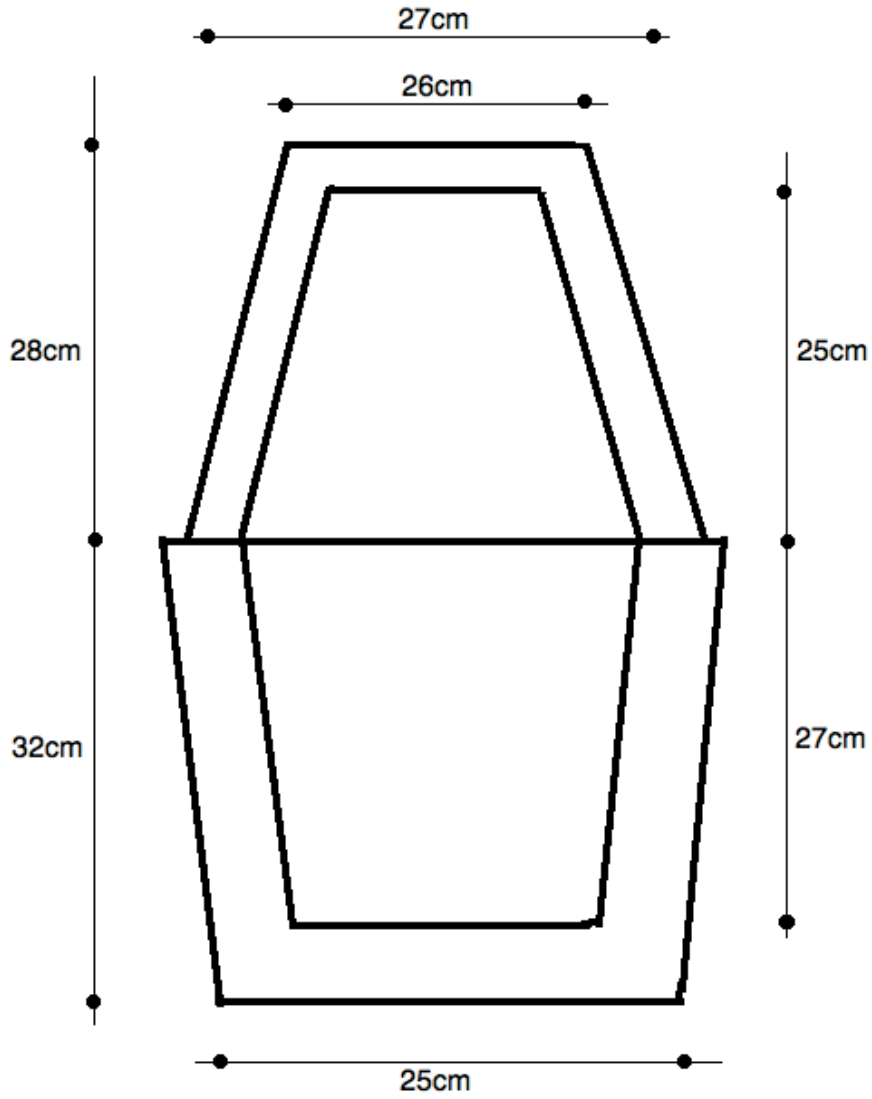


Figure 3:Top Cover

3.2.3 Reaction Chamber Housing

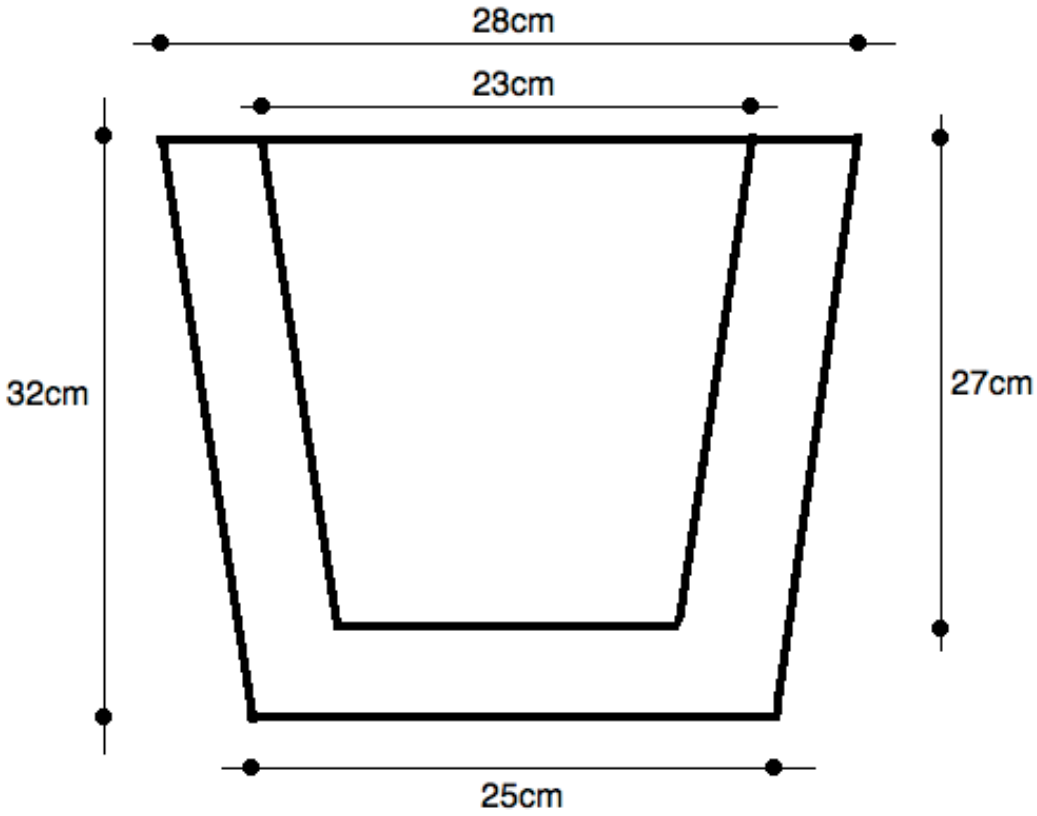


Figure 4:Reaction Chamber Housing

3.2.4 Circular Reaction Mesh Grating

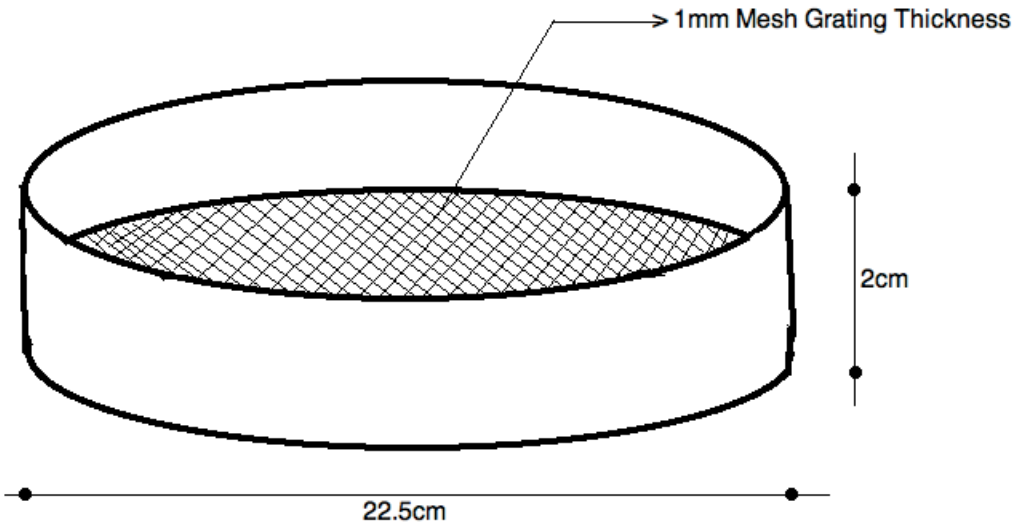


Figure 5:Circular Reaction Mesh Grating

3.2.5 Circular Separation Mesh Grating

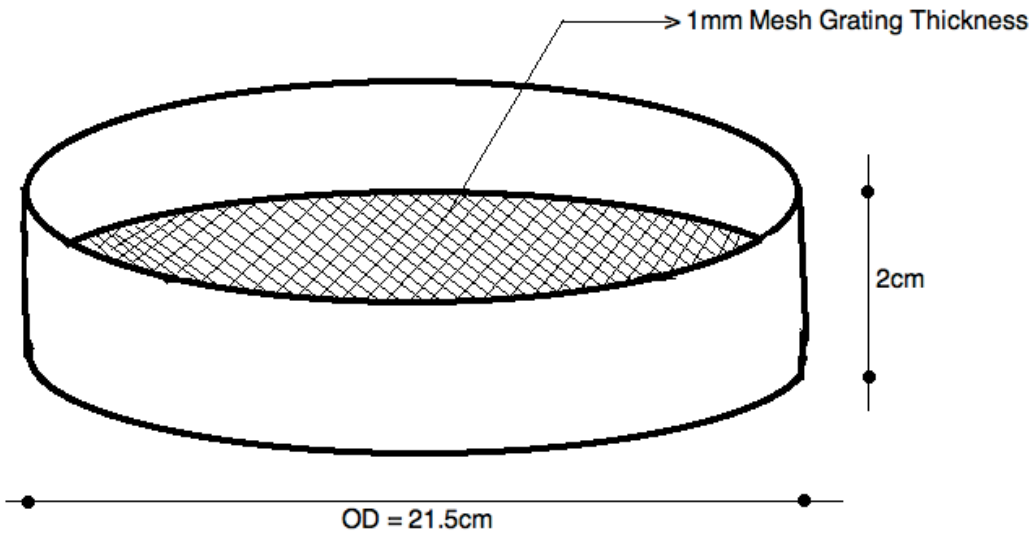


Figure 6:Circular Separation Mesh Grating

Figure 7: Image of Rector Chamber Housing

Figure 8: Interior View of Chamber

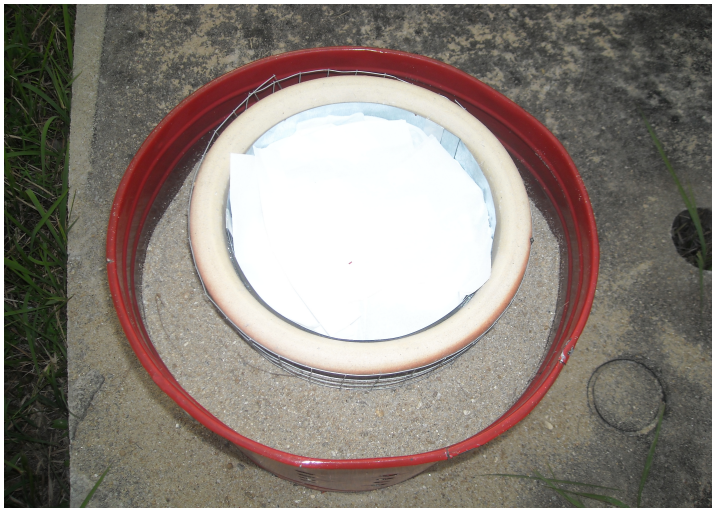


Figure 9:Chamber placed in the pool of sand

Figure 10:Fully Complete Reaction Chamber Ready to be ignited

3.3 Formulating the Mixture

The precise composition of the mixture for a 250 g reaction batch, is (quantities in gram):

Mixture Type	**Quantity (Grams)**	**Ratio of Mixture (Max 1)**
Titanium Dioxide	172.36	2.33
Aluminum Powder	77.64	1.0

Table 3:Ratio of Composition Mixture

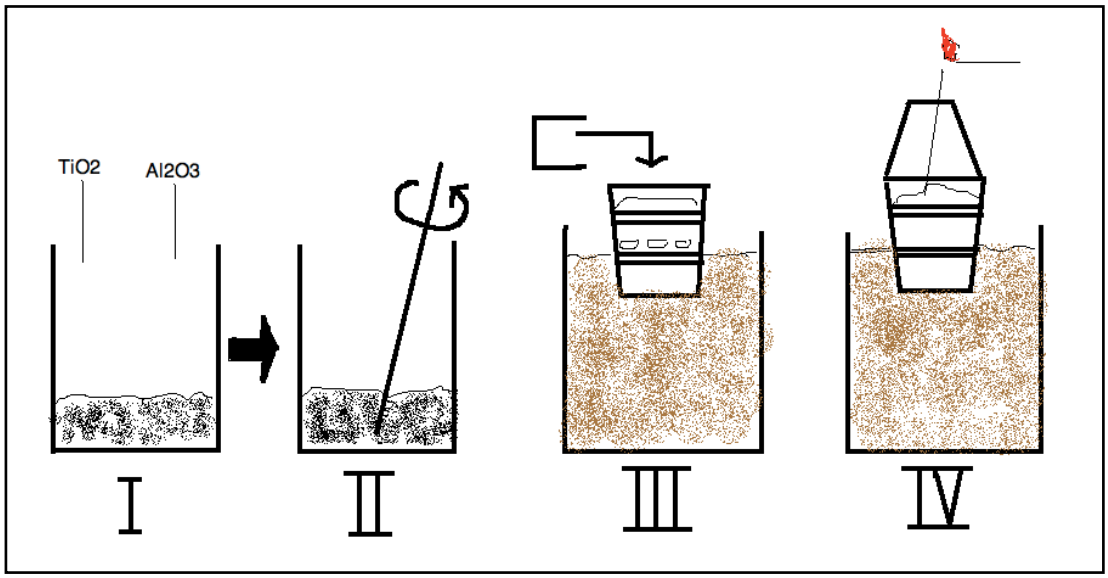
3.3.1 Weighing and dry mixing of the formulation

The mixture is weighed accurately using the electronic weighing machine.

The powders was dry mixed in a roomy container, typically a hermetically closed food container or similar, by shaking it. Couple of marbles is included in the container during mixing, as they slosh it greatly improves mixing efficiency. Mixing is done until a thoroughly homogeneous dry-mix is obtained, which was conducted for 30minutes.

3.4 Conducting the Experiment

The experiment is conducted in normal temperature (32 Celsius) in the late evening in order for more visible reaction.



I. Al & TiO2 is weighted and mixed in a beaker.
II. Both chemicals are well mixed
III. The chemicals mixture is poured into the ceramic compartment
IV. The reactor is closed by top casing and the sparkler ignited to allow reaction to take place

Figure 11:Steps of conducting the Thermite Experiment

3.5 Separation, Cleaning, & Photographing

The result of the metal oxide gathering process was separated in two ways:

1) Metal Oxide independently reacted and formed in the reaction chamber (Sample 1

2) Metal Oxide, which mixed with the carbon (Sample 2).

Both, Metal Oxide is cleaned using alcohol (40%)

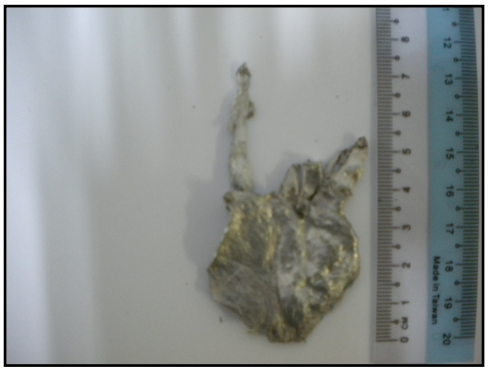


Figure 12: Independent Metal Oxide

Figure 13:Metal Oxide mixed with Carbon

3.6 Sample Preparation for XRD

X-ray diffraction (XRD) was used for identifying and confirming various reactant and product phases using Cu Kα radiation (wavelength = 0.154 nm) with a nickel filter and a secondary beam monochromatic.

Samples in the form of metal (5mm thickness) were scanned from 10◦ to 150◦ with a scanning rate of 2◦ min−1.

3.7 Sample Preparation for FSEM-EDS

Sample 1 is studied using FESEM with EDS with peak possibly omitted: 2.629 keV and all Processing option: (Normalized) with number of iterations = 6.

Sample 2 is studied using FESEM with EDS with peak possibly omitted: 2.629 keV and all Processing option: (Normalized) with number of iterations = 4.

Both samples in the form of metal (5mm thickness).

3.8 Sample Preparation for Optical Spectrometry Microscope

Optical study of the microstructure of the Metal Oxide was carried out using 5mm x10mm rectangular samples mounted on quick setting epoxy resin.

The mounted samples were polished with 60–600 grit papers followed by to ensure complete removal of all scratches. The polished samples were then etched with a chemical mixture containing hydrofluoric acid taken in the ratio of 6:2:1 by volume. The samples were then observed in an optical microscope under magnifications ranging from 50x to 200x.

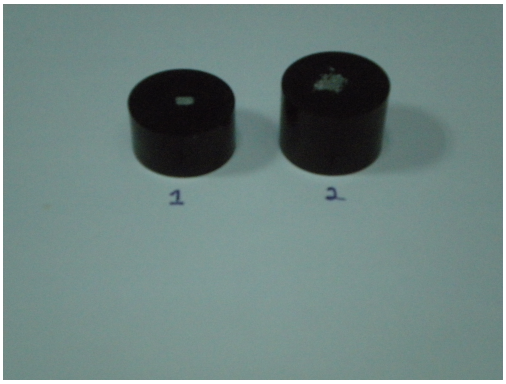


Figure 14:Sample Mounted on Epoxy Block

CHAPTER 4

RESULTS & DISCUSSION

4.1 Results of Thermite Reaction Experiment

The projects have major steps in producing the metal and also analysis steps. All this major steps can be predicted to have certain way of expected results.

Titanium Oxide + Aluminium ---> Titanium + Alumina + High Heat

The mixture of Titanium Oxide and alumina is referred to as the ***slag***. By combining the main reduction reaction (titanium dioxide + aluminium) with the booster reaction in the correct ratios, the required reaction temperature can be increased to almost any level, including that where all three reaction products (titanium metal, and alumina) are produced above their melting points.

After cooling, the metal (the most dense component of the mix) is then found at the bottom of the crucible, nicely protected by the slag from oxidation by air during the cooling step.

The experiment was conducted to gather the metal oxide. As the expected results the end product proven to be metal oxide. The metal will be scrapped out of the unwanted to get the solid to do the characterizes studies.

Figure 15 & 16: Reaction process of the mixture

Both sample 1 and 2 type of titanium is separated uniquely and is cleaned using methanol to prevent oxidation of the metals.

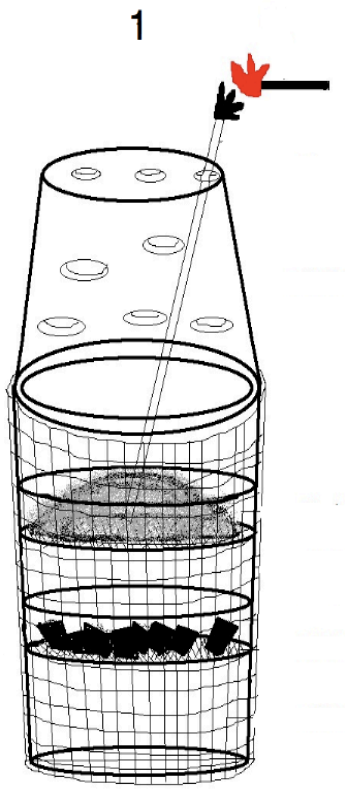
Mixture Type	**Quantity (Grams)**	**Ratio of Mixture (Max 1)**
Titanium Dioxide	172.36	2.33
Aluminum Powder	77.64	1.0
RESULTS		
Metal Oxide	0.645	

Table 4:Compostion of Mixture & Metal Oxide

4.2. Mechanism of Experiment

From analysis proves this experiment consist of 4 mechanism of reaction: -

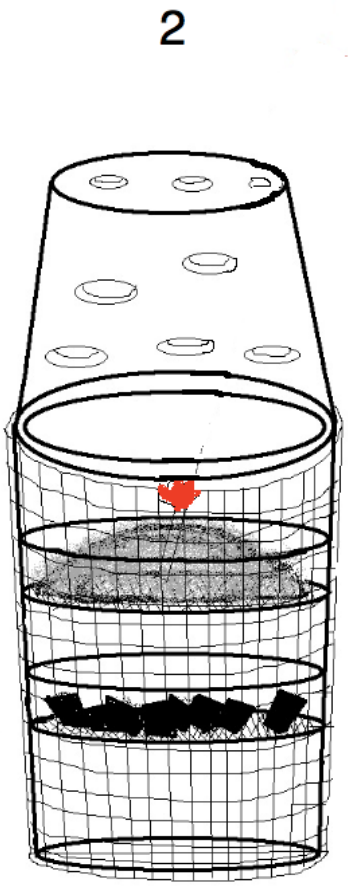
Mechanism 1



1. **The sparkler is ignited**
2. **The sparkler burn towards the mixture to create heat to activate the mixtures.**

Figure 17: Mechanism 1 of Experiment

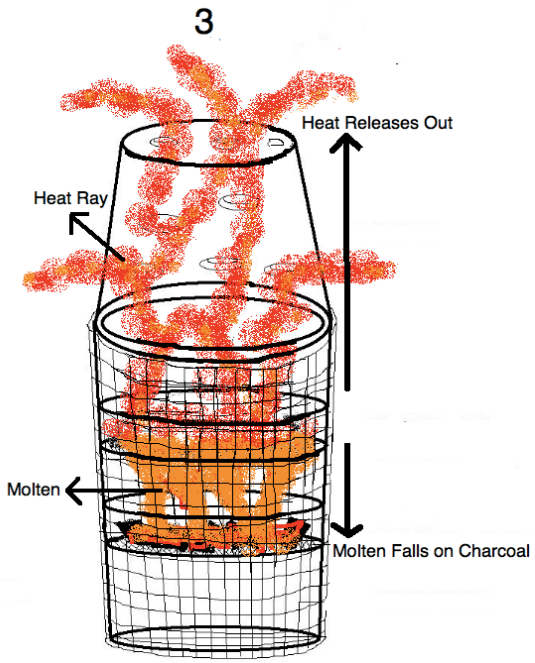
Mechanism 2



1. The sparkler is ignited
2. The sparkler burn towards the mixture to create heat to activate the mixtures.
3. **The sparkler hits the mixture and creates radiation heat on the mixture**
4. **The mixture actives & start reacting with minor sparks and light ray visible from outside**

Figure 18: Mechanism 2 of Experiment

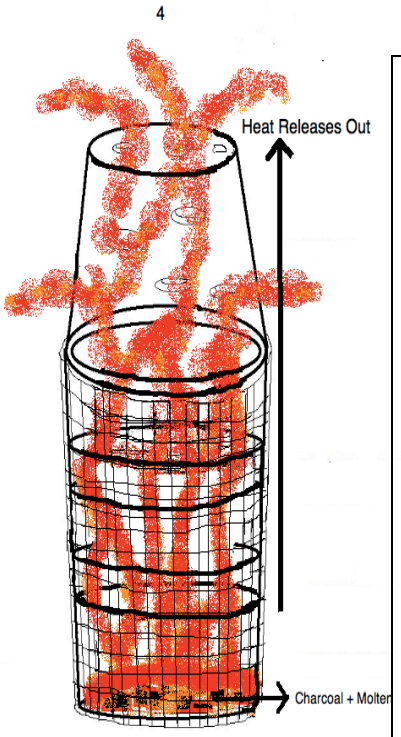
Mechanism 3



1. The sparkler is ignited
2. The sparkler burn towards the mixture to create heat to activate the mixtures.
3. The sparkler hits the mixture and creates radiation heat on the mixture
4. The mixture actives & start reacting with minor sparks and light ray visible from outside
5. **The mixture reacts and the molten falls on the charcoal**
6. **Due to high temperature the mesh grating melts as well**

Figure 19:Mechanism 3 of Experiment

Mechanism 4



1. The sparkler is ignited
2. The sparkler burn towards the mixture to create heat to activate the mixtures.
3. The sparkler hits the mixture and creates radiation heat on the mixture
4. The mixture actives & start reacting with minor sparks and light ray visible from outside
5. The mixture reacts and the molten falls on the charcoal
6. Due to high temperature the mesh grating melts as well
7. **The molten hits the charcoal and burns the mesh grating**
8. **This made both the charcoal and molten to fall into the collection portion**

Figure 20:Mechanism 4 of Experiment

4.3 Thermodynamic and Thermal Feasibility of the Reactions

$$3TiO_2 + 4Al \longrightarrow 3Ti + 2Al_2O_3 + \Delta H \quad (4.1)$$

The reactions involved during the thermite smelting and their corresponding free energy ($G\circ$) as well as heat ($H\circ$) changes are indicated below for their considerations.

Component	**ΔH_f^o (kJ/mol)**
Al(s)	0
Al_2O_3(s)	-1,669.8
TiO2 (s)	-940.1
Ti(s)	0

Table 5:Heat Formation of Component

$$H^o_{rxn} = (3\ mol)(\Delta H_f^o{}_{TiO2}) + (4\ mol)(\Delta H_f^o{}_{Al}) - (3\ mol)(H_f^o{}_{Ti}) + (2\ mol)(H_f^o{}_{Al2O3}) \quad (4.2)$$

$$H^o_{rxn} = (3\ mol)(-940.1) + (4\ mol)(0) - (3\ mol)(0) + (2\ mol)(-1669.8) \quad (4.3)$$

$$\mathbf{H^o_{rxn} = -366.69 kJ/mol}$$

$$\mathbf{H^o_{rxn(total)} = -519.40 kJ/mol}$$

The change in enthalpy of this reaction is calculated to be, **ΔH = -366.69 kJ** assuming that both the Titanium metal and Aluminum Oxide are in the liquid state after the reaction, as they solidify, they release additional energy, bringing the total change in enthalpy to, **ΔH = -519.40 kJ** for 250 grams of thermite (-1.495 kJ/g).

The reaction proceeds easily due to favorable free energy change, however, reaction

is not so favorable. TiO2 being a strong base easily combines with Al2O3 and an appreciable amount of titanium gets lost in the slag.

4.4 Results of FESEM-EDS

4.4.1 Sample 1

Sample 1 is studied using FESEM with EDS with peak possibly omitted: 2.629 keV and all Processing option: (Normalized) with number of iterations = 6.

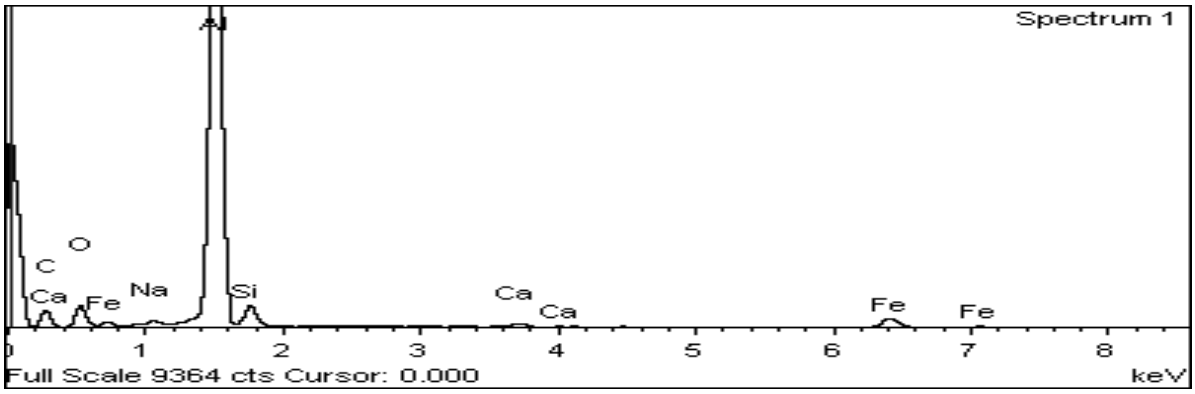


Figure 21:Spectrum of Sample 1 under FESEM-EDS

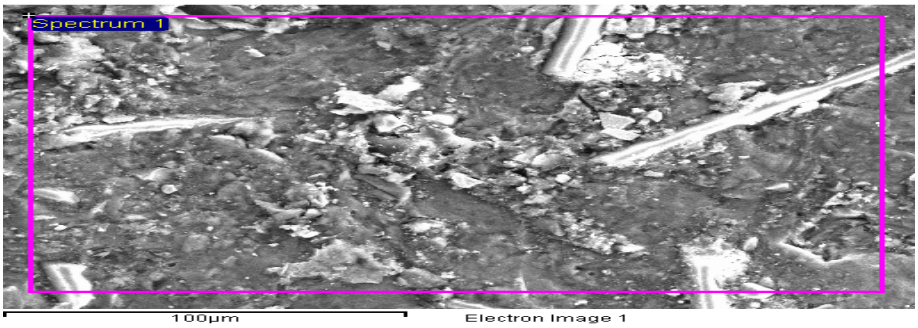


Figure 22:FESEM Image of Sample 1 Metal Oxide

Element	Weight%	Atomic%	Standard
C K	34.03	50.78	CaCO3
O K	15.45	17.31	SiO2
Na K	0.48	0.37	Albite
Al K	42.09	27.96	Al2O3
Si K	3.05	1.95	SiO2
Ca K	0.54	0.24	Wollastonite
Fe K	4.36	1.40	Fe
Totals	100.00		

Table 6:Element Composition for Sample 1

The results shows the most percentage of the Metal Oxide consist of Al to almost 43%. There is no Titanium Oxide element is detected in the Metal Oxide.

4.4.2 Sample 2

Sample 2 is studied using FESEM with EDS with peak possibly omitted: 2.629 keV and all Processing option: (Normalized) with number of iterations = 4.

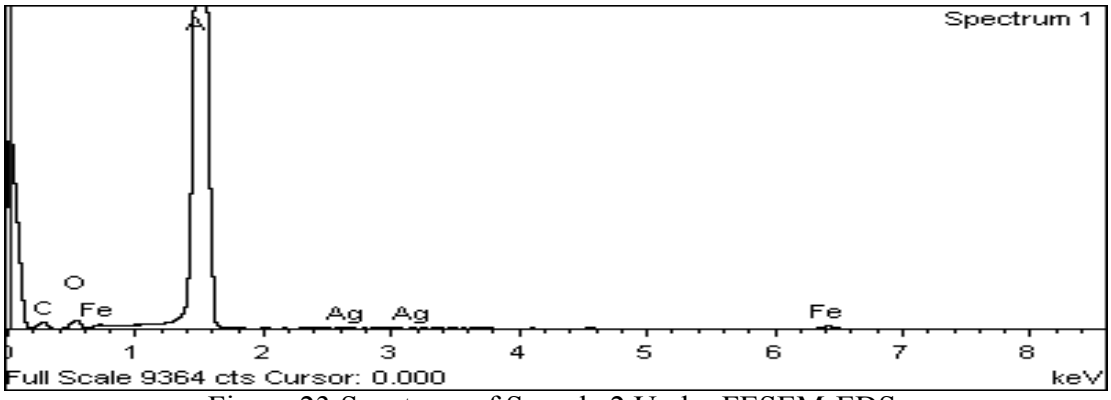


Figure 23:Spectrum of Sample 2 Under FESEM-EDS

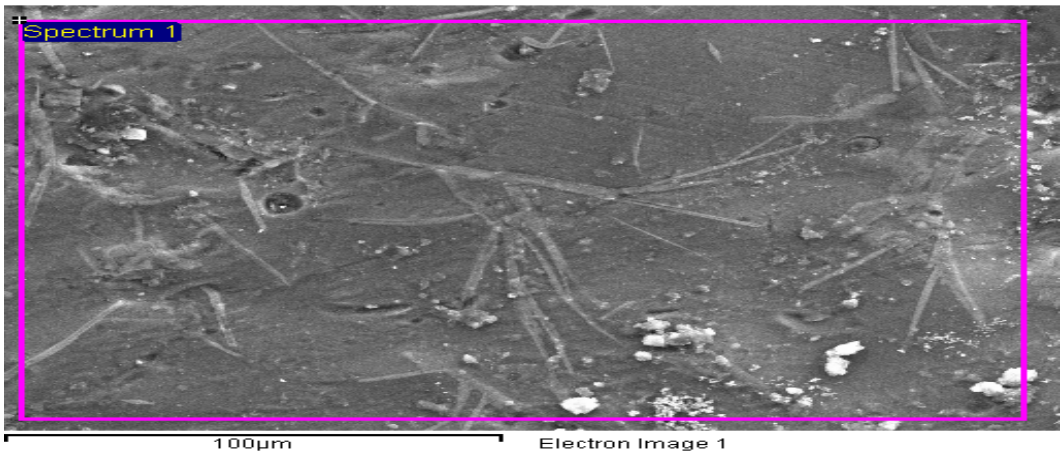


Figure 24:FESEM Image of Sample 2 Metal Oxide

Element	Weight%	Atomic%	
C K	23.95	40.39	CacO3
O K	6.61	8.37	SiO2
Al K	67.34	50.56	Al2O3
Fe K	1.58	0.57	Fe
Ag L	0.53	0.10	Ag
Totals	100.00		

Table 7:Element Composition for Sample 2

The results shows the most percentage of the Metal Oxide consist of Al to almost 68%. There is no Titanium Oxide element is detected in the Metal Oxide.

4.5 Results of Optical Spectrometry Microscope

Both the samples of titanium later obversed under the Optical Microscope to study the atomic structure of the samples. There are few steps involved to prepare the sample to be seen under the microscope. A small portion of the metal is taken from both the samples and mounted into a block. Both samples is etched using HCl

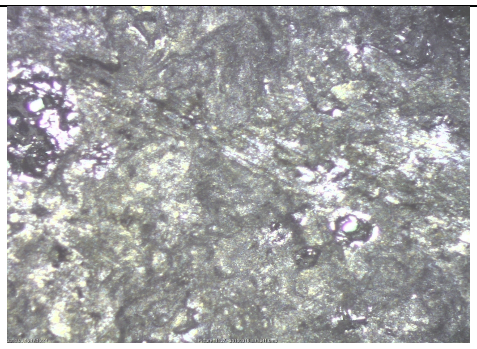
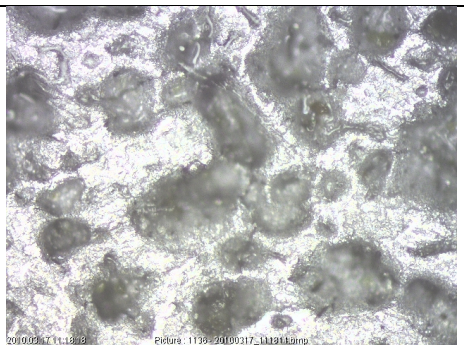
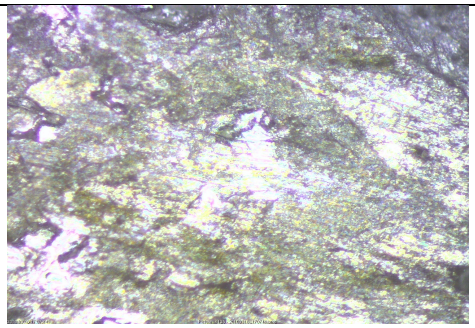
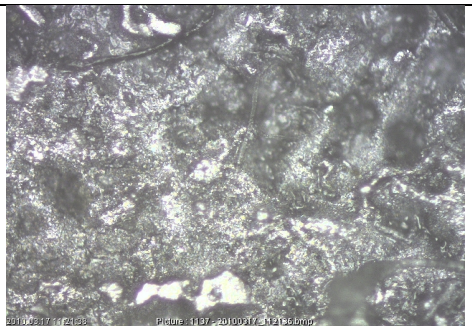
Sample	Before Etching	After Etching
1		
2		

Table 8: Optical Spectrometry Images

The image shows the Metal Oxide is Aluminum composition for both sample 1&

4.6 Results of X-Ray Diffraction

4.6.1 XRD Results for Sample 1

Phase identification of Metal Oxide was carried out by X-Ray diffraction technique. The diffraction pattern of the as reduced cum re-melted alloy obtained under optimum experimental. The plot exhibited 7 defined and sharp peaks altogether. Amongst these, the tallest peak was confirmed to be of elemental aluminum. However, no such peak for elemental titanium, or a peak corresponding to their compounds was observed.

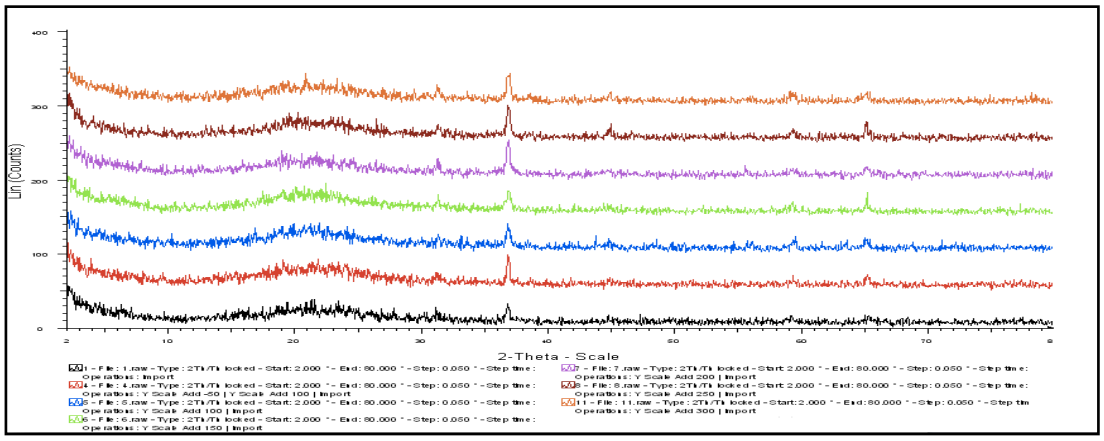


Figure 25:Spectrum for Sample 1 using XRD

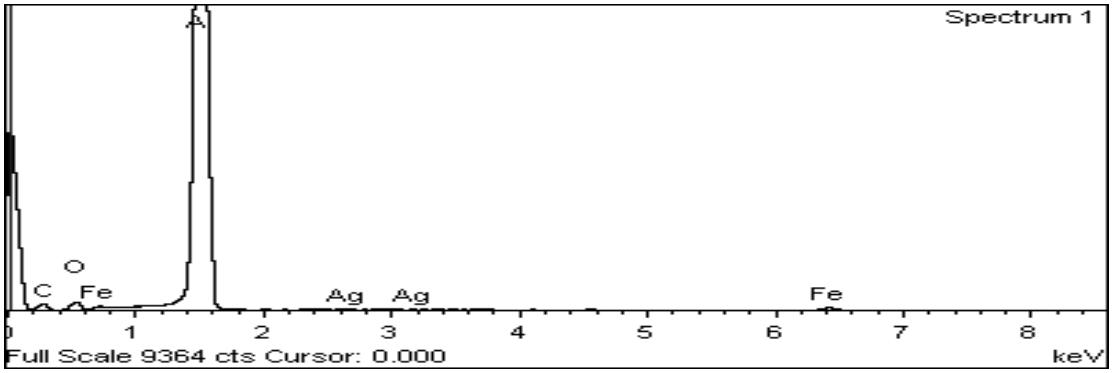


Figure 26:Spectrum Peak for Sample 1 using XRD

4.6.2 XRD Results for Sample 2

Phase identification of Metal Oxide was carried out by X-Ray diffraction technique. The diffraction pattern of the as reduced cum remelted alloy obtained under optimum experimental condition. The plot exhibited 5 defined and sharp peaks altogether. Amongst these, the tallest peak was confirmed to be of elemental aluminum. However, no such peak for elemental titanium, nor a peak corresponding to their compounds was observed.

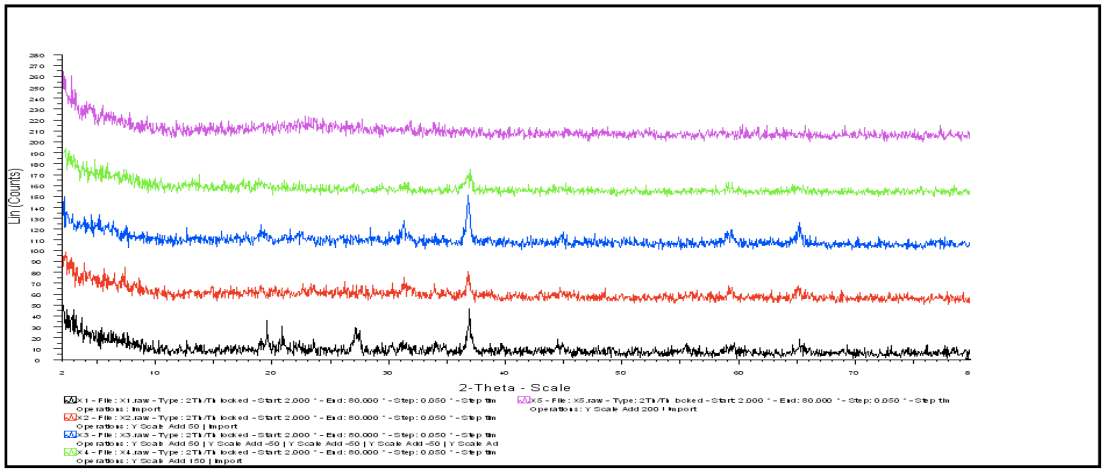


Figure 27:Spectrum for Sample 2 using XRD

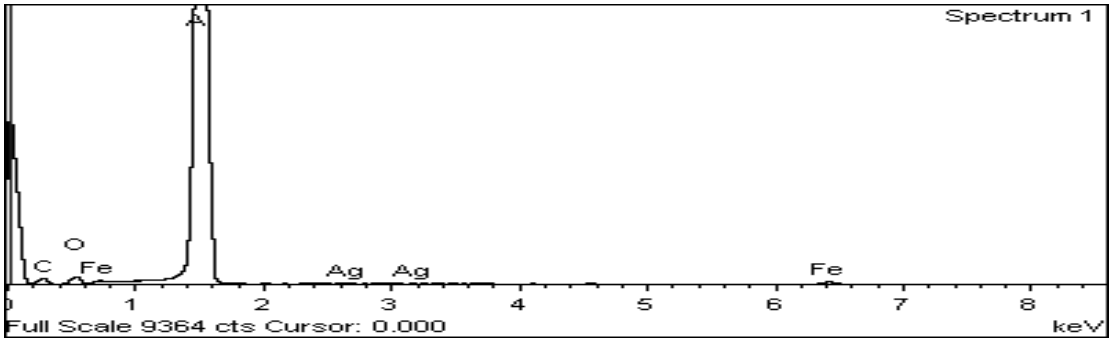


Figure 28: Spectrum for Sample 2 using XRD

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 Conclusion

Successfully synthesized aluminum using the thermite reaction. The composition of Aluminum & Titanium yield amount of 0.645g of Metal Oxide.

However the composition of Titanium in the Metal Oxide is not found in the spectrum composition of the metal. This may because of few reasons: -

1) The carbon used in the experiment might have oxidized the original metal to aluminium oxide due to the defects in the reaction chamber design.

2) Less oxygen intake into the reaction chamber caused the reaction to be less reactive and have and aluminium powder did not consume all titanium dioxide for the reaction

3) From thermodynamic point of view, combination of a fairly large amount of negative free energy and enthalpy changes (G° and H°) are desirable for a chemical reaction to proceed autogenously. It is evident from the above thermodynamic data of the reaction. In addition, mechanisms of the aluminothermy reduction of TiO2 also reflect difficulties in attaining complete reduction. TiO2 being a strong base easily combines with Al2O3 and an appreciable amount of titanium gets lost in the slag.

4) There is no reactant to behave as catalyst to enhance the reaction rate. The catalyst would help to increase the reaction rate between both reactants.

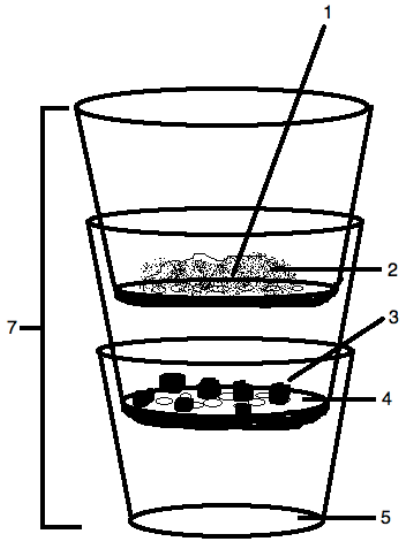
5) The selection of the material was misjudged, the temperature and heat factor of the reaction is more than expected, the construction component did not withstand the heat of the reaction. Further melted and affected the metal oxide composition.

5.2 Recommendations

There are few recommendation or alteration that can be done in this project to improve the results.

1) In order to counteract this tendency and improve the extraction of titanium, lime (CaO) is added to the charge. Lime being a stronger base than TiO2 combines preferentially with alumina and thereby facilitates reduction. CaO is more likely can be used as the catalyst for the reaction
2) Ensure the carbon do not fall into the collection portion of the chamber to avoid the oxide to re-oxide, which will affect the end product.
3) Allow more oxygen intake into the chamber in order to have sufficient oxygen for complete reaction of the reactants
4) Take note of the high temperature of the reaction to choose the higher heat and temperature resistance material to construct the reaction chamber.
5) Do not choose a very thick ceramic housing, the reaction must releases the energy by breaking the ceramic and releasing it outside.

5.2.1 Improvised Reactor Design (Recommendations)



1	**Sparkler**
2	**Mixture**
3	**Charcoal**
4	**The base of pot is more thick around 1.5cm & a lot of holes bored on the base (only for first 2 pot)**
5	**Collection Area**
7	**The stake consist of 3 individual pot Connected to each other**

Figure 29:Recommended Design of Reactor

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