Synthesis of Aluminum, Teflon & Viton (ATV) Pyrotechnic Composition and its Applications

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Certificate

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Abstract

Aluminum and Teflon based composition are widely used in IR decoy flares formulation because of their large energy output compared to other pyrotechnic mixture, low hygroscopicity, low dependence of burning rate on pressure and temperatures, and relatively high degree of safety in preparation. Viton (binder) is commonly added to AT mixture for easy processing by improving homogeneity of mixture and also protect aluminum against oxidation by moisture during storage. This work focuses on the synthesis characterization and thermal analysis of ATV composition. We have employed shock gel synthesis method using auto-clave at about 3500 kPa and 200^0C with continuous stirring and continuous supply of liquid CO_2. The main advantage of this procedure is that ATV composition energetic material can be synthesized without producing large volume of hazardous waste. The synthesized material has been characterized by XRD, TGA/DTA, FT-IR, SEM techniques. XRD results shows phase purity and crystalline nature of ATV. FTIR spectroscopy confirmed the formation of ATV composition while, SEM analysis shows the nano-morphology of aluminum particles. Thermal endurance of synthesized ATV was investigated by TGA/DTA, which showed that ATV has good environmental stability and only gets oxidized in air at higher temperature range. In addition to exceptional energy output ATV composition has excellent environmental and thermal stability characteristics and good compatibility with most materials used in pyrotechnic ordnance.

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“He who doesn’t thank to people is not thankful to Allah”

All praise for **Almighty Allah** alone, the most Merciful and most compassionate, source of all knowledge and wisdom and creator of the universe with its mysteries of logic, who I sour benefactor and has the power and can carry out His will. His revelations are source of knowledge and call us to think about what is happening around us, infact there are indications in them for all believers.

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Table of Contents

List of Figures

List of Tables

Chapter 1: Introduction

1.1. Energetic Materials

Large amount of energy is released when energetic materials are ignited. Main classes of energetic materials are like, pyrotechnic compositions; explosives propellants, and fuels etc, Energetic materials store energy chemically. There are two main approaches to obtain energetic materials; one is composite and other is monomolecular. In composite system required characteristics can be achieved by changing ratio of fuel and oxidizer, and as a result of complete balanced ratio of fuel to oxidizer we can obtain maximum energy density. Due to granular nature of composite energetic materials, reaction kinetics are largely controlled by the reactants, so the release rate of energy of a composite materials is below that may be attained in kinetically controlled process. [1]

1.2. Types of Energetic Materials

Types of energetic materials include:

1. Explosives
2. Propellants
3. Pyrotechnic

1.2.1. Explosives

Explosive materials are designed to release large amount of energy in a short time. The explosion arises through the reaction of a solid or liquid chemicals or vapors to form more stable products. So an explosive material is a reactive substance having great amount of energy, produce an explosion when it ignites, usually in the form of light, heat, sound, and pressure.

Fig 1.1 1.25-lb M112 demolition charges consisting of a C-4explosive atop degraded weaponry scheduled for destruction

Energy stored in above mentioned materials may be

I. Chemical energy
II. Pressurized compressed gas
III. Nuclear energy

Explosive materials are classified by the rate at which they release energy. Materials that can set off are called the "high explosives" and materials that can deflagrate are called the "low explosives". Rate of reaction of high explosive (i.e. 5,000 – 8,000 m/s) is faster than that of sound in explosion. Sensitive materials, which can initiate with comparatively small amount of pressure or heat, are called “primary explosives” and those that are comparatively insensate are called secondary or tertiary explosives.

Classically, most widely used explosive was black powder, was too sensitive to water, and as a result evolved lots of dark smoke. Nitroglycerin was the foremost useful explosive that is stronger than black powder but nitroglycerin was unstable. Hence, it was replaced by nitrocellulose due to its instability. Now new type of explosives such as trinitrotoluene and C-4 are widely used. In addition explosive devices are too much improved due to availability of chemicals.

1.2.1.1. Types of Explosive Material

a- Chemical Explosive

Since spontaneous chemical reaction occurs when an explosion takes place. When initiated, large exothermic change occurs with an addition large positive entropy change. Also, thermodynamically, it is very favorable. Huge amount of energy is present in chemicals bonds of explosive materials, and this is the result of formation of single, double, triple and multiple bond formation in these explosive materials. A number of phenomenons are taking place with explosive materials such as;

i) Decomposition

Decomposition of explosive materials may take place in a very short time or in a very long time it depends upon demand. Normally in storage slower processes of decomposition take place. Too rapid form of decomposition takes place in micro or nano seconds.

ii) Deflagration

Deflagration is initiated in open air and the reaction propagates in a very slow manner or fast again depends upon the concentration of explosive material.

iii) Detonation

A shock wave is used to initiate the explosive material; such phenomenon is known as detonation. The shock wave propagates from the explosive materials in a very rapid way and mostly speed of the shock wave from the material is in thousand of meters per second.

b- Exotic Explosive

Exotic method of explosion is the major cause of exotic explosive materials. Examples of such materials include antimatter, high intensity lasers which have the ability to change the phase of substance into plasma state.

1.2.1.2. Classification of Explosive Materials

Explosives materials can be categorized on the basis of their rate of reactions. Therefore we can classify explosive materials into three types and their details are given below.

a- Primary explosives

Explosive materials which are too much sensitive to stimuli such as heat, static charges, impact, friction etc. a very small amount of energy is required to initiate these explosive materials. Normally whenever we define or compare primary explosive materials then we compared it with PETN and says that the explosive material that is sensitive then PETN. Nitrogen triiodide is a primary explosive material and it is not possible to handle it even without detonating. Primary explosives are normally used in that place where we require sensitivity and are commonly used in physical shock system and blasting caps. A very large amount of energy can be produced from few mg of primary explosive material. [2]

Example:

Alkali metal ozonides, Azo-clathrates, Chlorine oxides, Copper acetylide, Acetone peroxide, Hypofluorous acid, Ammonium permanganate, Lead azide, Lead styphnate, Azidotetrazolates, Lead picrate, Chlorine azide are certain examples of primary explosives

b- Secondary explosives

If we compare secondary explosive with primary explosive materials then it is clear that secondary explosive materials require normally more energy to be initiated than primary explosive. Since secondary explosive are less responsive therefore these explosive materials have lot of applications as compared to primary explosive like are largely used in an explosive train.

Example:

TNT and RDX are the examples of secondary explosive.

c- Tertiary explosives

Tertiary explosives are insensitive to shock, hence cannot be detonate to that amount, as primary and secondary explosive materials. They have low cost of material and handling as compared to other two types of explosive materials. Normally terrorists use this type of explosive material for attacks b/c it is easily available in the market like nitrate fertilizers.

Example:

Example of a tertiary explosive is ANFO.

1.2.2. Propellants

If we see that technically, the word propellan**t** is used generally for the chemicals which create thrust. And in case of vehicles we say that it is the propellants that propel vehicles on earth. And in case of rocket propellant is the combination of two propellants that is normally fuel and oxidizer. Now combination of three propellants is also used in order to generate more thrust in smaller time.

Fig 1.2 The Space Shuttle Atlantis during ascent.

Chemical propellants which are commonly available in the market are petrol, kerosene oil, liquid hydrogen, and an oxidizer. When these chemical fuels are burnt, they produce pressurized gas as propellant. Numbers of propellants are liquids that can easily be vaporized into the desired pressurized gas. Such propellants include the nitrous oxide that is dissolved in the butterfat canned whipped cream, and the dim ethyl ether or low-boiling alkenes used in hair spray.

Fig 1.3 Aerosol spray can

When a propellant is initiated, large amount of pressurized gas is produced that can be used as:

- For the production of thrust like produces in rocket motor.
- Chamber of a gun and the interior of an ammunition cartridge filled with propellant commonly known as gun propellant.
- Expansible bag or membrane can fill with gas generator propellants.
- Can be used as pressurized gas blaster in mining and demolition.

1.2.2.1. Types of propellants

a. Solid propellant

Normally propellant are made up of low explosive material but if we use such a material that is high explosive then we can reduce its concentration in such a way that whenever it burns, it should burn in a control way such that it should deflagrates rather than detonates. As a result of this burning, gas pressure is produced that accelerate the rocket and other motor etc. There are large numbers of propellants that are used in different fields which are normally solid in state. There are lot of examples of solid propellants such as gun powder or black powder, cordite, ballistite and smokeless powder etc

Solid propellant may be subdivided on the basis of grain and composition.

I Grain

Grain is defined as individual particle of propellant regardless of its size or shape. Grain shape and size properties define its burn time amount of heat and gas that is produced by a propellant.

With different grains size and shape there are three types of burns that can be achieved.

- **i- Progressive Burn:** Usually a grain with multiple perforations or a star cut in the center providing a lot of surface area.
- **ii- Degressive Burn:** Usually a solid grain in the shape of a cylinder or sphere.
- **iii- Neutral Burn:** Usually a single perforation; as outside surface decreases the inside surface increases at the same rate.

II Composition:

Solid propellant compositions are classified into four different types**:**

i- Single Based Propellant

Nitrocellulose is the chief explosives ingredient for single based propellant. Stabilizers and other additives are used to control the chemical stability and enhance the propellant's properties.

ii- Double Based Propellant

Nitrocellulose with nitroglycerin or other liquid organic nitrate explosives are added in double based propellant. Stabilizers and other additives are also used in double based propellant. Nitroglycerin reduces smoke and increases the energy output. Double based propellants are used in small arms, cannons, mortars and rockets.

iii- Triple Based Propellant

Nitrocellulose, nitroquanidine, nitroglycerin or other liquid organic nitrate explosives Triple based propellants are used in cannons.

iv- Composite

Nitrocellulose, nitroglycerin, nitroquanidine or any other organic nitrate are not used in composite. Composites usually consist of a fuel such as metallic aluminum, an oxidizer such as ammonium per chlorate, and a binder such as synthetic rubber. Composite propellants are also used in rocket motors.

b. Liquid propellant

Common propellant combinations used for liquid propellant rockets include:

- Liquid oxygen and liquid hydrogen
- Chlorine pentafluoride and hydrazine
- Hydrogen peroxide and alcohol or RP-1
- RFNA and Unsymmetrical dimethylhydrazine (UDMH)
- Red fuming nitric acid (RFNA) and kerosene or RP-1
- Dinitrogen tetroxide and UDMH, MMH, and/or hydrazine
- Liquid oxygen and kerosene or RP-1
- Liquid oxygen and ethanol

Common monopropellant used for liquid rocket engines include: Hydrazine, Hydrogen peroxide, Red fuming nitric acid (RFNA)

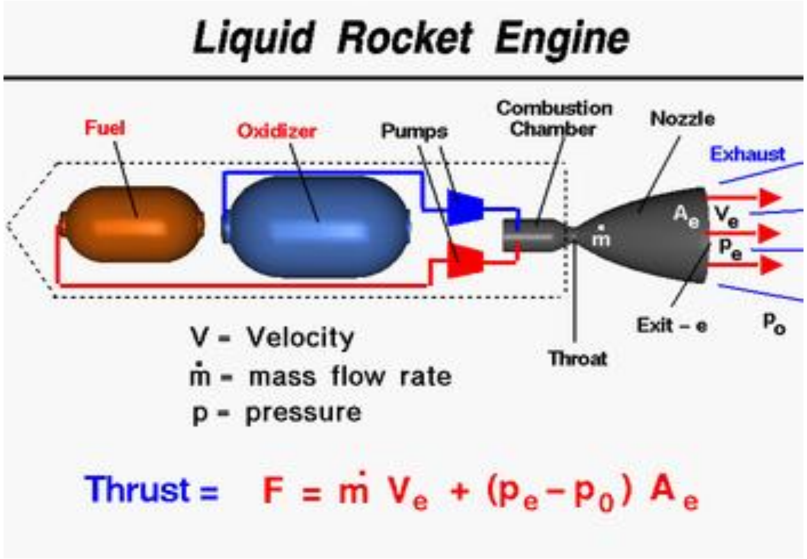


Fig 1.4 Schematic of a pumped bipropellant rocket

In rockets, three main liquid bi-propellants combinations are used:

- Cryogenic hydrogen oxygen combination system - Used in upper stages and sometimes in booster stages of space launch systems. This is a nontoxic combination. This gives high specific impulse and is ideal for high velocity missions.

- Liquid oxygen hydrocarbon propellant system - Used for booster stages of space launch vehicles. It has high density and hence allows for a more compact booster design
- Storable propellant combinations - used in large rocket engines for first and second stages of ballistic missiles and in almost all bi propellant low-thrust, auxiliary or reaction control rocket engines. They are instant starting and suitable for long-term storage.

1.2.3. Pyrotechnics

If we use an explosive material as self-contained and self-sustained exothermic chemical reaction for the production of heat, light, gas, smoke then such science is known as pyrotechnic. Pyrotechnics include not only the manufacture of fireworks but items such as safety, oxygen candles, explosive bolts and fasteners, components of the automotive airbag and gas pressure blasting in mining, quarrying and demolition. Pyrotechnicians are the individuals which are responsible for the safe storage, handling, and functioning of pyrotechnic devices.

Fig.1.5 Pyrotechnics gerbs used in the entertainment industry

1.2.3.1. Pyrotechnic composition

A pyrotechnic composition is a substance or mixture that produces heat, light, sound, gas or smoke or a combination of these. It is the result of a non-detonative self-sustaining exothermic chemical reaction. Pyrotechnic substances are not mostly relying on oxygen from external sources in order to sustain the reaction. [3]

1.2.3.2. Types of Pyrotechnic composition

There are number of types of pyrotechnic compositions and some of which are given below:

Flash Powder – it burns very fast and produces explosions and/or bright flashes of light

Gun-Powder – it burns slower than flash powder but produces large amount of gases

Solid Propellants – it produce large amount of hot gases and used as sources of kinetic energy for projectiles and rockets

Pyrotechnic Initiators – it produce large amount of heat, flames, and/or hot sparks and commonly used to ignite other compositions

Gas Generators – it produce large amount of gas of very high volume in a very short time in a controlled way

Ejection Charges – it burn fast and produce large amount of gas in a very short time and due to this reason it is used to eject payloads from containers

Burst Charges – it burn fast and produce large amount of gas in short time and used to fragment a container and eject its content

Smoke Compositions – it burn slowly and produces smoke which may be plain or colored

Delay Compositions – it burn at constant slow speed and due to this reason it is used to introduce delays into the firing train

Pyrotechnic Heat Sources – it produces large amount of heat and no gases and due to its slow-burning it is just like a thermite compositions

Sparklers – it produces white or colored sparks on ignition.

Flares – burn slowly, produce high amount of light, used for illumination or signaling

Colored Fireworks Compositions – produce light, white or colored

Pyrotechnic compositions are sometimes used in industry and aerospace for generation of large amount of gas in gas generators. Pyrotechnic compositions are also used in military when production of large amount of noise, light, or infrared radiation is required; e.g. missile decoy flares, flash powders, and stun grenades.

1.2.3.3. Pyrotechnic initiator

A pyrotechnic initiator is a device containing a pyrotechnic composition and commonly used to ignite other compositions which are very difficult to ignite e.g. thermites, gas generators, and solid-fuel rockets. Pyrotechnic initiator work in a very controlled way that's way it has lot of applications.

1.3. Pyrolant

Introduction

Energetic material that produces flame upon combustion known as pyrolant. It is a metal based pyrotechnic composition with any oxidizer. [4]

1.3.1. Thermite

Thermite is a pyrotechnic composition that consists of metal powder and metal oxide and exothermic reduction reaction takes place when it ignites. As we know that aluminum is a reducing agent therefore its reaction is an aluminothermic reaction. It is mixture of metal and oxidizer but its reaction is very similar to that of a fuel oxidizer mixture like black powder. Aluminum, Magnesium, Titanium, Zinc and others metals are also used as a fuel in thermite. But the most commonly used metal is aluminum, due to the property of high boiling point. [5]

1.3.1.1. Types

a. Iron thermite

Iron thermite is the most common composition. Mostly iron (III) oxide or iron (II, III) used as a oxidizer in this composition. Iron (III) is used when production of more heat is required but when we require easier ignition then we use iron (II, III) combination. Copper or manganese oxides are used for improved and easy ignition. [6]

Fig 1.6 A thermite reaction using iron (III) oxide. The sparks flying outwards are globules of molten iron trailing smoke in their wake.

b. Copper thermite

It is prepared by using either copper (I) oxide (red) or copper (II) oxide (black). This composition is used when less heat per mass is required as compared to iron thermite. But the burn rate tends to be very fast and the copper melting point is relatively low and as result this reaction produces a significant amount of molten copper in a very short time. Explosions can occurred when copper drops to considerable distance is send. [7]

Fig 1.7 Thermite reaction proceeding for a railway welding. Shortly after this, the liquid iron flows into the mould around the rail gap

1.3.2. Applications

Ignition

Under suitable conditions, metals are burnt and it is just like burning of wood or petrol etc. Since rust is the result of oxidation of steel or iron, as it takes place in very slow rates. The basic requirement is the correct mixing of metallic fuels which ignites, however ignition itself requires high temperature and such temperature is not achieved with conventional powder fuses, nitrocellulose rods, detonators, pyrotechnic initiators, or other common igniting substances. [8]

Civilian uses

There are number of uses of thermite reactions. It is not an explosive instead but it operates by exposing a very small area of metal at extremely high temperatures. As a result of this burning, intense heat is produced, which is focused on a small spot, can be used to cut through metal or weld metal components together. [9]

Military uses

Thermite hand grenades and charges are the most common used by armed forces in both an anti-materiel role and in the partial destruction of equipment. Its requirement is very common, when time is not available like emergency destruction. Iron-thermite is difficult to ignite, burns with practically no flame and has a small radius of action so additives are added in order to improve its incendiary effects. Thermate-TH3 is a mixture of thermite and pyrotechnic additives which have been found to be superior to the iron thermite. [10]

1.3.3. Safety / Fire Hazards

It is hazardous to use thermite due to its extremely high temperatures production and this production is so rapid that it is very difficult to stop the reaction when once initiated. One of such violent effect is shown in the figure 1.8. In industrial areas once these reactions initiated than a very violent reaction started and aluminum in this situation a very important rule that it oxides and capable the reaction up to a violent explosion. Pouring water on to thermite is very dangerous and it causes steam explosion.

Fig 1.8 The violent effects of thermite

1.4. Fluoropolymer Based Pyrotechnic Materials

1.4.1. Fluoropolymer

Multiple strong carbon-fluorine bonds exists in a fluorocarbon based polymer. Therefore it is characterized by high resistance to solvent, acid and bases. Fluoropolymer share the properties of fluorocarbon in that they are not as susceptible to the van der Waals force as hydrocarbons. This property contributes to their non-stick and friction reducing behavior. Due to the addition of multiple carbon fluorine bonds in chemical compound, fluoropolymer compounds are very stable. These compounds are mechanically characterized as asthermosets or thermoplastics. A fluoropolymer can be a homo-polymer or a co-polymer. Examples of fluoropolymer are PVF (polyvinyl fluoride), PVDF (polyvinylidene fluoride), PTFE (polytetrafluoroethylene), PCTFE (polychlorotrifluoroethylene) and PFPE (perfluoropolyether) etc.

1.4.2. Magnesium/Teflon/Viton (MTV)

It is assumed that the development of the MTV (Magnesium/Teflon/Viton) pyrolant system occurred sometimes in the mid of 1950s after large scale production of polytetrafluoroethylene (PTFE). MTV shows superior performance in terms of specific infrared radiant intensity as compared to many other pyrolant compositions. Polytetrafluoroethylene acts as oxidizer in propellants together with metallic fuels such as in MTV composition. In World War I a scientist French Berger invented a mixture of CCl_4 and zinc dust that produces black smoke, since then research started on infrared decoy flares amid at compositions yielding much heat and black carbon for the improved emission in the infrared region. MTV composition gives twice yield as compared to the old composition of CCl_4 and Zinc dust. In MTV composition PTFE acts as a fluorine source to serve exothermic fluorination of magnesium.

1.4.3. Aluminum/Teflon/Viton (ATV)

Aluminum/Teflon/Viton (ATV) is a pyrolant. Where Teflon is polytetrafluoroethylene, $(C_2F_4)n$, and Viton is vinylidenfluoride-hexafluoroisopropene-copolymer, $(CH_2CF_2)_n(CF(CF_3)CF_2)n$.

Thermites based on Aluminum/Teflon/Viton ATV-compositions, have been in use from mid of nineteenth century and use as a payload in infrared decoy flare applications.

1.5. Basic Components of Aluminum/Teflon/Viton

1.5.1. Aluminum

Aluminum is the third most abundant element on earth crust after oxygen and silicon. Its density is about 2.7 g/cm^3 and its melting point is about 660^{0}C. And it is ductile at 100 to 150^{0}C. In market main use of aluminum metal is as an alloying agent to make aluminum-magnesium alloys, sometimes called "magnalium" or "magnelium". [11]

1.5.2. Viton

Viton is a synthetic rubber and it is fluoropolymer elastomer. Viton polymer contains rubber goods from different sources. Viton performance depends upon nature of the polymer and compounding ingredients that is used to make the final product. Compatibility with hydrocarbon is very high but in case of ketones such as acetone it is incompatible.

1.5.3. Polytetrafluoroethylene (PTFE)

It is a synthetic fluoropolymer of tetraflouroethylene. Since it is fluoropolymer of tetraflouroethylene therefore it has lot of applications in different fields of work. And the best well known brand of polytetrafluoroethylene is Teflon. PTFE is a solid and it has high molecular weight and this compound completely consisting of carbon and fluorine. It is mostly non reactive due to strong bond of b/w carbon and fluorine. [12]

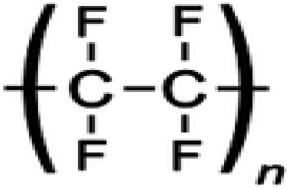


Fig 1.9 Polytetrafluoroethylene

1.5.4. Applications

a) Flare (pyrotechnic)

A flare which is also sometimes called a fuse and is a type of pyrotechnic that produces a brilliant light and intense heat without any explosion. Normally flares are used for illumination, signaling in both civilian and military. For maximum illumination of time for a large area it may be ground, projectile or parachute pyrotechnic. Projectile pyrotechnic may be used from an aircraft, from rocket or any other effective method.

Fig 1.10 Illumination flares being used during military training exercises.

b) Flare (countermeasure)

Flare is normally a pyrotechnic composition that is based on Aluminum or any other metal whose burning temperature is equal to the temperature of engine exhaust. And this is the basic reason to seek out infrared guided missile with the help of flare rather than the aircrafts engines.

Fig 1.11 A C-130 Hercules deploying flares

c) Decoy

Decoy is normally a personal device that is used for hunting and in wartimes it is used for resolving crimes.

Military Decoy

From military point of view decoy is way to make fool enemy forces that is attack on them. Quaker gun is one of the most prominent examples of military decoy. Large amount of military decoys are developed.

Fig 1.12 An inflatable dummy tank

g) Infrared homing

As infrared radiation is strongly radiated from a hot body whose temperature is very high. And infrared homing is basically a missile guidance system that follows and tracks the aircraft on the basis of radiation that it emits in infrared region. And we know that infrared region is just below the visible region. [13]

Fig 1.13 Infrared homing air-to-air missile

1.5.5. ATV vs. MTV

Project Inspiration

Fluorine is the most electronegative element, and thus is a superb oxidizing agent in reduction –oxidation reactions. Teflon is used as an oxidizer in pyrotechnic compositions. One of the earliest developed and the most reactive mixture containing PTFE is MTV composition which normally incorporates the binder Viton. MTV is used as a pyrotechnic in such applications as infrared decoy flares, signaling, igniters, and tracer rounds etc. The improvement of the MTV performance in this manner is limited due to its smallest available Mg particle size powder. Nanometer scale Mg particle is not available in bulk quantities; recent technological advances have enabled the commercial production of nanometer scale of aluminum (Al) powder. Nano Al powder is produced by the wire process. The stoichiometry of MTV and ATV composition are too much similar i.e. 32.7% and 26.5% respectively. Although MTV composition has advantages over ATV composition such as higher heat of combustion, but the advantage of nano particle combustion, such as increased in reactivity, contribute to making aluminum valuable substitution for Mg in many applications. ATV composition has a slow energy release rate compared to other pyrotechnic compositions having the same molecular density. The polymer oxidizer (PTFE) transparent to the laser and the nano particles heated quickly to such a high temperature that produces rapid vapor expansion of Al generated a spherical shock front in the surrounding oxidizer. It was found that PTFE more rapidly decomposes under shock conditions as compared to more energetic material like nitrocellulose. A linear relationship is found b/w the diameter of shock induced chemistry volume and energy absorbed by the Al from the laser. It is also found that energy absorption increases with the increment of size of nano Al particle, and this is due to that larger particles have more total energy available. All these advantages motivated us to study the Al/Teflon mixtures by varying their composition and examining their reaction behavior.

Chapter 2: Experimental Work

2. This chapter deals with the instruments, methodology, characterization and synthesis of aluminum, Teflon and viton pyrotechnic composition.

2.1. Aims and Scheme of Work

This project aims the synthesis, characterization and combustion behavior of Al/Teflon mixtures using Viton as a binder. A simple shock gel process was followed for the synthesis of ATV composition. The advantage of this process is that ATV composition energetic material can be synthesized without producing large volume of hazardous waste. As produced ATV composition has excellent environment and thermal stability characteristics. It also has Good compatibility as compared with most materials used in pyrotechnic ordnance.

The goal of this work is also to extend previous studies analyzing the mechanisms associated with combustion of Al/Teflon mixtures through heating rates. Thermo gravimetric analyses (TGA) was used to study the heat flow and reaction behavior of Al=Teflon mixtures containing Al particles.

ATV is a fuel rich composition and it was found that when it ignites its temperature rise is too much rapid as compared to other compositions. Temperature rise of 1600 K occurs in a few moments while rise in temperature of 3600 K just in 7 µs. But maximum of its ingredients was observed at temperature of 1600 K. Its maximum ingredients include AlF, CF and CF_2 that lie in 1600 K region but decline thereafter in the favor of AlF_2 and C. [14]

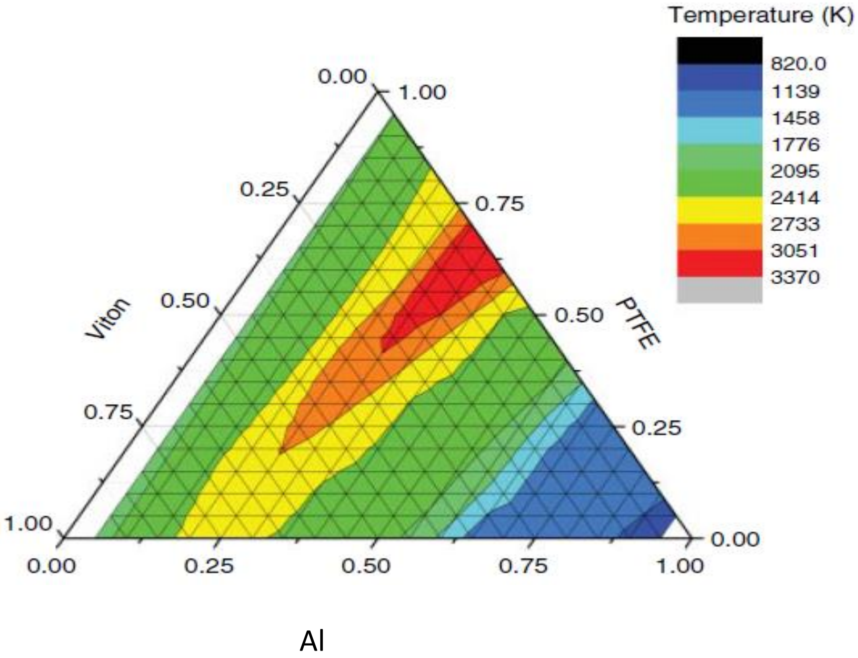


Fig2.1 Adiabatic combustion temperature of ATV at 0.1 MPa [15]

If we neglect the actual combustion kinetics the above picture shows good influence of atmospheric oxygen on exothermicity of the overall reaction. It is also noted that stoichiometric range of high combustion temperature with oxygen is too much broad than with fluorine.

2.2. Materials and Equipments

2.2.1. Sample Materials

All the chemicals used were of analytical grade and highest purity available including;

- Aluminum powder of grade A_16164_2I
- Teflon of particle size 35 μm
- Viton
- Acetone (AR)
- liquid CO_2

2.2.2. Instrumentation

Pilot plant and reactor system (Auto Clave) CH- 8610 Uster/Switzerland placed in SCME NUST was used in this work.

Fig 2.2 Auto Clave SCME NUST Islamabad

First of all we dissolve Viton in Acetone with a fix ratio i.e. (16:40) in order to form slurry in steel beaker and placed it for 24 hours in an inert environment. On the other hand we mix Aluminum and Teflon (PTFE) with different ratios in order to form three samples and the ratios of these three samples are (70:14),(54:30) and (30:54). At the end the slurry that is form in first step and the mixture which is formed in second step are mixed in Auto Clave at 3500 kPa pressure and 200^0C temperature for 4 to 5 hours with continuous supply of liquid CO_2 and stirring of 1500 rpm.

Fig 2.3 Mixing of Acetone & Viton in steel beaker in order to make slurry

2.2.3. Experimental Design

Different experiments were designed by varying composition of Aluminum and Teflon.

2.2.4. Effect of Aluminum Teflon ratio on synthesis product

- Effect of varying Al/T ratio on the synthesized ATV.
- Effect of varying Al/T ratio on combustion rate of ATV.

2.3. Methodology

2.3.1. Experiment 1: To synthesize ATV with ratio of 70:14:16

Dissolve Viton in Acetone with a fix ratio i.e. (16:40), in order to form slurry, in steel beaker and place it for 24 hours in an inert environment. On the other hand, mix Aluminum and Teflon (PTFE) with a fixed ratio (70:14). At the end the slurry that is form in first step and the mixture which is formed in second step were mixed in Auto Clave, adjusted at 3500 kPa pressure and 200^0C temperature for 4 to 5 hours with continuous supply of liquid CO_2 and stirring at 1500 rpm.

2.3.2. Experiment 2: To synthesize ATV with ratio of 54:30:16

Dissolve Viton in Acetone with a fix ratio i.e. (16:40), in order to form slurry, in steel beaker and place it for 24 hours in an inert environment. On the other hand, mix Aluminum and Teflon (PTFE) with a fixed ratio (54:30). At the end the slurry that is form in first step and the mixture which is formed in second step were mixed in Auto Clave, adjusted at 3500 kPa pressure and 200^0C temperature for 4 to 5 hours with continuous supply of liquid CO_2 and stirring at 1500 rpm.

2.3.3. Experiment 3: To synthesize ATV with ratio of 30:54:16

Dissolve Viton in Acetone with a fix ratio i.e. (16:40), in order to form slurry, in steel beaker and place it for 24 hours in an inert environment. On the other hand, mix Aluminum and Teflon (PTFE) with a fixed ratio (54:30). At the end the slurry that is form in first step and the mixture which is formed in second step were mixed in Auto

Clave, adjusted at 3500 kPa pressure and 200^{0}C temperature for 4 to 5 hours with continuous supply of liquid CO_2 and stirring at 1500 rpm.

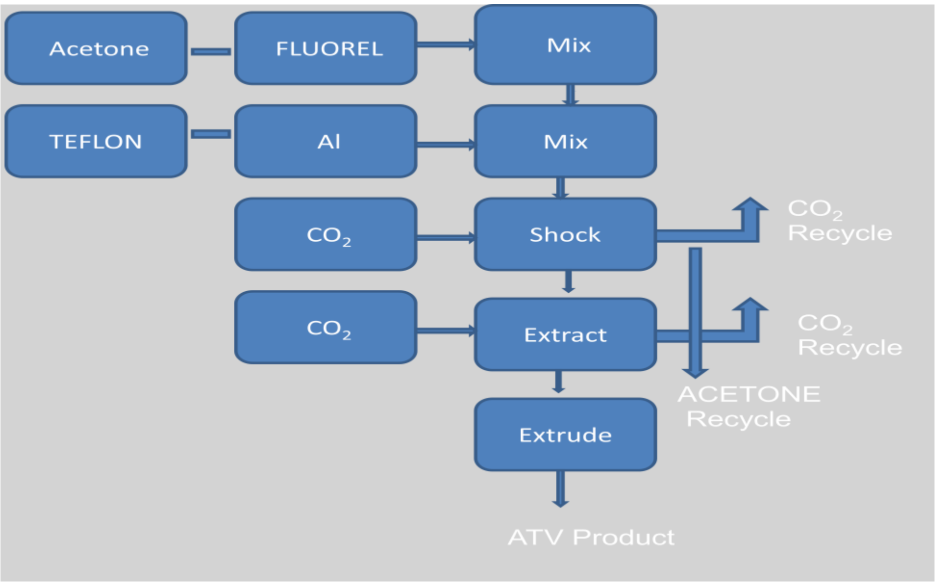


Fig 2.4 Block diagram for synthesis of ATV composition

2.4. Characterization Techniques

Number of techniques has been developed to characterize pyrotechnic materials. Some of these are quantitative and some are qualitative. Quantitative procedures produce a numerical measure of a property (in absolute or relative units), whereas qualitative experiments give a general overview of the property of interest without supplying a numerical value.

Following techniques were employed for characterization of samples prepared by set of experiments;

1- X-Ray Diffraction (XRD)

2- Scanning Electron Microscopy (SEM)

3- FTIR Spectroscopy (FTIR)

4- Thermo gravimetric Analysis (TGA/DTA)

2.4.1. X-Ray Diffraction

This method examines how X-rays are diffracted from atoms in a material. X-ray diffraction is used to determine structures of crystals, phase transitions and its indexing to check phase purity level. Since the wave length of X-rays (0.5-50Å) is similar to the distance between atoms in solid, so these rays are ideal to show structural details of crystals. Diffraction of X-rays occurs only when incident rays are scattered by atoms in a way that it interfere constructively and reinforces the waves according to Bragg's law.

$$2d\text{Sin}\theta = n\lambda$$

XRD analysis was performed by using STOE diffractometer at SCME NUST, using Cu Kα monochromatic radiation source and scans were recorded in the range of 20° to 70° with step size of 0.5 and stay time at each step of 5sec. Silicon was used to calibrate the instrument. XRD data was analyzed using software of same machine to get information about phase's present, crystallinity and grain sizes.

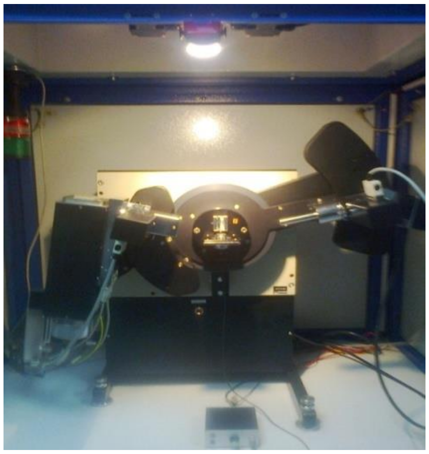


Fig 2.5 STOE diffractometer at SCME NUST

2.4.2. Scanning Electron Microscopy

Fig 2.6 Sputter coater at SCME NUST

SEM analysis was performed using JEOL Japan, JSM 6490A analytical scanning electron microscope, the resolution of this SEM can approach a few nm but its magnifications can easily adjusted from about 10-300,000 range. Topographical information can also provide composition of the surface being observed by energy dispersive spectroscopy (EDX) by using the fluorescence X-rays that is produced as a result of interaction of highly accelerated electrons with the material.

As the samples, under consideration, were compositions of Aluminum, Teflon and Viton, that are unable to conduct charge so much, striking by electron beam, and would have started deflecting further incoming electrons due to charge accumulation and resulting image is not clear but blurred. In order to overcome this problem all of the powder was initially coated with 200 Å thick layer of gold to make them conductive with the help of JFC 1500 ion sputtering device. Powder samples were initially ultra-sonicated for half an hour, and placed it over the sample stub of the machine and coated with gold. The samples were thermally etched at 800°C for grain boundary grooving to show its granular structure. SEM analysis was conducted to get information about particles size, shape surface morphology, packing and composition.

Fig 2.7 JEOL Japan, JSM 6490A Analytical Scanning Electron Microscope

2.4.3. FTIR Spectroscopy

FTIR spectroscopy gives information and measures the interaction of infra red radiations with the molecules of the material. For a material that gives response to IR radiations it must possess a permanent dipole and those lacking this property are transparent to IR spectroscopy. Since a polar bond can be thought of as oscillating or vibrating with a certain frequency. This is occurring in three dimensions so there should be numbers of vibrations that are possible for any given bond including bending, rocking and stretching movements. When the frequency of IR radiation matches with this frequency of vibration, then they reinforce the action and amplitude of vibration increases but the frequency with which the bond vibrates remains the same. Since there are many type of bonds which are present in any material so there will be number of chances of interaction at many frequencies giving a finger prints of that material specific to that only.

Fig 2.8 FTIR Instrument at SCME NUST

IR spectroscopy gives information about the functional groups and nature of bonds present in a material, therefore FTIR test was suggested. FTIR spectra were collected over the range of 450-4000 cm^{-1} (Perkin Elmer, US). Test samples prepared for FTIR were in pallet form. These pallets were made by mixing analytical grade KBr and synthesized powder of ATV in ratio 99:01 then pressing in hydraulic press at a pressure of 15 ton. Individual FTIR spectra of Al, Teflon and Viton were also obtained in order to check and compare with the synthesized ATV composition. The purity of material gives the functionalization of ATV composition.

2.4.4. Thermo Gravimetric Analysis (TGA)

In thermo gravimetric analysis (TGA) % weight loss of the specimen is calculated as a function of the temperature of that material. The sample is heated up to a certain limit with a predefined heating rate in oxidizing, inert and reactive atmosphere. The sample weight loss or percent weight loss is plotted against the temperature profile. Whenever material will react with environment or losses its weight then % weight loss change occurred. TGA measurements of the samples were carried out at heating rate of 10°C min_{-1} in nitrogen atmosphere (TGA Q50 V6.2 Build 187 in PMO) between 20°C and 700°C. TGA analysis was carried out to find the thermal losses, dehydroxylation, and decomposition of ATV.

Fig 2.9 TGA/DTA Instrument at SCME NUST

Chapter 3: Results & Discussion

3. In This chapter, results so obtained from characterization and analysis of various samples are discussed in detail.

3.1. RESULTS AND DISCUSSION

The results which are obtained from different tests of all the experiments conducted during this project are summarized below.

3.1.1. XRD Analysis

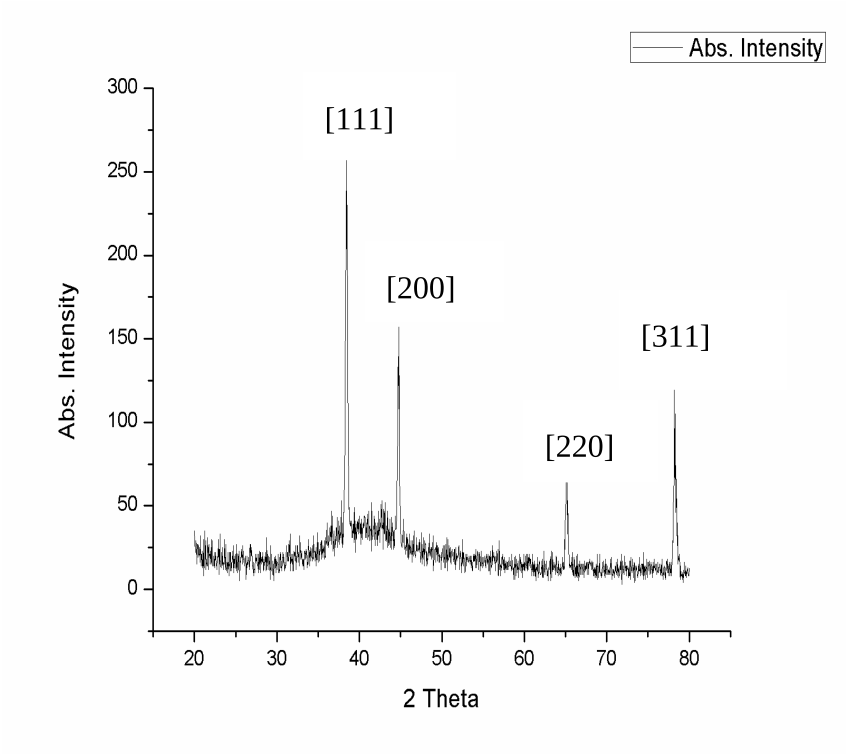


Fig. 3.1 (a): XRD pattern of the synthesized ATV sample # 1

This XRD pattern shows that as synthesized ATV is well crystalline and phase pure. Such crystallinity is very rare in as synthesized ATV composition. Peaks are broad indicating that crystallites produced are micro crystalline in nature.

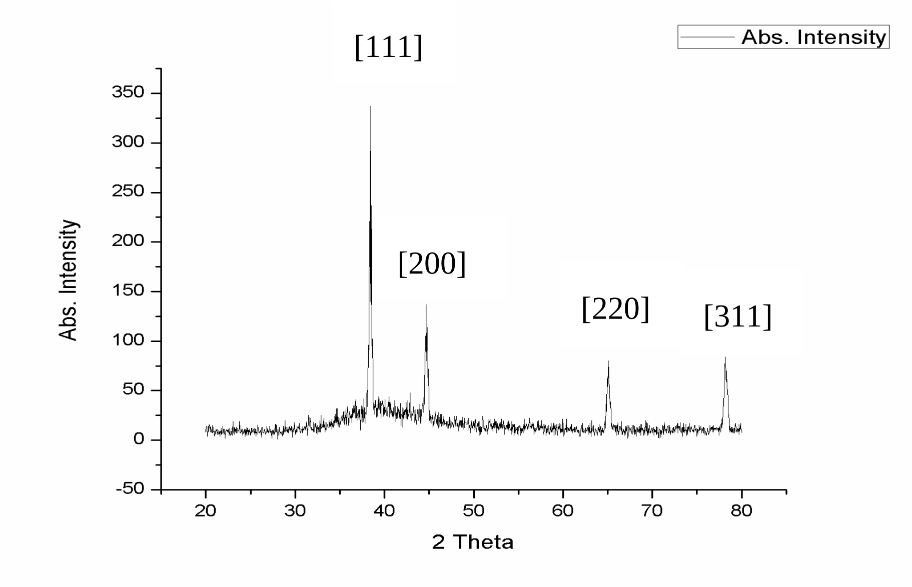


Fig 3.1 (b): XRD pattern of the synthesized ATV sample # 2

Crystalline phase clearly indicates the formation of in ATV composition.

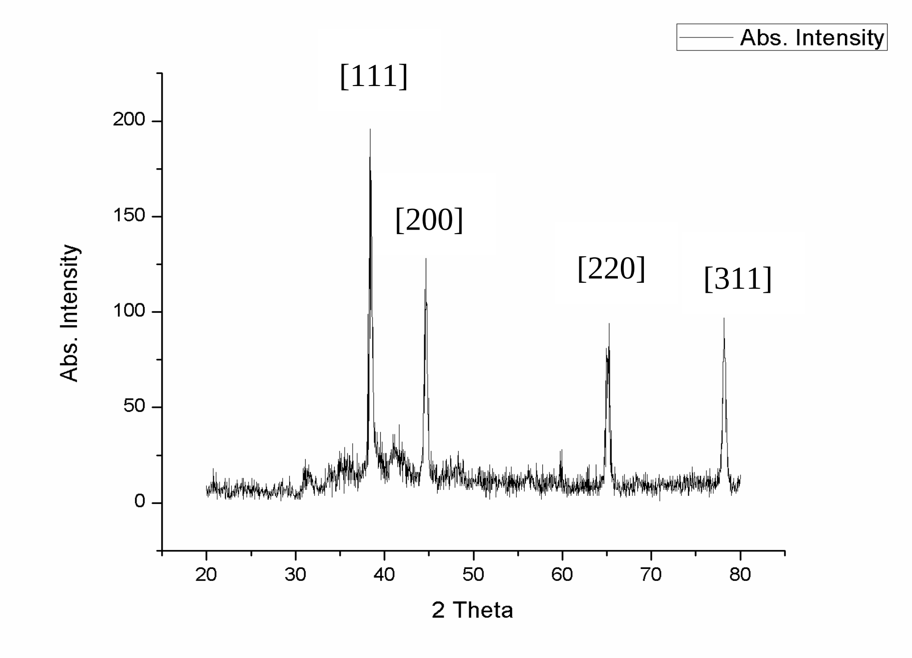


Fig 3.1 (c): XRD pattern of the synthesized ATV sample # 3

3.1.2. FTIR Spectroscopy

The FTIR spectrum of microwave synthesized ATV composition after drying at about 70°C is shown in fig 4.2(a,b,c) of different samples . The peaks at 3600 cm-1 is of hydroxyl group. While the peaks at 1600cm-1 and 1400cm-1 are of moisture and carboxylic respectively, the source of carboxylic group is due to adsorbed carbon dioxide and carbon mono oxide. FTIR spectrum also confirms the formation of ATV composition.

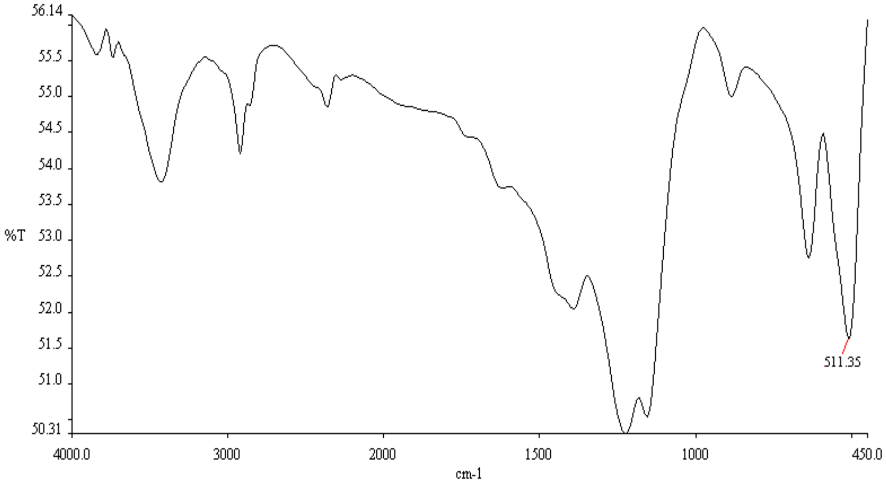


Fig 3.2 (a): FTIR spectrum of synthesized ATV Composition sample # 1

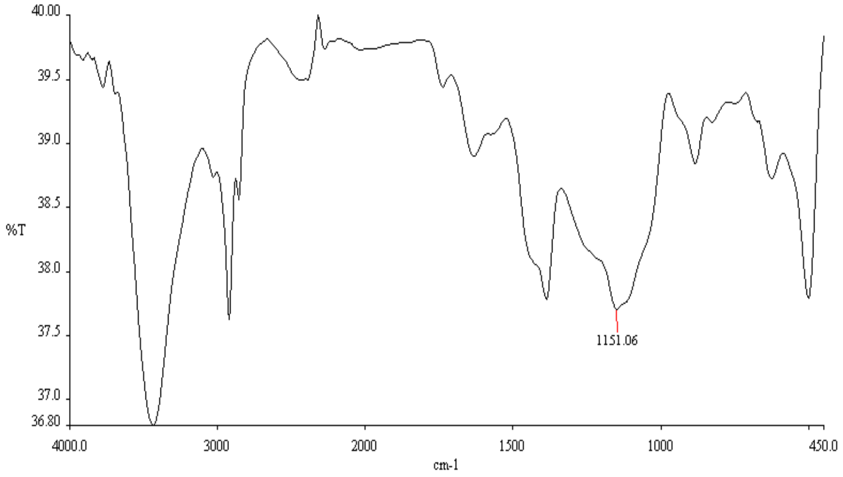


Fig 3.2(b): FTIR spectrum of synthesized ATV Composition sample # 2

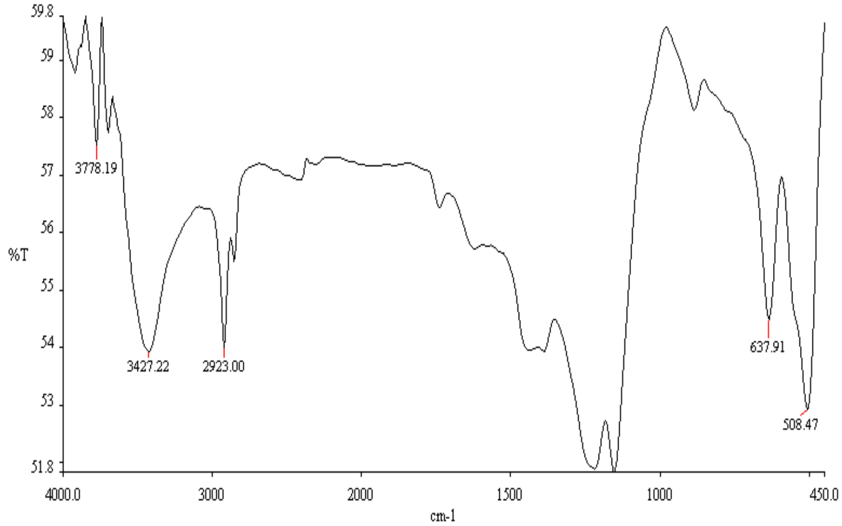


Fig 3.2(c): FTIR spectrum of synthesized ATV Composition sample # 3

3.1.3. SEM Analysis

Samples for SEM were prepared by dispersing 0.2g of synthesized ATV powder was stacked on stub and then sonicated for 30 minutes. A few particles are visible in image as layer was made very thin in order to properly disperse the nano particles. Particle morphology appears to be spherical with size ranging from 25.30nm to 80.62nm. Hence it is also possible to achieve nanosized particles in such short time along with crystallinity, phase purity and good yield.

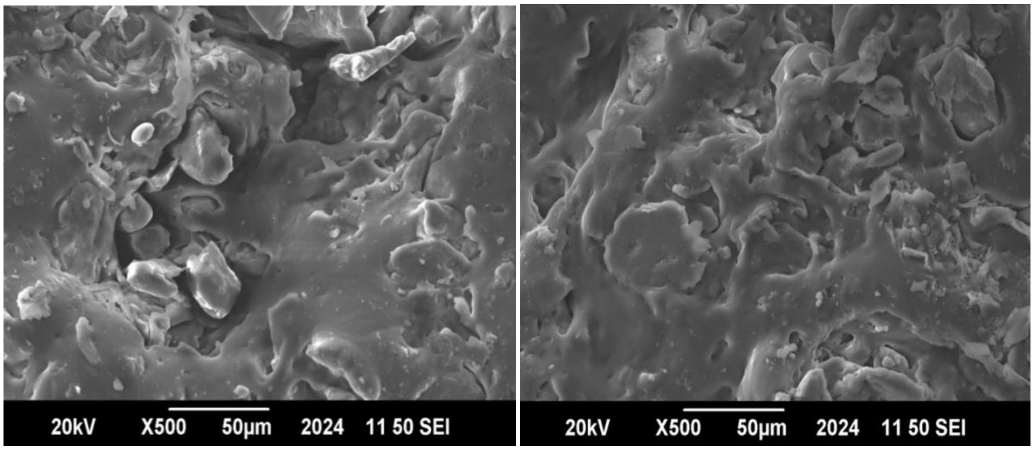


Fig 3.3: SEM of synthesized ATV Composition

3.1.4. Thermo Gravimetric Analysis

The results of the TG experiments for Al/Teflon compositions are displayed in Figures 3.4(a,b,c) respectively. Al figures show thermal gravimetric (TG) measurements on the left vertical axis which can be interpreted as the percent weight loss of the sample as a function of temperature. Weight can be lost when a solid reaction produces gaseous products that escape the sample crucible such that a weight change of the sample is detected and recorded.

The TGA curves of three samples after drying are shown in fig 4.4(a, b, c). With increasing temperature to 700°C number of knicks were observed with different % weight losses are observed due to continuous dehydroxylation of the ATV powder.

The heat flow as a function of temperature plot of the Al/Teflon mixture can be seen n Figure 3.4. These plot show definite exotherms around 400 and 550^{o}C. **Sarbak (1997**) [16] obtained similar results from thermal analysis and reported an exothermic phase transformation of Al-F species at 440^{o}C and the transformation from β-AlF_3 to a-AlF_3 at 560^{o}C. The 400^{o}C exotherm may be caused by the Al-F species reacting to form β-AlF_3. The 550^{o}C peak presumably corresponds to the transformation of β-AlF_3 to α-AlF_3, which was reported by **Reitsma and Boelhouwer** (1974) [17] to occur at 550^{o}C.

This change can be represented chemically by the following equation.

$$Al_2O_3 + 1.5\ (C_2F_4)\ n \quad \rightarrow\ 2AlF_3 + 3CO + 230\ kJmol^{-1}$$

$$AlF_3 \ \rightarrow\ \alpha\text{-}AlF_3 + heat$$

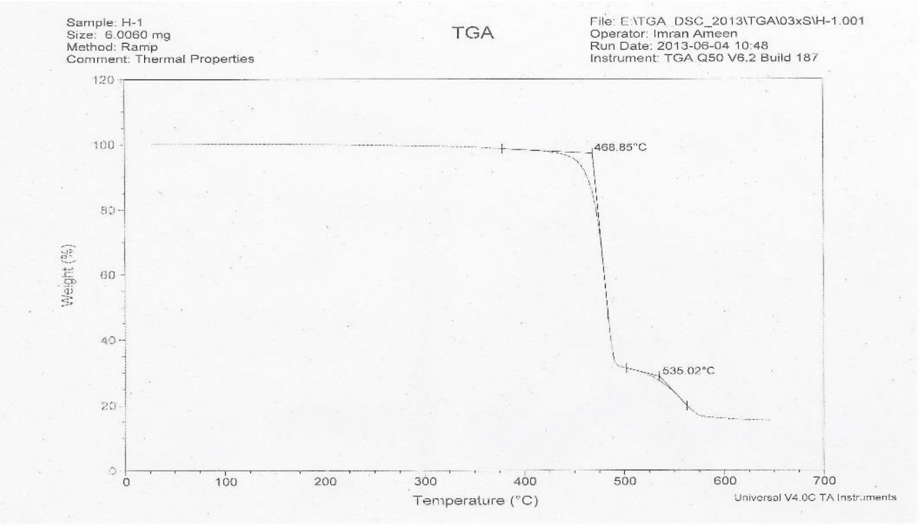


Fig 3.4: Thermo gravimetric losses in synthesized ATV Composition

3.2. Characterization of samples with different compositions:

3.2.1. Model 1: ATV Composition (70:14:16)

3.2.1.1. XRD Analysis

XRD analysis shows that ATV composition with a ratio of 70:14:16 is in pure crystalline form and such crystallinity is very rare in the formation of different pyrotechnic compositions.

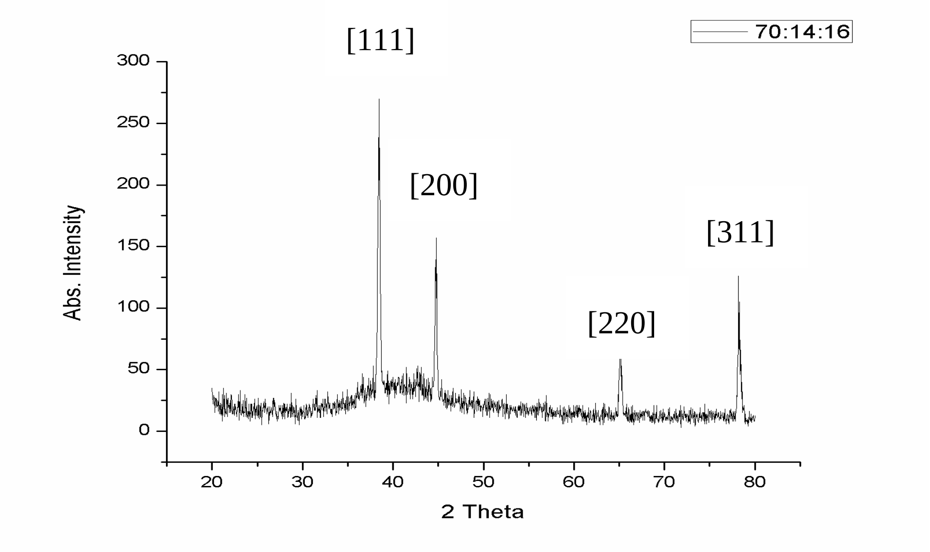


Fig. 3.5: XRD pattern of the synthesized ATV composition (70:14:16)

3.2.1.2. FTIR Analysis

The absorbance peaks at a wavelength of 886cm^{-1} corresponds to C–F bond, while peak at 1392.18 cm^{-1} is due to -CH_2-, and at 2364.44 cm^{-1} is due to C=C bond formation. In addition, peaks at 2922.48 cm^{-1} and 3500 cm^{-1} to 4000 cm^{-1} also appeared due to C-H bond and for moisture present. Hence formation of ATV was confirmed.

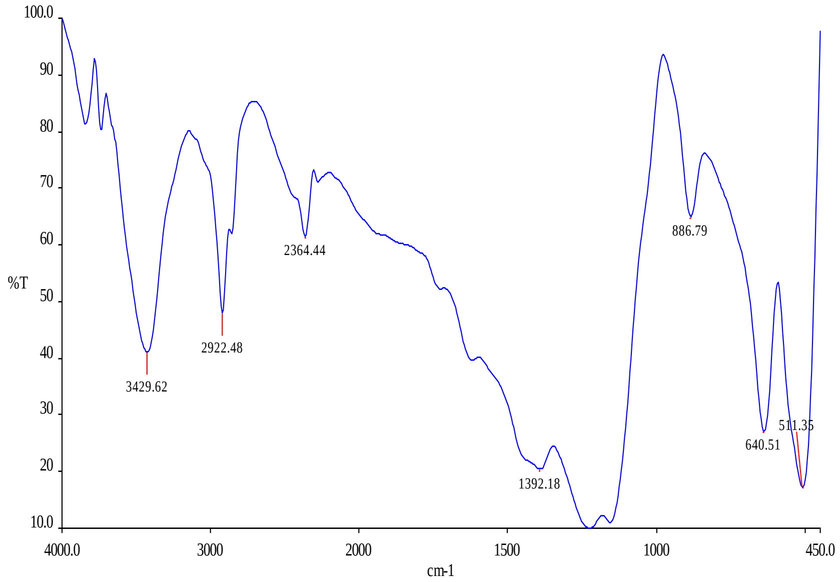


Fig 3.6: FTIR spectrum of synthesized ATV Composition (70:14:16)

3.2.1.3. SEM Analysis

SEM micrograph of ATV is shown in fig 3.7, Aluminum particles of nanosized can be viewed in image, which are embedded in Teflon. Fig 3.7 also gives the diametric dimension of ATV composition which is about 73.54nm. This low aspect ratio ATV is highly suited for composite applications.

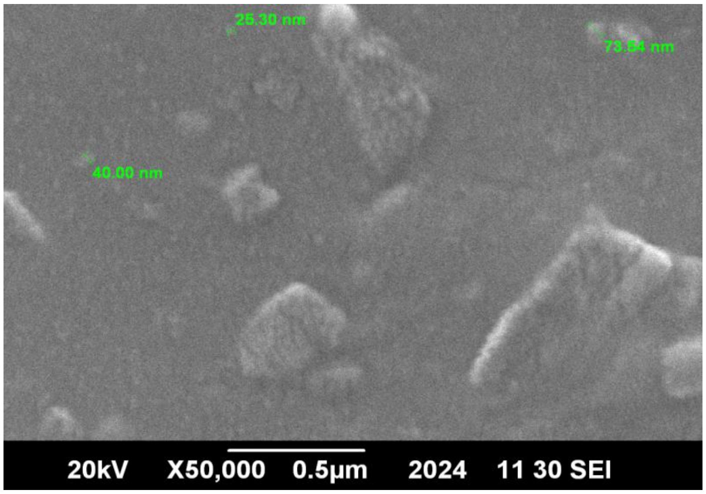


Fig 3.7: SEM of synthesized ATV Composition (70:14:16)

3.2.1.4. Thermo Gravimetric Analysis

The TGA curves of the sample after drying are shown in fig 3.4. With increasing temperature up to 468.85°C, composition is thermally stable but after 468.85°C to 535.02°C, around 67% weight loss occurred in composition. And after 535.02°C to 575°C about 15% weight losses with an increment of 39.98°C. From 575°C to onward the composition is thermally stable. This change can be represented chemically by the following equation.

$$Al_2O_3 + 1.5\,(C_2F_4)\,n \rightarrow 2AlF_3 + 3CO + 230\ kJmol^{-1}$$

$$AlF_3 \rightarrow \alpha\text{-}AlF_3 + heat$$

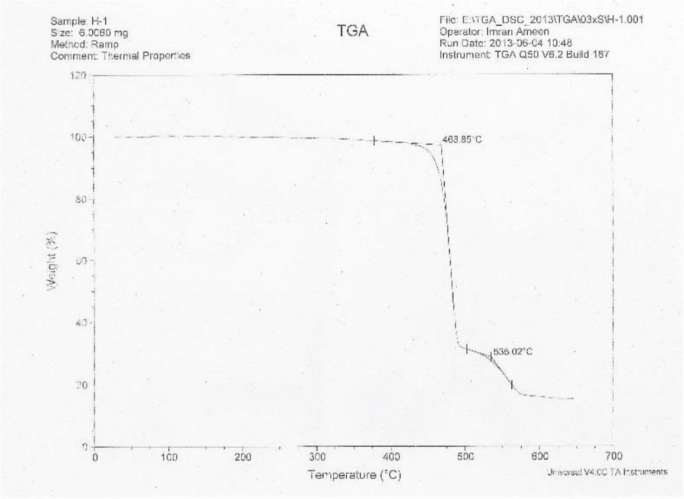


Fig 3.8: Thermo gravimetric losses in synthesized ATV Composition (70:14:16)

3.2.2. Model 2: ATV Composition (54:30:16)

3.2.2.1. XRD Analysis

XRD analysis shows that ATV composition with a ratio of 54:30:16 is in pure crystalline form and such crystallinity is very rare in the formation of different pyrotechnic compositions. If we observe our last result of XRD i.e. with the ratio of 70:14:16 is very similar to our result. This clearly indicates that change in ratio of Aluminum and Teflon does not effects overall composition.

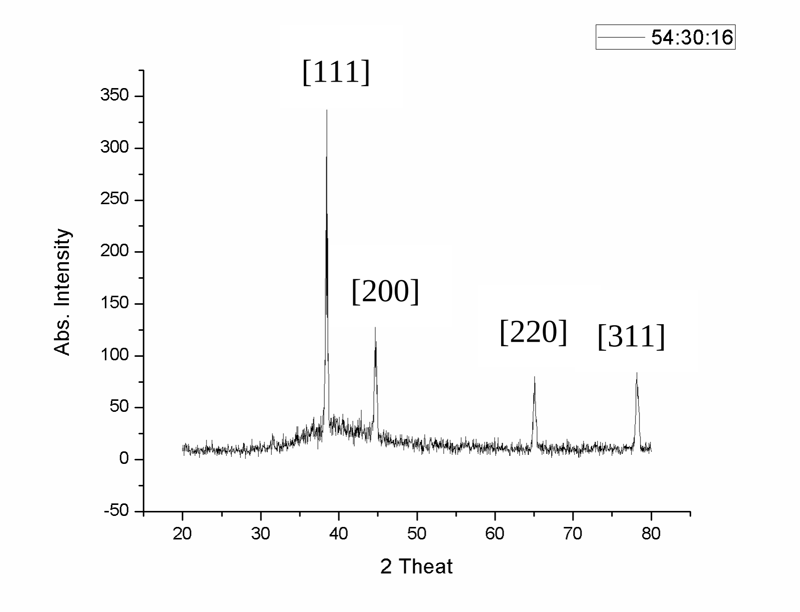


Fig 3.9: XRD pattern of the synthesized ATV Composition (54:30:16)

3.2.2.2. FTIR analysis

The absorbance peaks at 886.00 cm-1 are due to C–F bond, 1138.88 cm-1 is due to C-C, at 1408.17 cm-1 is -CH2- linkage and 2436.25 cm-1 due to C=C bond and at 3500 cm-1 to 4000 cm-1 due to moisture present. Thus the formation of ATV was confirmed.

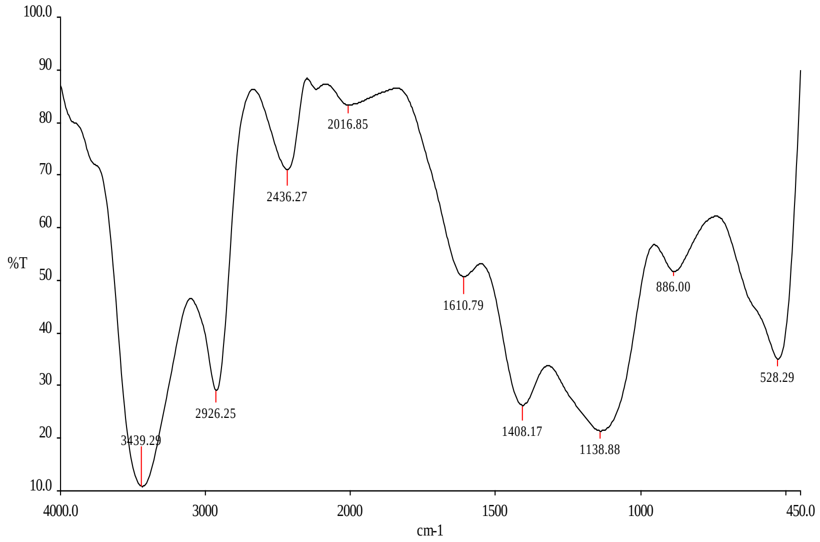


Fig 3.10: FTIR spectrum of synthesized ATV Composition (54:30:16)

3.2.2.3. SEM Analysis

SEM micrograph of ATV is shown in fig 3.11; there are Aluminum particles that are embedded in Teflon particles. Fig 3.11 also gives the diametric dimension of ATV composition which is about 80.62nm and this also shows that grain size increased. This low aspect ratio Aluminum and Teflon is highly suited for composite applications.

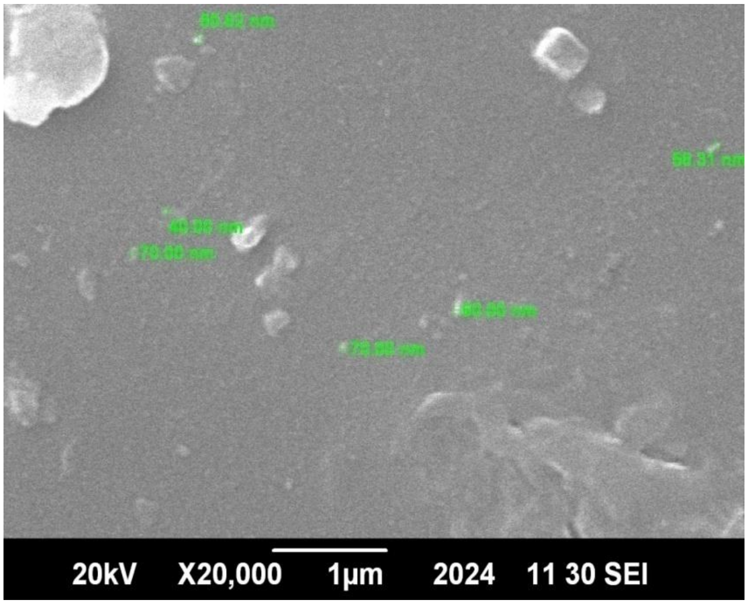


Fig 3.11: SEM of synthesized ATV Composition (54:30:16)

3.2.2.4. Thermo Gravimetric Analysis

The TGA curves of the sample after drying are shown in fig 3.12. With increasing temperature up to 469.36°C composition is thermally stable but after 469.36°C to 543.33°C round about 37% of its weight losses by the composition just an increment of 73.97°C. After 543.33°C to 597°C further 40% weight losses with an increment of 53.67°C. But 597°C the composition is thermally stable. This change can be represented chemically by the following equation.

$$Al_2O_3 + 1.5\,(C_2F_4)\,n \rightarrow 2AlF_3 + 3CO + 230\ kJmol^{-1}$$

$$AlF_3 \rightarrow \alpha\text{-}AlF_3 + heat$$

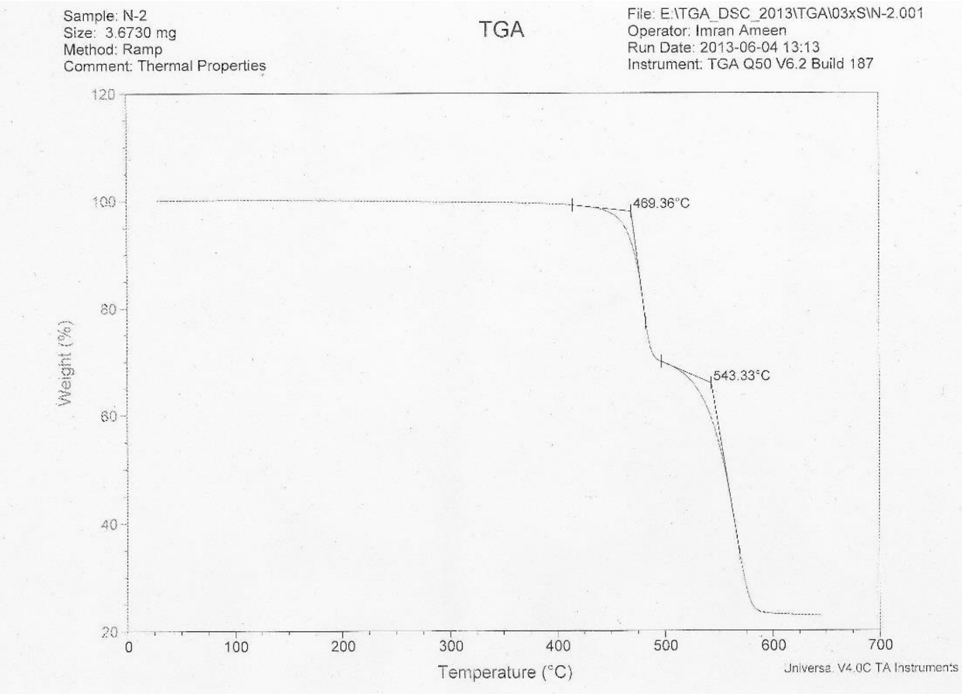


Fig 3.12: Thermo gravimetric losses in synthesized ATV Composition (54:30:16)

3.2.3. Model 3: ATV Composition (30:54:16)

3.2.3.1. XRD Analysis

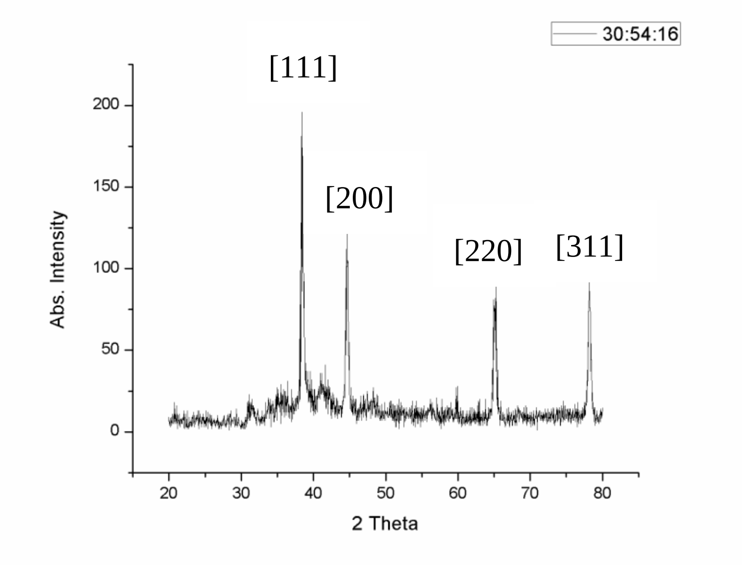


Fig 3.13: XRD pattern of the synthesized ATV Composition (30:54:16)

XRD analysis shows that ATV composition with a ratio of 30:54:16 is in pure crystalline form and such crystallinity is very rare in the formation of different pyrotechnic compositions. If we observe our last two results of XRD i.e. with the ratio of 70:14:16 and 54:30:16 then it is clear that results are too much similar. This clearly indicates that with the change of ratio of Aluminum and Teflon does not too much effect on composition

3.2.3.2. FTIR analysis

This spectra show that formation of ATV has occurred. The absorbance peaks at 529.60cm^{-1} are due to C–H bond, 123.83 cm^{-1} is due to C-H$_3$, at 1408.17 cm^{-1} is -CH2- linkage and 2448.28 cm^{-1} due to C=C bond and at 3500 cm^{-1} to 4000 cm^{-1} due to moisture present and this indicates that our sample is not completely dried.

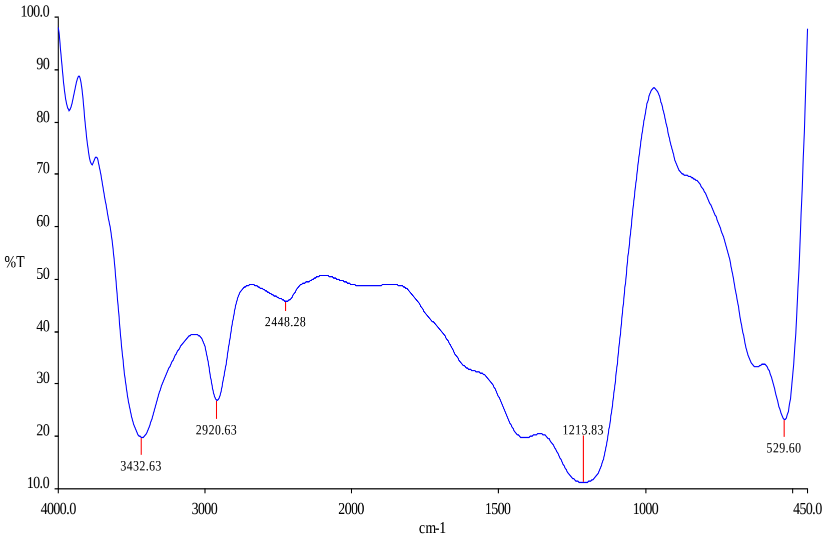


Fig 3.14: FTIR spectrum of synthesized ATV Composition (30:54:16)

3.2.3.3. SEM Analysis

Fig 3.15 also gives the polished diametric dimension of ATV composition which reveals rough surface morphology and this also shows that production of AlF_2 in gas phase which gives larger agglomerates. This low aspect ratio Aluminum and Teflon is highly suited for composite applications. [18]

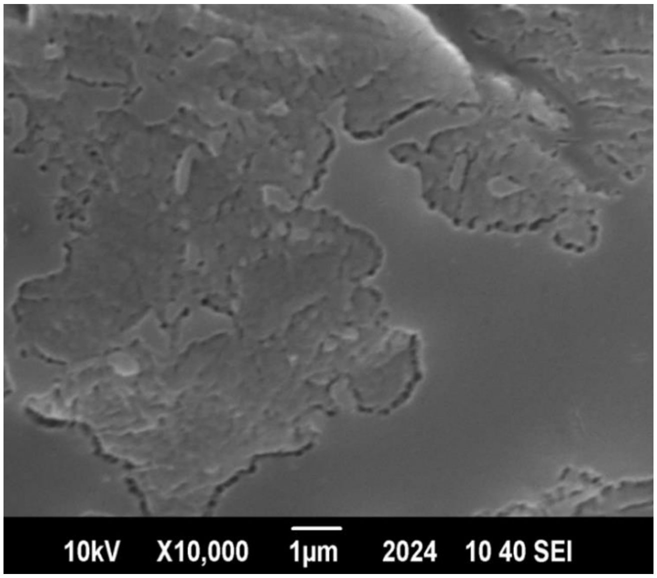


Fig 3.15: SEM of synthesized ATV Composition (30:54:16)

3.2.3.4. Thermo Gravimetric Analysis

$$Al_2O_3 + 1.5\ (C_2F_4)\ n \rightarrow 2AlF_3 + 3CO + 230\ kJmol^{-1}$$

$$AlF_3 \rightarrow \alpha\text{-}AlF_3 + heat$$

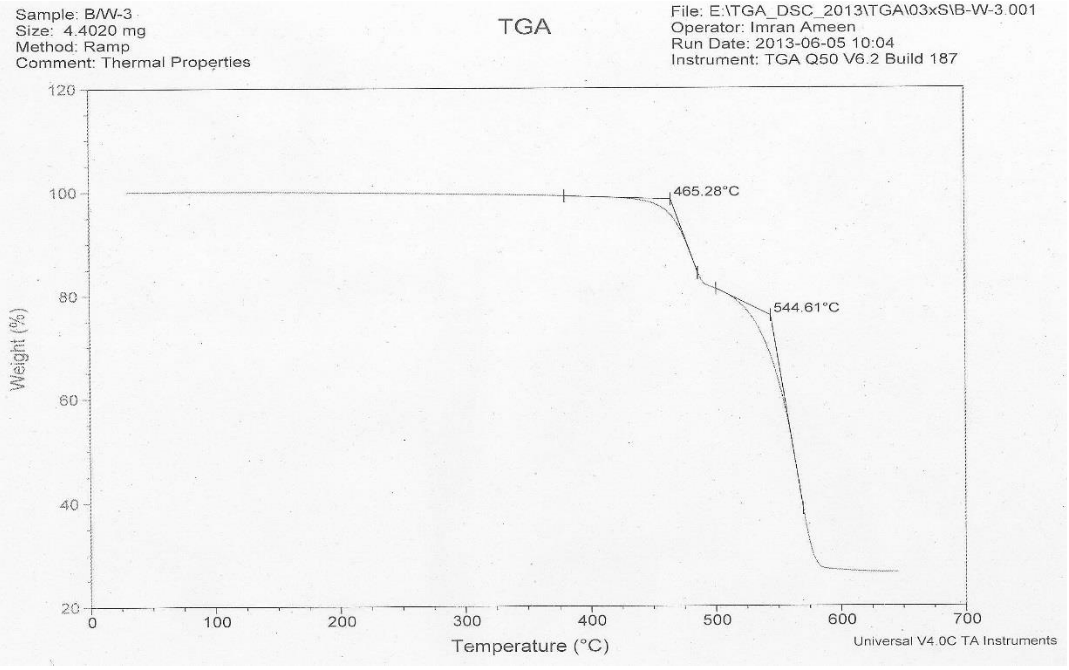


Fig 3.16: Thermo gravimetric losses in synthesized ATV Composition (30:54:16)

3.3. Discussions

3.3.1. Effect of varying ATV ratio on the composition

X-ray diffractograph show that there is no obvious effect of varying Al/PTFE ratio on the phase purity of ATV composition in any of the above process In addition, the effect on phase purity is not much that is countable, which is very clear from XRD patterns obtained from XRD Diffractometer. This comparison can be viewed from figure 3.17. This treatment was done by STOE Diffractometer at SCME NUST using Cu Kα monochromatic radiation at scan range of 20^0 to 80^0 with a step size of 0.5 and stay time for each step is 5, 10 seconds up to one minute.

Sample ID	***ATV Ratio***
Sample 1	70:14:16
Sample 2	54:30:16
Sample 3	30:54:16

Table 3.1 Different ratios of ATV

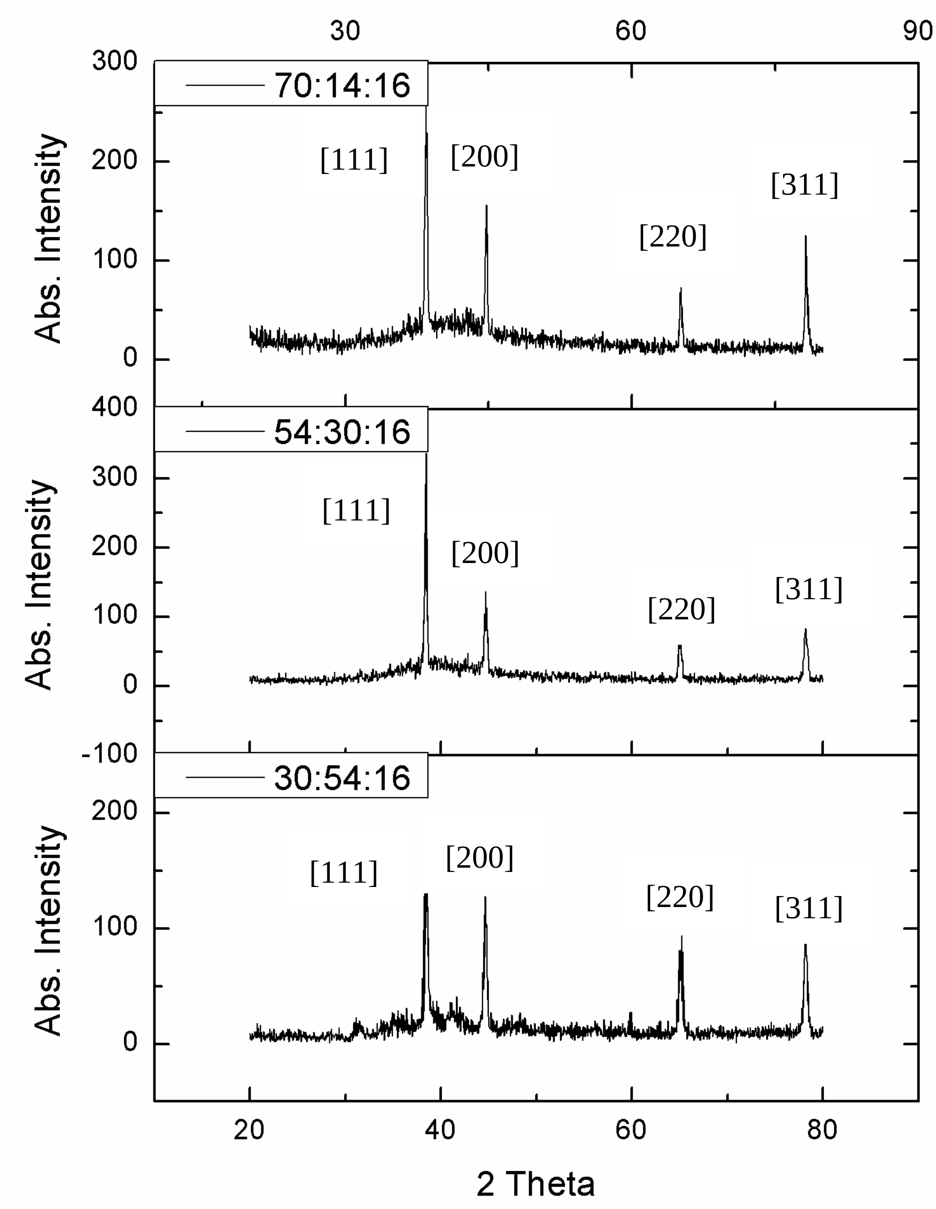
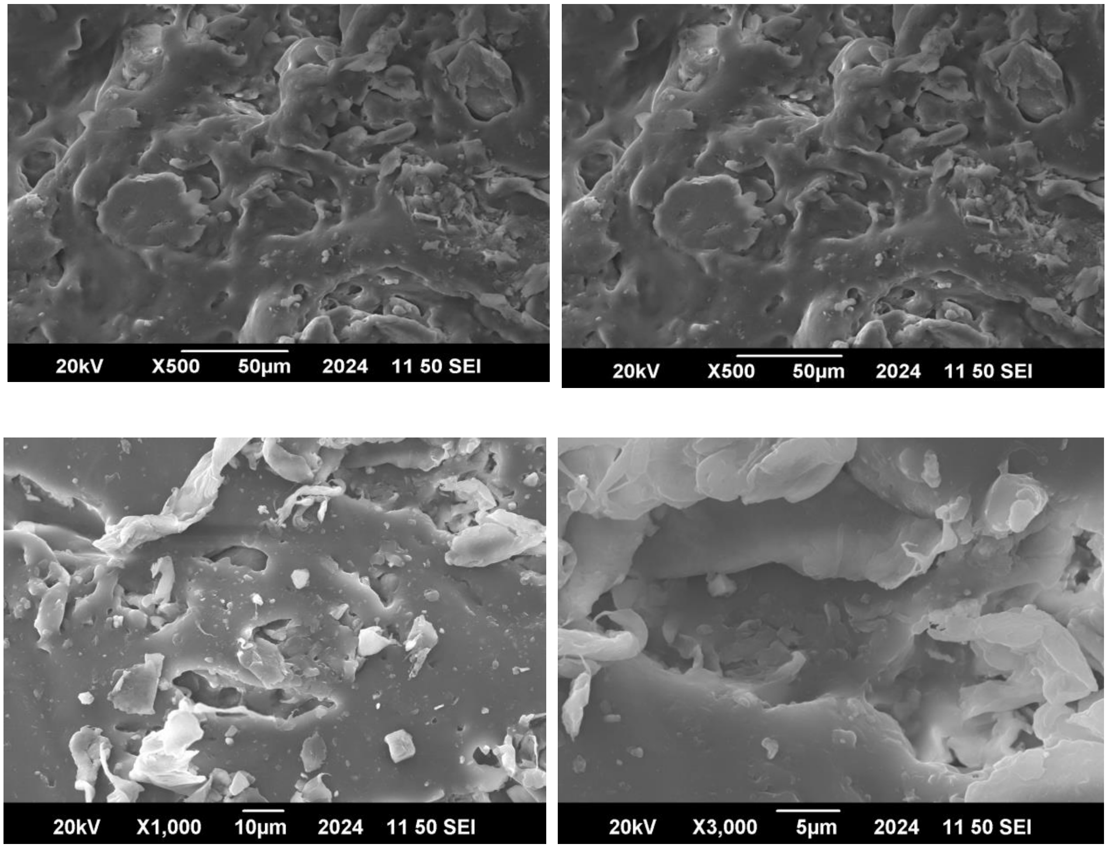
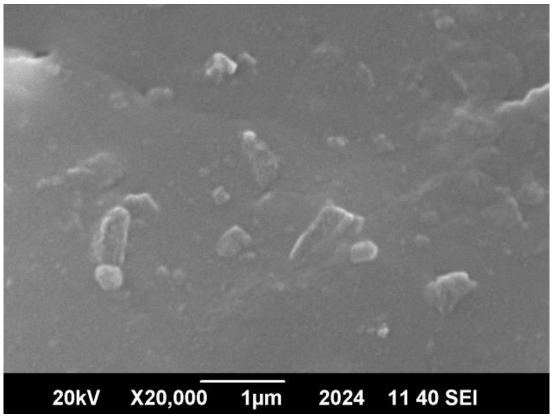


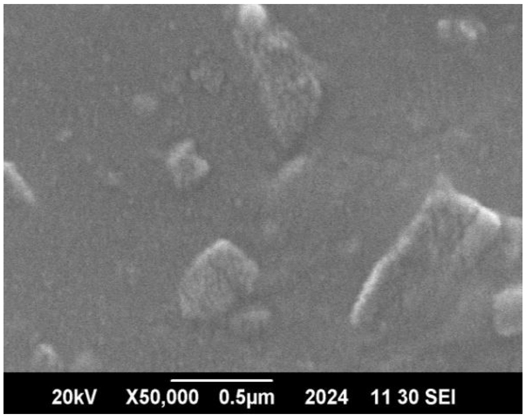
Fig 3.17: XRD patterns of ATV compositions prepared by varying Al, T and V ratio.

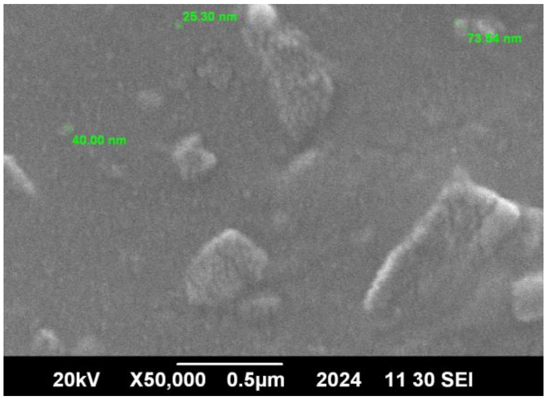
These samples, exposed to varying time for refluxing at 500W, were analyzed by X-ray Diffractometer (Cu-Kα).

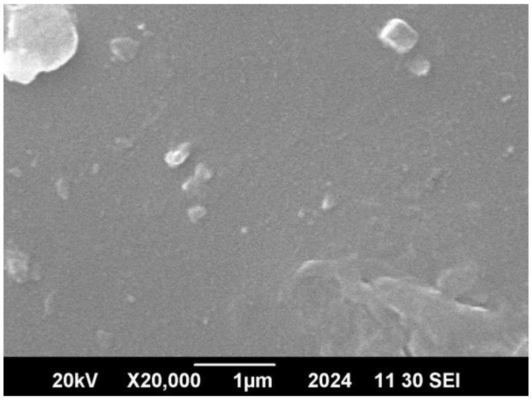
SEM micrographs of ATV are shown in fig 3.18, Aluminum particles can be viewed in image, which are embedded in Teflon particles. Fig 3.18 also gives the polished diametric dimension of ATV composition which reveals rough surface morphology and this also shows that production of AlF_2 is in gas phase which gives larger agglomerates. Grain size of ATV composition also increased from a certain lower value to a certain high value and this high aspect ratio of Aluminum and Teflon is highly suited for composite applications in ordnance field.

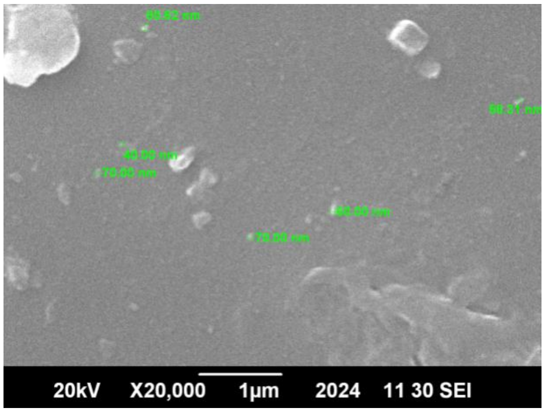


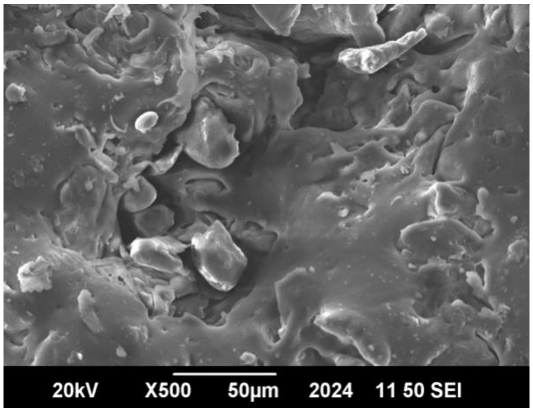

20kV X20,000 1µm 2024 11 40 SEI


20kV X50,000 0.5µm 2024 11 30 SEI


25.30 nm
73.04 nm
40.00 nm
20kV X50,000 0.5µm 2024 11 30 SEI


20kV X20,000 1µm 2024 11 30 SEI


88.82 nm
98.31 nm
40.00 nm
70.00 nm
80.00 nm
70.00 nm
20kV X20,000 1µm 2024 11 30 SEI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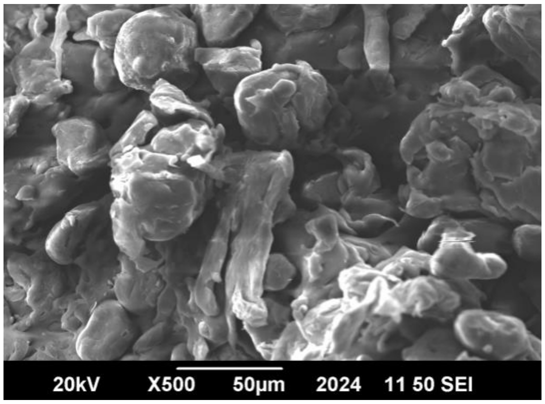
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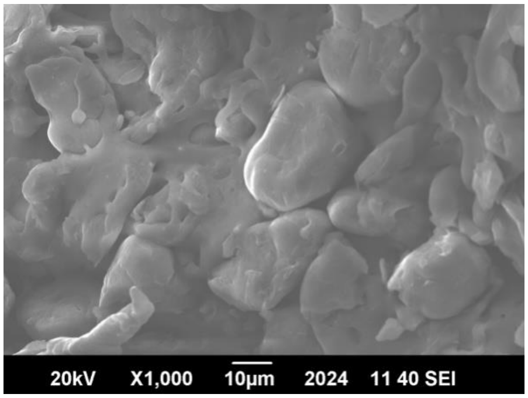
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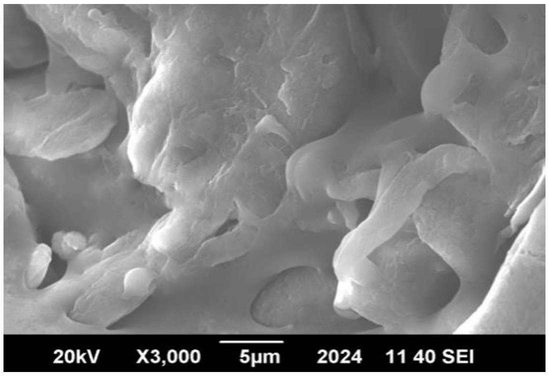
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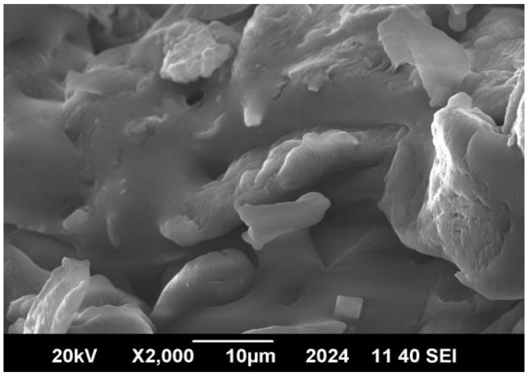
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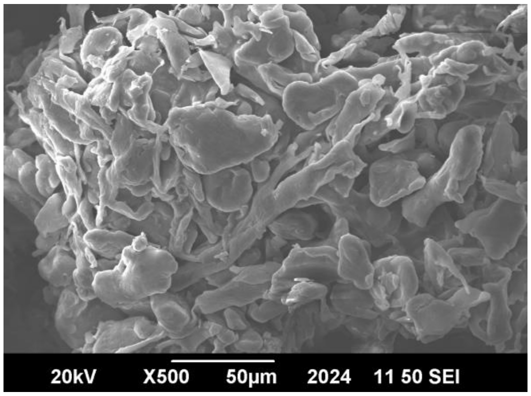
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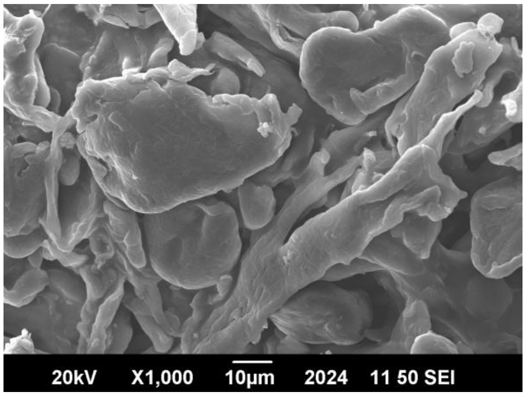
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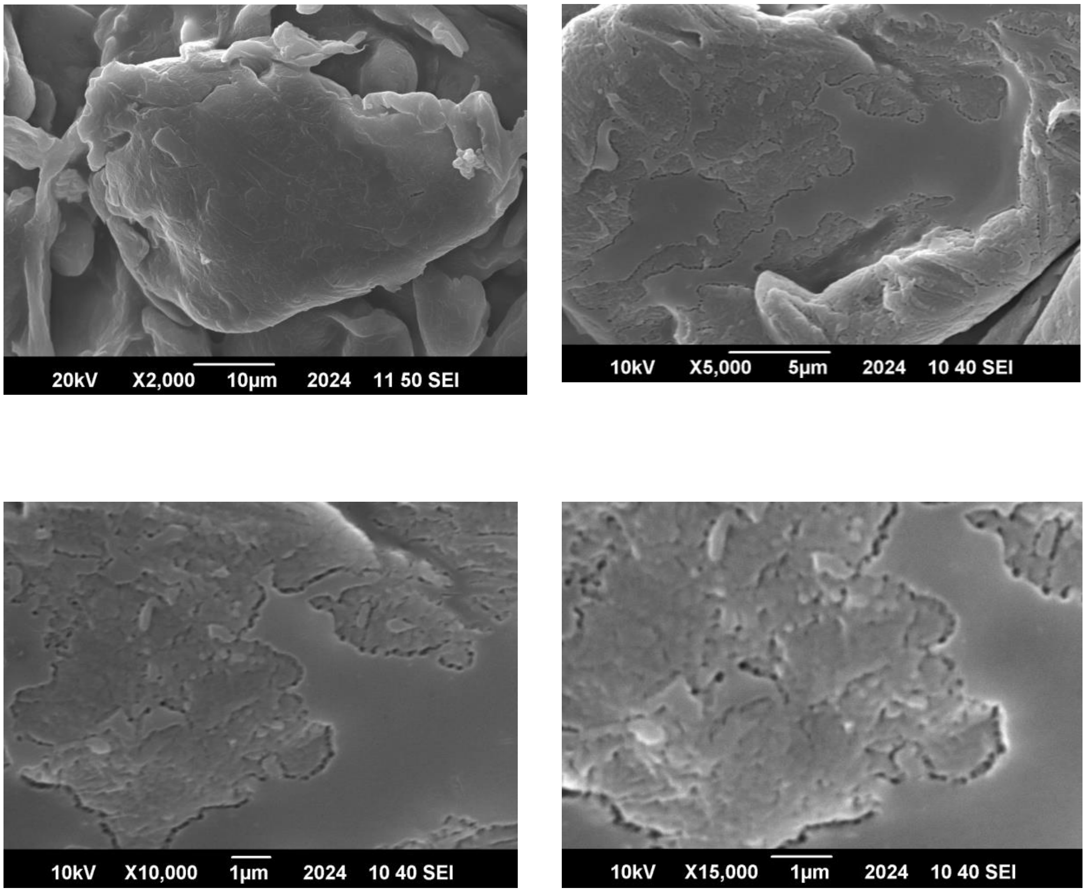

Fig 3.18: SEM images of all three samples of synthesized ATV

XRD plot showed that a well crystalline and phase pure ATV was formed with different ratios of Al and PTFE.

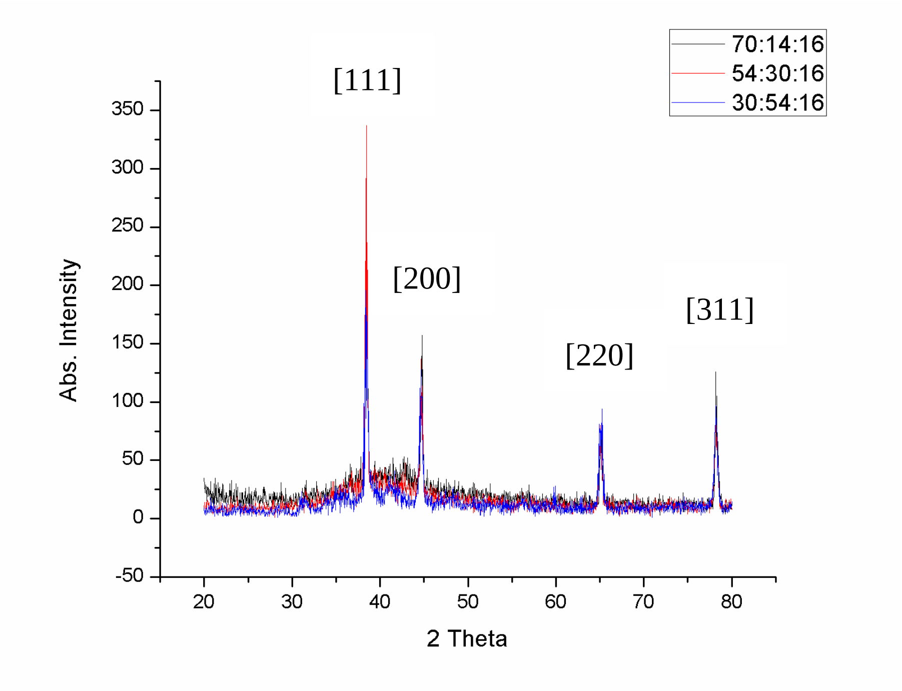

Fig 3.19: XRD pattern of all three samples of different ratios

If we further proceed in order to confirm functionalization of ATV that takes place in composition, then we can compare it with the individual components of composition i.e. Aluminum, Teflon & Viton and this comparison is shown in the figure 3.11 given below. The absorbance peaks at a wavelength of 886cm^{-1} corresponds to C–F bond, while peak at 1392.18 cm^{-1} is due to -CH$_2$-, and at 2364.44 cm^{-1} is due to C=C bond formation. In addition, peaks at 2922.48 cm^{-1} and 3500 cm^{-1} to 4000 cm^{-1} also appeared due to C-H bond and for moisture present. Hence formation of ATV was confirmed.

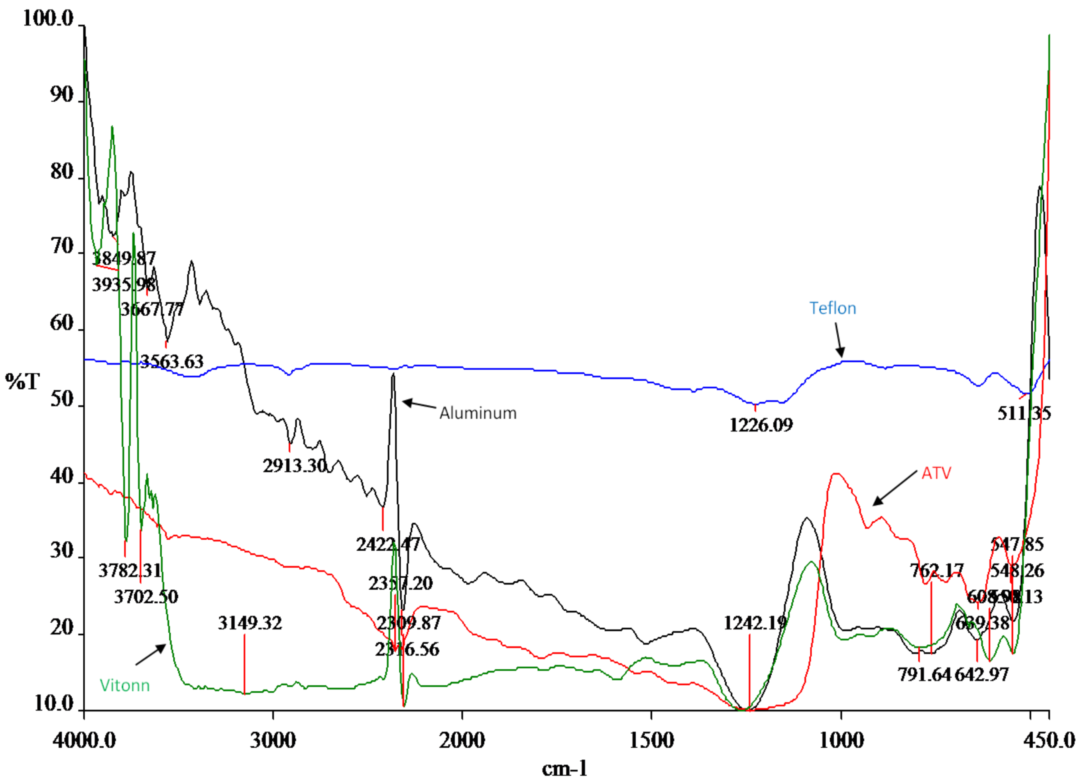

Fig 3.20: FTIR of Aluminium, Teflon & Viton and compare it with ATV composition of ratio 70:14:16

KEY WORDS

TNT (Trinitrotoluene), RDX (Hexogen), AN/FO (Ammonium nitrate / fuel oil), RP-1 (Rocket propellant-1), RFNA (Red fuming nitric acid), UDMH (Unsymmetrical dimethylhydrazine), MMH (Monomethylhydrazine), PVF (polyvinyl fluoride), PVDF (polyvinylidene fluoride), PTFE (polytetrafluoroethylene), PCTFE (polychlorotrifluoroethylene), PFPE (perfluoropolyether), CCl4 (Chemokine ligand-4), MTV (Magnesium, Teflon & Viton composition), ATV (Aluminum, Teflon & Viton composition), XRD (X-Ray Diffraction), SEM (Scanning Electron Microscopy), TGA/DTA (Thermo Gravimetric Analysis), FTIR (Fourier Transform and Infrared Spectroscopy)

Conclusions

Metal and fluorocarbon based pyrotechnic is very similar to black powder used in ancient times and have versatile applications in both strategical and tactical fields. A lot number of such compositions are used one of which is MTV composition. ATV provides a very good base in order to study the ATV pyrotechnic composition. Another reason of selecting of Aluminum is also the oxidizing property of Magnesium and as a result eases handling of Aluminum as compared to Magnesium. Through a number of tests composition is studied and it is found that composition gives too much similar results which are obtained from MTV pyrotechnic composition. SEM analysis shows nano morphology of ATV pyrotechnic composition and again much similar to that of MTV pyrotechnic composition. FTIR results clarify that functionalization of ATV takes place in composition. This study showed that nano-Al/Teflon mixtures exhibit an exothermic reaction prior to ignition that is caused by the fluorination of the Al particles. Chemical kinetics was discussed along with particle morphology to explain the thermal degradation process of the mixtures. These results are helpful in the fundamental understanding of Al particle size effects on the reactivity of Al/Teflon composites.

Future Work

- Since ATV gives us very similar results to that of MTV therefore it is useful as a pyrotechnic composition.

- Since Aluminum does not undergo vapour phase diffusion flame with PTFE so it is more reasonable to investigate the reaction of solid Aluminum particles with PTFE decomposition products.

- Since topology of Al-13 cluster is very similar to that of closed structure of bulk Aluminum so it is selected as a model compound to study the reaction b/w solid Aluminum particles and gaseous oxidizers like hydrogen chloride and chlorine.

Publications

Paper on synthesis & application of ATV pyrotechnic composition was presented in poster presentation and oral in conference on emerging materials & processing (C.E.M.P) in August 22nd to 24th, 2013 at SCME NUST Islamabad. Meanwhile a full length paper is proceeded for publication in NJES and IEEE.

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