Development and Characterization of Graphite Fluoride Dry Lubrication System by using Gamma Radiation

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ABSTRACT

Graphite is the most suitable material as dry lubricant. However, its application as a lubricant is limited with necessity of moisture vapors. Alternatively, polytetrafluoroethylene (PTFE) has an excellent lubrication property but does not have mechanical characteristics. The effect of gamma radiation doses on the lubrication behavior of graphite –PTFE (Inoflon A-408) system has been studied using (TR-TW-30L) tribotester with thrust washer attachment. Different compositions of graphite and PTFE were prepared and irradiated by gamma rays. Improved tribological properties of graphite –PTFE system showed by decrease in coefficient of friction and wear. SEM-EDX, XRD and contact angle analysis confirm fluorination of graphite by gamma radiation exposure leading to the development of novel dry lubrication system.

KEY WORDS: *Graphite, PTFE, dry lubricant, gamma radiation.*

1.0 INTRODUCTION

The technology of solid lubrication has advanced rapidly in the past four decades, primarily in response to needs of the aerospace and automobile industries. In most tribological applications, liquid or grease are used to combat and wear but service conditions become very severe. Solid lubricants may be the only choice for controlling friction and wear. Solid lubricants are used where the containment of liquids is a problem and when lubricants do not meet

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the advanced requirements. Under high vacuum, high temperatures, cryogenic temperatures, radiation, dust, clean environments or corrosive environments and combination therefore, solid lubrication may be the only feasible system. [1-7].

When present at a sliding interface, solid lubricant functions the same way as their liquid counter parts, specifically, they shear easily to provide low friction and to prevent wear damage between the sliding surfaces. Several inorganic materials (e.g. molybdenum disulphide, graphite, hexagonal boron nitride, boric acid) can provide excellent lubrication. Most of these solid lubricants owe their lubricity to a lamellar or layered crystal structure. A few others (e.g soft metals, PTFE, polyimide, certain oxides and rare-earth fluorides, diamond like carbons, fullerenes) can also provide lubrication although they do not have a layered crystal structure. In fact, diamond like carbon films are amorphous, but provide some of the lowest friction coefficients of all solid materials. The solid lubricants with a layered crystal structure are graphite, hexagonal boron nitride, boric acid, and transition -metal dichalcogenides MX_2 (where M is molybdenum, tungsten or niobium and X is sulphur, selenium or tellurium) [8-12].

Well -known solid lubricants (Graphite, HBN and transition dichalcogenides) owe their lubricity to a unique layered structure. The crystal structures of these solids are such that while the atoms lying on the same layer are closely packed and strongly bonded to each other, the layers themselves are relatively far apart and the force that bonds them are weak. When sliding present between sliding surfaces, these layers can align themselves parallel to the direction of relative motion and slide over one another with relative ease, thus providing low friction. In addition strong inter atomic bonding and packing in each other is thought to help reduce wear life, a favorable crystal mechanism is largely responsible for low friction and is essential for long wear life, a favorable structure in selfless is not sufficient for effective lubrication. The presence or absence of certain chemical adsorbents is also needed for providing easy shear in most solids, e.g. moisture or some other. [13-15].

Graphite is another classic example of lamellar solids that provides low friction and high wear resistance to sliding surfaces. Because of its good lubricity, abundance and low cost, it is used in any industrial applications. Like diamond, graphite is a polymorph of carbon. Both occur naturally and are recovered from deposits around the world; both can also be produced by synthetic means. Synthetic graphite is primarily produced by heating petroleum coke to about 2700^{0}C. Chemically, both graphite and diamond are same, but differ totally in their structures and properties. For example, graphite is perhaps one of the softest materials, while diamond is the hardest of all natural materials. Diamond has highest thermal conductivity, whereas graphite is relatively poor thermal conductor. However, graphite is a good electrical conductor, but diamond is an excellent electrical insulator. Graphite has a sheet -like crystal structure in which the entire carbon atoms lie in plane joins to three adjacent carbon atoms at a 1200^{0}C angle and at distance of 0.1415 nm. The distance between atomic layers is 0.335 nm at room temperature and the layers are held together by Van der Waals forces. In moist air, the friction coefficient of graphite varies from 0.07 to 0.15, depending on test conditions, sliding contact configuration, form of graphite used (powder, bulk, thin film, purity, crystallite orientation) and test machine. In dry air, inert atmospheres, or vacuum graphite's lubricity degrades rapidly, the friction coefficient increases to as high as 0.5 and it wears out quickly. Graphite is inexpensive and readily available in various forms. It is resistant to both acids and bases. In practice, graphite is used in powder, colloidal dispersion, solid and composite forms to combat friction and wear. It is key ingredient of electric brushes used in many motors. It can be dispersed in water, solvents, oils and greases to achieve better lubrication.[16-19].

Graphite is available in the form of amorphous, crystalline, natural and synthetic grades and needs moisture vapors for lubricity. Blended graphite and pure graphite dry film are widely used in many applications such as hot and cold forming, wire drawing and billet coatings, on high speed cutting tools, as a mold release for die casting process, plastic and rubber mold applications, cylinder head and exhaust bolts, armaments applications, automotive engine and many common industrial applications [20-21].

PTFE is widely used solid lubricant to provide dry sliding friction coefficient <0.2 on the variety of counter face types including stainless steel. PTFE is a popular polymer solid lubricant because of its resistance to chemical attack in wide variety of solvents and solutions, high melting, low coefficient of friction and biocompatibility. [22-26].

The present study [27--28] was undertaken by introducing different percentages (1%, 3%, 5%, 7% and 10%) of PTFE (Inoflon A-408) in graphite and exposed them by gamma irradiation to develop novel graphite fluoride dry lubrication system. PTFE Inoflon A-408 prepared by emulsion polymerization. TR-TW-30L tribotester was used for measuring the tribological properties of developed solid lubricant compositions. The purpose of the research was to develop novel graphite fluoride solid lubricant from graphite and PTFE (Inoflon A-408) using gamma radiation. In this paper, the lubrication behavior of graphite -PTFE (Inoflon A-408) compositions were studied by loading different levels of PTFE (Inoflon A-408) in graphite and exposing them to gamma radiation doses ranging from 50-100 kGy.

Preparation of graphite fluoride is very cumbersome rather than making fluorinated graphite as solid lubricant. The advantages of graphite fluoride compared to graphite are that it does not require adsorbed vapors or adjuvant impurities in order to lubricate. Fluorinated graphite improves the lubricity and durability and makes it less sensitive to variations in ambient humidity. Fluorinated graphite is capable of providing friction coefficients of 0.1 or less up to about 480 ^{0}C in open air compared to those of others under the same test conditions.

2.0 EXPERIMENTAL PROCEDURES

2.1 Materials used

PTFE (Inoflon A-408) prepared from emulsion polymerization of commercial grade was used of Gujarat Fluoro Chemicals Ltd. Natural graphite of industrial grade was procured from M/s Starke & Co; Pvt. Ltd. The materials were used as such, without any treatment or purification. The properties of graphite, PTFE (Inoflon

A-408) are shown in Table 1. Number average molecular weight of PTFE (Inoflon A-408) is 2.7 x 10^4. Copper plates with purity of 99.99% were used for the experiments.

TABLE 1: Characteristics of graphite and PTFE

Material	Particle Size (μ)	Specific gravity (g/cm³)	M.P (ºC)
Graphite	90	2.25	3500
PTFE (Inoflon A-408)	3-4	2.15	331

2.2 Methods

2.2.1 Particle size analysis:

The particle size analyser (Zetatrac, Microtrac Inc.) wherein diffraction patterns of laser beam is used to measure geometrical dimensions of a particle when is passed through any object ranging from nanometers to millimeters in size. Particle sizes of graphite and PTFE (Inoflon A-408) were measured by particle size analyzer. Particle size reduction of ball milled graphite was also studied by the particle size analyzer and the results are shown in Table 2.

TABLE 2: Particle size of graphite after ball milling

S.No	Time (hrs) ball milling	Particle size at D50
1	0	87.45
2	4	72.54
3	8	54.33
5	12	50.82

2.2.2 Molecular weight of PTFE

Heat of crystallization of a polymer is closely related to its molecular weight. It can be obtained from DSC cooling curve and applying the following equation.

$$M_n = 2.1 \times 10^{10} \times \Delta H_c^{-5.16}$$

Where M_n is the number average molecular weight and Delta H_c is heat of crystallization in Cal/g.

The crystallization peak is found to be remarkably affected by the polymer molecular weight. The larger the molecular weight, the smaller is the crystallization peak. This is explained by considering that the longer polymer chain has a greater difficulty in the regular rearrangement or orientation during crystallization because of smaller mobility and greater intra and intermolecular entanglement.

2.2.3 Preparation of Lubricant Compositions

Different percentages of PTFE (1%, 3%, 5%, 7%, 10%) powder were introduced into graphite and ball milled to get homogeneous mixtures. These mixtures were irradiated by gamma radiation in air at ambient temperature at different gamma radiation dose. Irradiated powder sample was put on the stationary bottom square plate of copper. Specific load is applied along axial direction to make proper contact between moving top and bottom plate.

2.2.4 Gamma irradiation

The graphite compositions with PTFE (Inoflon A-408) (1%, 3%, 5%, 7% and 10%) were irradiated in air at ambient temperature with absorbed doses 50 and 100 kGy. The dose rate of 2.85kGy/h was applied using a ^{60}Co gamma radiation source available at Shriram Applied Radiation centre, Delhi which is a wet storage, Type - IV, Stationary gamma irradiator. Dosimetric method used is ceric -cerous dosimeters with potentiometric technique (supplied by Board of radiation & Isotope technology, Govt. of India) for the determination of absorbed radiation dose at each location.

2.2.5 Tribological analysis

Experiments were carried out using a tribotester (TR-TW-30L) which equipped thrust washer attachment within plane contact rubbing pair. The annular top sample with an outside diameter of 18 mm and an inside diameter of 16 mm was made of EN-31 stainless steel with hardness 58-62HRC. There were four notches along the circumferential direction in order to facilitate powder entering the tribopair. The bottom plate was made of copper with dimensions of 32 mm x 32 mm x 3 mm.

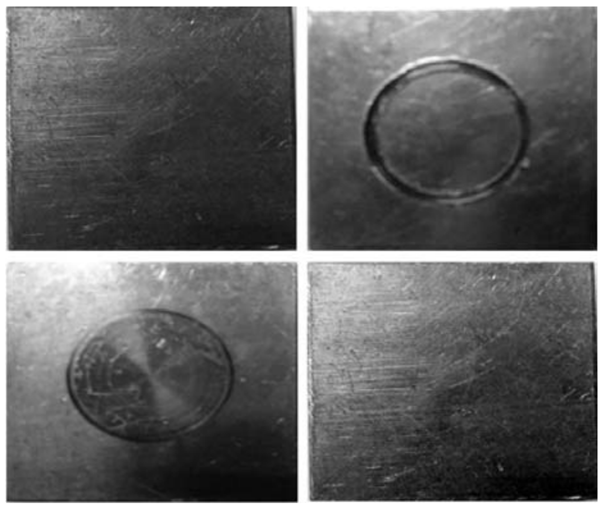

Fig. 1. a) Copper plate, b) Copper plate after dry frictional test, c) Copper plate after graphite test and (d) Copper plate after PTFE (Inoflon A-408)

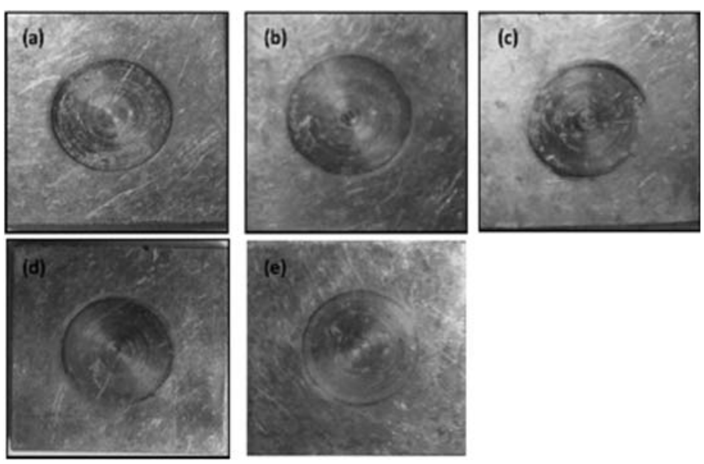

Fig. 2: Copper plate after wear test with a) graphite –PTFE (Inoflon A-408) (1%), b) graphite –PTFE (Inoflon A-408) (3%), c) graphite –PTFE (Inoflon A-408) (5%), d) graphite –PTFE (Inoflon A-408) (7%) and e) graphite –PTFE (Inoflon A-408) (10%) as a lubricant

During the experiment, top plate was rotatory and the bottom sample was fixed. Because the bottom surface is stationary and top sample was rotatory, the powder is in frictional interface. The specific load is applied along axial direction to make a proper contact between top and bottom plate. The tribological properties of dry lubricants were measured in real time by electronic measurement. Frictional torque, wear loss and coefficient of friction were measured using specifically designed tribometer of above said configuration. The surface of bottom samples was recorded and depicted in (Fig. 1, 2).

2.2.6 X-ray Diffraction (XRD) Analysis

The crystallinity analysis of graphite, PTFE (Inoflon A-408) and graphite –PTFE (Inoflon A-408) lubrication system were done using a Philips Xpert XRD System at a voltage between 5-80V, 30mA current. Spectra were recorded in the range of Bragg's angle $2\theta = 5^0 – 80^0$ with scanning step size 0.03^0 and time per step 1s.

2.2.7 Scanning Electron Microscopic (SEM) analysis

Morphology studies were performed on graphite, PTFE (Inoflon A-408) and graphite–PTFE (Inoflon A-408) lubrication system using SEM, S-3700N of HITACHI for imaging. The samples were mounted on aluminium stub with the help of double-sided adhesive carbon tapes (Agar Scientific, UK). The mounted samples were then exposed to ~20 nm gold coating at 20mA for 165s by a Sputter coater SC7620 (Quorum Technologies Ltd., UK) to make them electrically conductive. The elemental analysis of graphite and modified graphite -PTFE was measured by thermo Fisher Scientific energy dispersive X-Ray spectroscopic (EDAX) analyzer with a spectrum resolving power less than 132eV and accelerating voltage 15.0 kV.

2.2.8 Contact Angle analysis:

The sessile drop technique is a method used for the characterization of contact angle of materials. The main premise of the method is that by placing a droplet of liquid with a known surface energy, the shape of the drop, specifically the contact angle. Contact angle analysis of developed lubrication system was performed on instrument Kruss drop shape analyzer DSA 100 S.

2.2.9 Compressive strength:

A solid lubricant possessing high compression strength is capable to withstand high loads sufficient direct contact between the resulting surfaces. Compressive strength test was done using UTM (Model No. 5982), Star Testing System, Mumbai with maximum capacity of 100 kN using standard test method for compressive strength of carbon and graphite as per specification [29].

2.2.10 Thermal Stability:

The thermal stability of solid lubricant samples was done by using thermogravimetric analyzer (Model No. STA 449F3), Netzsch, Germany ranging from 25^0C to 1200^0C at of ramp 10^0C.

2.2.11 Fourier Transform Infrared Spectroscopy (FT- IR Results):

FT- IR of developed graphite fluoride dry lubricant was performed using FTIR instrument (Model Schimadzu –IR Affinity 1S) with KBr powder.

3.0 RESULTS AND DISCUSSIONS:

3.1 Lubrication studies under dry contact and with graphite and PTFE (Inoflon A-408)

The experiments were carried out under variable load with metal to metal, graphite, and PTFE (Inoflon A-408). The tribometer cell was loaded with powder lubricant so that powder could dynamically enter the frictional clearance between annular top sample and bottom sample during tests. The experimental load was varied from 6MPa to 18MPa and speed of the motor was maintained as 0.4m/s. The powder entry in frictional clearances is confirmed as the values observed for metal to metal friction is higher than PTFE (Inoflon A-408) and graphite. The lowering in coefficient of friction, PTFE (Inoflon A-408) and graphite powder is mainly due to the entry of dry lubricant powders into the moving state of tribopair. A decrease in coefficient of friction was observed upto 12MPa load. There onwards, not much affect was observed in values of coefficient of friction (Fig. 3).

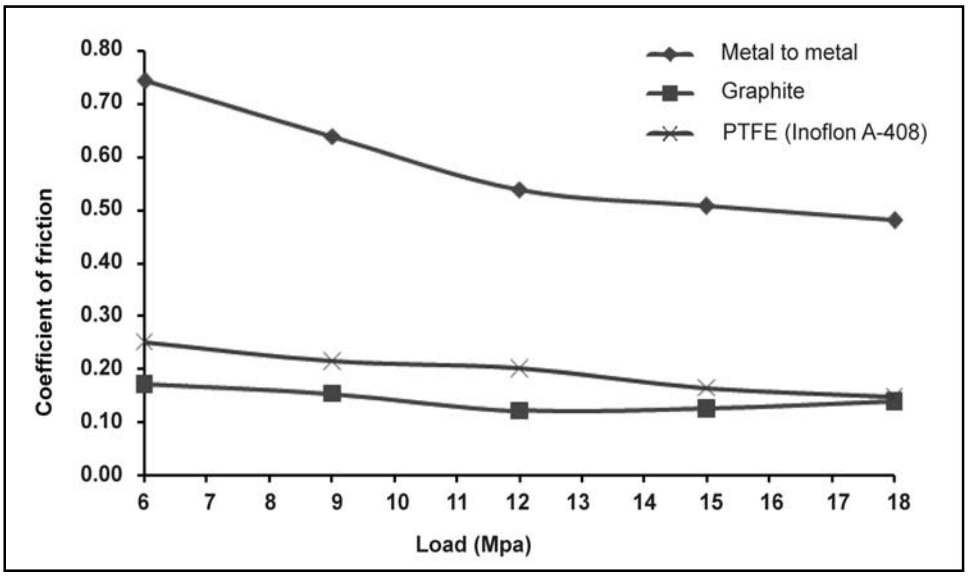

Fig. 3: Coefficient of friction of dry condition, graphite and PTFE (Inoflon A-408)

TABLE 3: Wear loss under Metal to Metal, graphite, and PTFE (Inoflon A-408)

S. No.	Sample	Wt of Cu- plate before test, g (W_1)	Wt of Cu-plate after test, g (W_2)	Wt Loss (W_1-W_2)	Wear (%)
1	Metal to Metal	26.9063	26.8730	0.0333	0.1237
2	Graphite	26. 9431	26.9421	0.0010	0.0037
3	PTFE (Inoflon A-408)	27.0604	27.0604	0.000	0.0000

Thus, further studies were carried out using 12MPa load. The lower frictional torque as indicated with graphite and PTFE powder clearly showed lubrication behavior of dry powders used (Fig. 4). Copper plates get affected in case of metal to metal and graphite, showed considerable wear loss, whereas copper plate remain unaffected with PTFE powder (Inoflon A-408) (Fig. 3) and (Table 3). The results showed that wear loss under metal to metal was 0.1237 % and with graphite was 0.0037%, whereas no wear loss was observed with PTFE powder (Inoflon A-408) (Table 3). This indicates that PTFE powder has superior lubrication properties than graphite upto 12 MPa load.

3.2 Lubrication studies of Irradiated Graphite and PTFE compositions

In five compositions were prepared by loading PTFE (Inoflon A-408) powder (1%, 3%, 5%, 7% and 10%) in micronized graphite (50 micron). The radiation exposure doses for these experiments were used as 50kGy and 100kGy. These compositions were studied before and after irradiation at different gamma radiation doses. The results of coefficient of friction of these compositions are presented in Fig. 5.

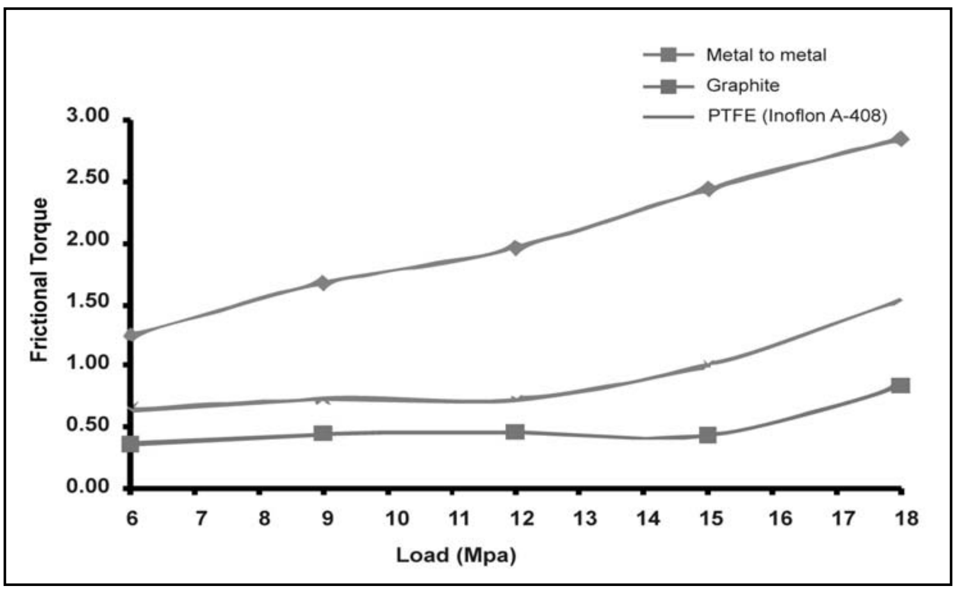

Fig. 4: Frictional Torque of Metal to Metal, graphite and PTFE (Inoflon A-408)

The drop in coefficient of friction was observed both with increased loading of both PTFE grades upto 10% as well as increased radiation exposure dose. But in case of compositions of graphite –PTFE (Inoflon A-408), there is not much difference in coefficient of friction was observed between 50 &100 kGy exposed compositions of graphite –PTFE (Inoflon A-408).

Frictional torques showed for these compositions are shown in Fig. 6. It means that 50 kGy dose is optimum dose that imparts

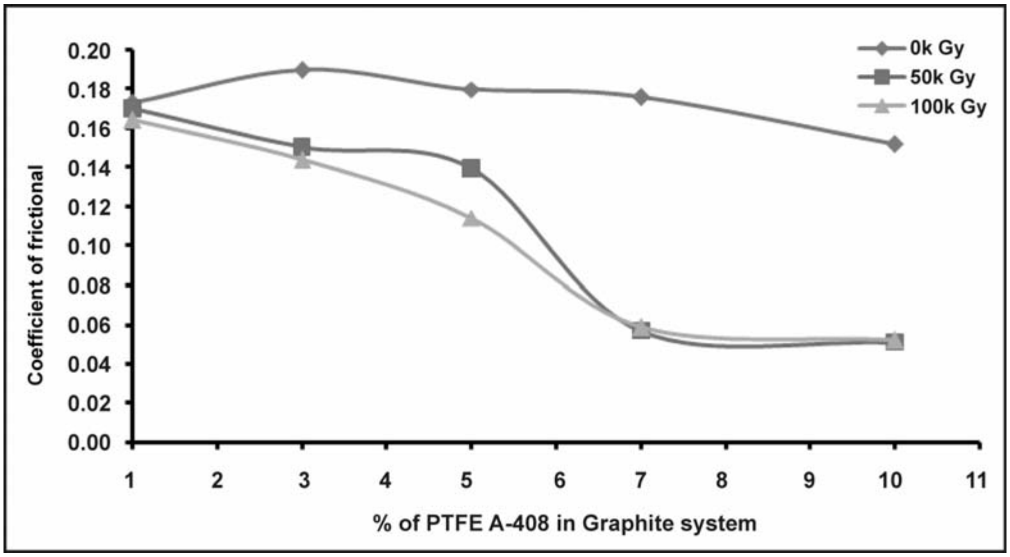

Fig. 5: Coefficient of friction of graphite-PTFE (Inoflon A-408) compositions exposed to gamma radiation

wear loss was observed in case of graphite-PTFE (Inoflon A-408) dry lubrication system. (10%) of PTFE (Inoflon A-408) in graphite is optimized to make dry lubrication system of graphite-PTFE lubrication system resulting into improved lubrication behaviour. The photographs of copper base plate after performing test with modified graphite-PTFE (Inoflon A-408) lubrication system (50 kGy) are shown in Fig. 2. Wear loss values are summarized in Table 4. Wear losses with graphite-PTFE (Inoflon A-408) 1%, 3%, 5%, 7% and 10% were observed as 0.0018, 0.0014, 0.0011, 0.0003 and 0.000 respectively. However no wear loss was observed in case of graphite-PTFE (Inoflon A-408) 10% composition.

The results showed that when graphite-PTFE (Inoflon A-408) dry lubrication systems are exposed to high energy radiation, the lubrication properties of exposed materials get improved upon the physical mixture of graphite –PTFE (Inoflon A-408) lubrication system of both grades mainly due to the changes. In addition to the physical introduction of PTFE into graphite lattices, the ionizing environment is facilitating the fluorination of graphite in presence of PTFE (Inoflon A-408), as the PTFE is well known for molecular degradation under radioactive environment. The lubrication system of Graphite-PTFE (Inoflon A-408) requires 50kGy gamma dose for development of lubrication system of graphite-PTFE (Inoflon A-408). Due to small particle size PTFE (Inoflon A-408) is easily incorporated lamellar structure and lesser dose is required for improvement of lubrication properties. Thus, the improvement of lubrication properties of irradiated graphite-PTFE mixtures is attributed mainly due to the physio-chemical changes occurring in radiation environment. Hence, it has been reported from the results observed for gamma irradiated samples that due the chemical modification of graphite in presence of fluoropolymer such as

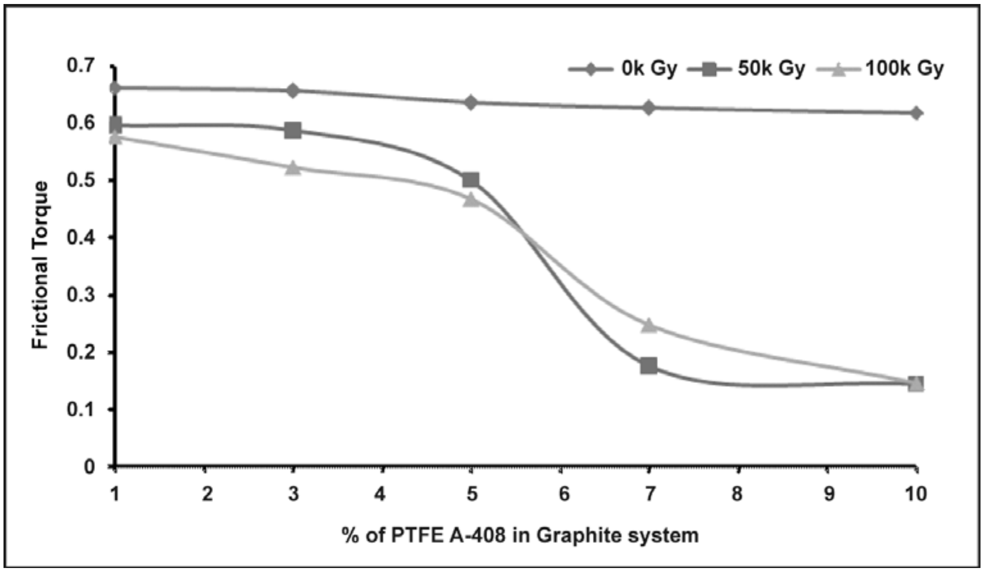

Fig. 6: Frictional torque of graphite-PTFE (Inoflon A-408) compositions exposed to gamma radiation

TABLE 4: Wear loss of developed graphite -PTFE A-408 dry lubrication systems

S. No.	Sample	Wt of Cu- plate before test, g (W_1)	Wt of Cu-plate after test, g (W_2)	Wt Loss (W_1-W_2)	Wear (%)
1	Graphite-PTFE (1% Inoflon A-408)	27.0475	27.0470	0.0005	0.0018
2	Graphite-PTFE (3% Inoflon A-408)	27.0395	27.0391	0.0004	0.0014
3	Graphite-PTFE (5% Inoflon A-408)	27.0702	27.0669	0.0003	0.0011
4	Graphite-PTFE (7% Inoflon A-408)	26.6528	26.6527	0.0001	0.0003
5	Graphite-PTFE (10 % Inoflon A-408)	27.1766	27.1766	0.000	0.000

PTFE, resulting into improved lubrication properties and increased thermal stability.

3.3 XRD Analysis

XRD diffraction patterns of graphite powder, PTFE and graphite-PTFE (Inoflon A-408) lubrication system are shown in Fig. 7. The graphite used in the study is found to be crystalline in nature shows characteristic peaks at 26.73° and 54.87°. Peak at $2\theta = 26.73°$ corresponds to the diffraction line C (002) with the intercellular spacing in the crystal having d-spacing of 3.37A°. Comparatively; PTFE, there were peaks observed at $2\theta = 18.15°$, 31.8°, 37.15° and 41.5°, which could be indexed to (100), (110), (107) and (108) of PTFE, respectively. The peak at 2θ = 18.4° was considered as the crystallinity phase of PTFE. The phase of PTFE is found completely missing in the radiated and developed graphite-PTFE dry lubricant systems, mainly due to the fact that PTFE experiences physico-chemical degradation changes under radioactive environment and the crystalline nature of graphite is found dominant in graphite –PTFE (Inoflon A-408) lubrication system.

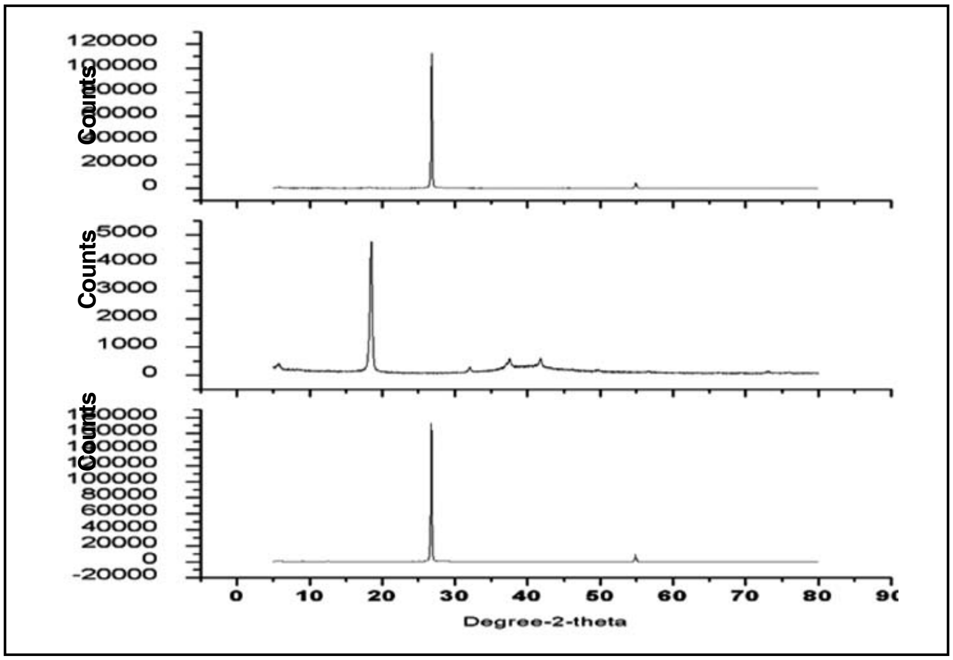

Fig. 7: XRD analysis of graphite, PTFE (Inoflon A-408) and Graphite–PTFE (Inoflon A-408) lubricant system at 50 kGy

3.4 SEM analysis

SEM micrographs of graphite, PTFE and graphite-PTFE (Inoflon A-408) system are shown in Fig. 8. The micrographs showed that top surface morphology of graphite is retained and shape of natural graphite does not change much. Graphite-PTFE (Inoflon A-408) lubrication systems represent white stripes and some white floccules can be seen on the surface, clearly showing PTFE embedded graphite matrix.

3.5 EDAX analysis

EDAX spectra of graphite, graphite –PTFE (Inoflon A-408) lubrication system and graphite –PTFE (Inoflon A-408) lubrication system are shown in Fig. 9 and results are shown in Table 5. In graphite-PTFE lubrication system, the signal of carbon decreases and the peak of fluorine appears which indicates the fluorination of graphite take place.

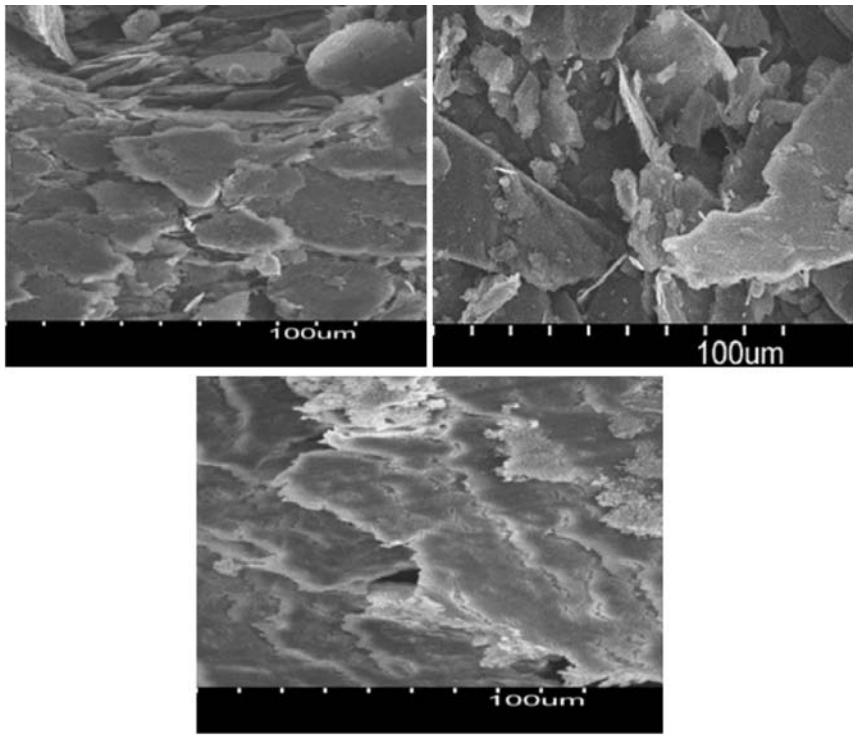

Fig. 8: SEM analysis (a) graphite (b) PTFE (Inoflon A-408) (c) graphite-PTFE (Inoflon A-408) lubrication system

3.6 Contact angle analysis

Contact angle analysis of PTFE (Inoflon) A-408, natural graphite, hand mixing of graphite and PTFE Inoflon A-408 (10%) and radiated sample of graphite-PTFE (Inoflon A-408) (10%) are shown in Table 6. Contact angle of radiated sample of Graphite-PTFE (Inoflon A-408) increases 117.36 to 142.72. The result indicates the fluorination of graphite take place.

3.7 Compressive Strength Analysis

Compressive strength of natural graphite, hand mixed graphite-PTFE (10% Inoflon A-408) and irradiated graphite-PTFE (10% Inoflon A-408) are shown in Table 7. The results indicate the systemic increase in compressive strength of solid lubricant system.

3.8 Thermal Stability Analysis

Thermal analysis of natural graphite, hand mixed graphite-PTFE (10% Inoflon A-408) and irradiated graphite-PTFE (10% Inoflon A-408) shown in Table 8. Thermal stability of radiated sample of Graphite-PTFE (Inoflon A-408) increases 77% to 94%. The result indicates that fluorination of graphite take place. It may due to the chemical modification of graphite by gamma radiation in presence of PTFE, resulting into improved thermal stability.

TABLE 5: EDAX analysis of graphite and graphite PTFE (Inoflon A-408)

Element	Graphite		Graphite-PTFE	(Inoflon A- 408)
	Wt %	Atom %	Wt %	Atom %
C	52.99	64.96	42.36	61.41
O	26.23	24.14	7.05	7.66
Si	20.79	10.90	7.45	4.63
F	—	—	25.60	24.75
Au	—	—	17.54	1.55

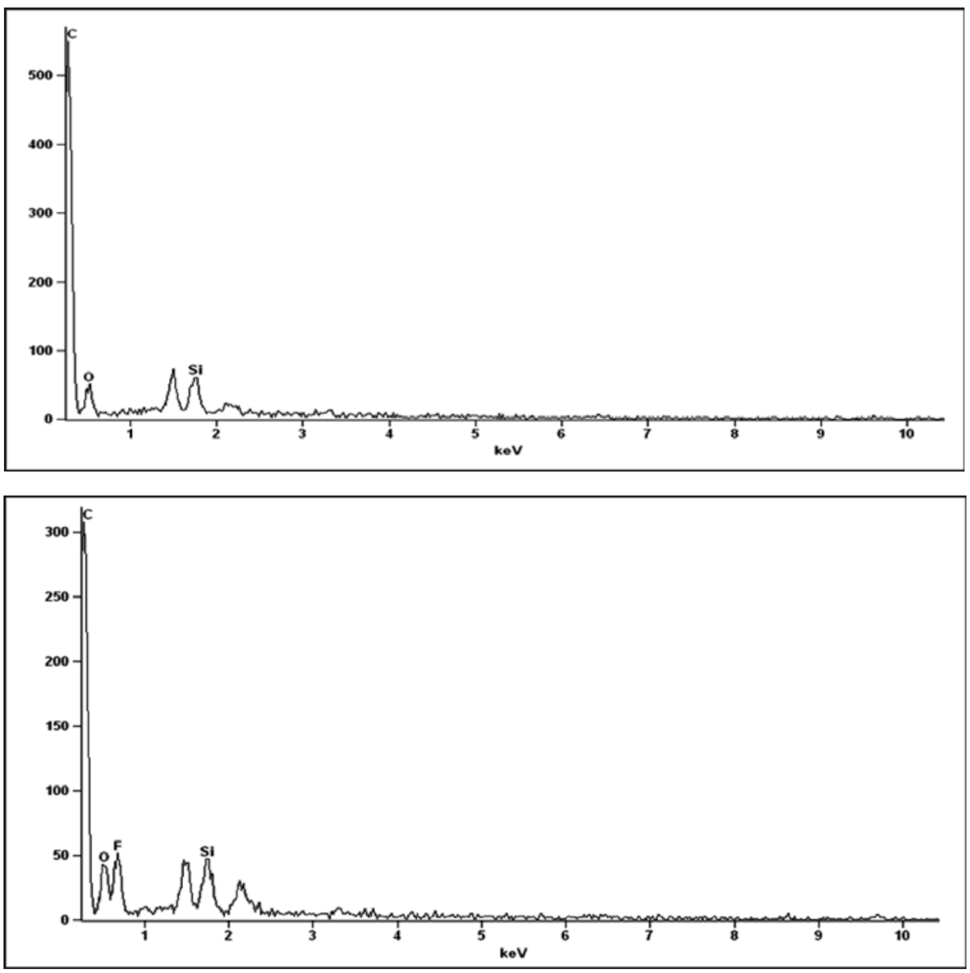

Fig. 9: EDX analysis of a) Graphite b) Gaphite -PTFE (Inoflon A-408) lubrication system

TABLE 6: Contact angle analysis of graphite and graphite -PTFE (Inoflon A-408)

S. No.	Sample	Contact angle (Degree)
1	PTFE (Inoflon A-408)	117.36
2	Natural Graphite	90.00
3	Graphite and PTFE Inoflon A-408 (10%) at 0 kGy	128.23
4	Graphite + PTFE Inoflon A-408 (10%) at 50 kGy	142.72

TABLE 7: Compressive of graphite and graphite -PTFE (Inoflon A-408)

S. No.	Sample	Compressive Strength (MPa)
1	Graphite-PTFE (10% Inoflon A-408) at 0 kGy	5.42
2	Graphite-PTFE (10% Inoflon A-408) at 50 kGy	8.46

TABLE 8: Thermal analysis of graphite and graphite -PTFE (Inoflon A-408)

S. No.	Sample	Residual mass (%) at 1200 ºC
1	Graphite-PTFE (10% Inoflon A-408) at 0 kGy	71
2	Graphite-PTFE (10% Inoflon A-408) at 50 kGy	94

Thus, based on the result of SEM, contact angle analysis and XRD, it can be inferred that the modification of natural graphite with PTFE (Inoflon A-408) in presence of gamma radiation takes place mainly in terms of physico-chemical intermolecular structure of graphite fluoride resulting into improved lubrication behavior, improved mechanical property and improved thermal stability. Thus, lubricant system of graphite with 10% loading of PTFE (Inoflon A-408) irradiated at 50 kGy is considered to be the best with least coefficient of friction, frictional torque and wear loss.

3.9 Fourier Transform Infrared Spectroscopy (FT- IR Results)

Although graphite is a form of carbon and not much bonding is found in graphite, but some IR peaks were observed in graphite. Peaks at 800—600 cm^{-1} were observed in graphite are due to O-H stretching and peak at 1004 cm^{-1} due to C-O stretching. The peaks in IR spectra (Fig. 10) of PTFE were found at 1201, 1153 due to symmetrical and symmetrical –CF2 stretching. The peak at 638cm^{-1} is due to C-C-F bending, 555 and 505cm^{-1} are due to –CF2 bending. In case of graphite fluoride peaks at 1201, 638, 555, 505 cm^{-1} were disappeared in spectra of graphite fluoride and new peaks at 669 and 1213cm^{-1} were observed which indicates the formation of new compound. The peak at 1004cm^{-1} was shifted to 1028cm^{-1}. The new peak at 1213cm^{-1} [30-32] in graphite fluoride due to the C-F stretching vibration of a tertiary carbon atom shows the formation of graphite fluoride by bonding of PTFE and graphite via

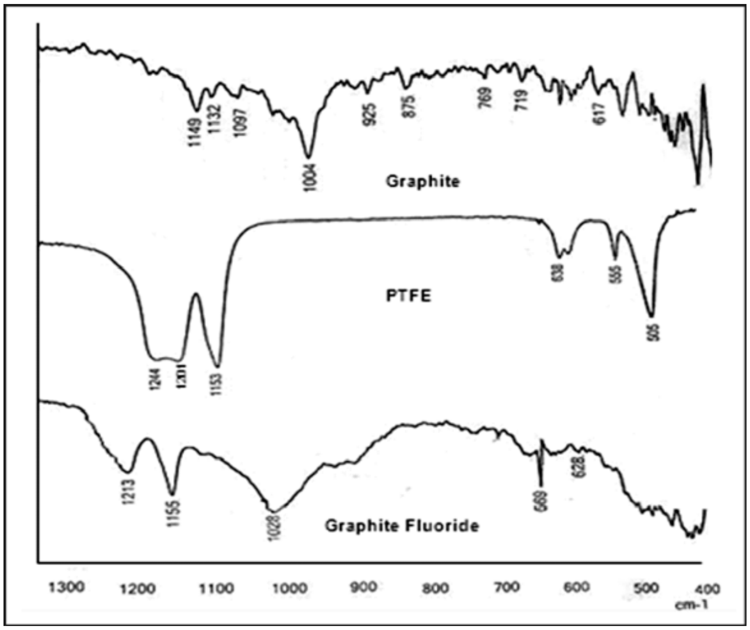

Fig. 10: FTIR spectra of (a) Graphite, (b) PTFE and (c) Graphite Fluoride

gamma radiation. The increased contact angle (142.72°) of developed dry lubricant also favors the results of FTIR showing the formation of graphite fluoride.

4.0 CONCLUSIONS

The results clearly showed improvement in tribological behaviour of graphite-PTFE (Inoflon A-408) compositions exposed to gamma radiation. Moreover, the XRD analysis exhibited predominantly the crystalline phase & morphological pattern of graphite-PTFE lubrication system showed white strips and floccules representing penetration of PTFE into lamellar lattices of graphite. Contact angle analysis and FT-IR analysis results showed that the fluorination of graphite take place. Contact angle analysis showed that developed graphite fluoride dry lubrication system is highly hydrophobic system. The findings of the study are highlighted as under:

- A lubricant system with reduced coefficient of friction, frictional torque and wear loss can be produced by loading 10% PTFE (Inoflon A-408) in graphite and exposing it to gamma radiation at 50 kGy for molecular interaction, physically as well as chemically. Gamma radiation showed the chemical modification of graphite in presence of fluoropolymer such as PTFE, resulting into improved performance of lubricating system. Graphite fluoride does not require adsorbed vapors or adjuvant impurities in order to lubricate and improves the lubricity and durability and makes it less sensitive to variations in ambient humidity. Cost effective and environmentally safe technique is used for development of graphite fluoride dry lubricant of Inoflon (A-408) and natural graphite.

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