STUDYING THE EFFECT OF PARTICLE MORPHOLOGY AND DENSITY ON DUST CLOUD DYNAMICS IN MINIMUM IGNITION ENERGY (MIE) DEVICE BY DIGITAL INLINE HOLOGRAPHY (DIH) TECHNIQUE

A Thesis

by

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ABSTRACT

Dust explosions can cause the most serious and widespread of explosion hazard which can have irreversible impacts on properties, environment, and lives. Thus it becomes imperative to understand dust explosions is a much detailed and lucid way. Several studies have been done in the past focusing on Dust explosions. Though researchers have worked and tried to understand the impact and consequence of the dust explosion, limited work has been done to decouple the role of parameters affecting the dust explosion like dust's morphology, concentration, density, etc. The current experiment aims to explain the role of change in morphology and change in density in a dust explosion by studying the dust cloud dynamics.

The current experiment is divided into two parts-first finding the role of morphology in a cloud dynamics and other roles of density in the cloud dynamics. For morphology experiment two aluminum dust samples (spherical and irregular) with similar size distribution, polydispersity and chemical composition are taken and the density experiment, two common industrial dust, one with high density and other with low density is taken. Soda-lime glass (SLG) is taken as high dense dust and Polymethyl Methacrylate (PMMA) is taken as low dense dust. For both the experiments, the dust particles are dispersed in the Kühner MIKE3 MIE apparatus and the dust cloud is analyzed by Digital Inline Holography (DIH) Technique. The experiment has shown that irregular shaped particle has a higher concentration in the dust cloud than spherical shaped particles. In density experiment it was concluded that less dense samples had more dust particles in the dust cloud and could be suspended in the air for a longer duration, making less dense particles more susceptible to a dust explosion. This study helps in highlighting

the role of two of the most important parameters in dust explosion namely, shape and density. This understanding will help in developing a more viable and potent mitigation measure.

DEDICATION

This thesis is dedicated to my father and mother Mr Radha Krishna Prasad and Mrs Usha Devi,

for their unwavering support.

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NOMENCLATURE

APS	Active Protection System			
ASTM	American Society for testing and Materials			
BS	British Standard			
CSB	Chemical Safety Board			
DIH	Digital Inline Holography			
ISD	Inherent Safer Design			
K _{St}	Deflagration Index			
LOC	Limited Oxygen Concentration			
MEC	Minimum Explosible Concentration			
MIE	Minimum Ignition Energy			
MOC	Management of Change			
NFPA	National Fire Protection Association			
OSHA	Occupational Safety and Health Administration			
Р	Pressure			
PPS	Passive Protection System			
R	Universal gas Constant			
SDSs	Safety Data Sheets			
SCM	Scanning Electron Microscope			
Т	Temperature			
TFV	Terminal Fall Velocity			
TNT	Trinitrotoluene			

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1. INTRODUCTION

Dust explosion is a big threat to process industries with an average of more than one major incident per month in the United States since 1980 [1]. BS 2955: 1958, says that any particle having a size less than 1000 μ m falls under the category of powder and particles size less than 76 μ m are dusts[2], and as per NFPA [3][4], particles having sizes less than 420 μ m are referred as "dusts". These two guidelines are disparate, at least on the sizing of the dusts, as there is almost 6 times difference in the order of magnitude of the particle size. This makes a very subjective approach for designing mitigation measures for dust explosions.

Though proof of dust explosion dates several centuries back, the actual work on dust explosion was started only in the late 1800s. A study by in 2004[5], showed that more than 70 % of the dusts used in process industries are vulnerable to a dust explosion.

1.1 Dust explosion background

A dust explosion is a fast ignition of flammable suspended particles in an oxidizing medium. Any solid material that can burn in air will do so with violence and speed that increases the degree of subdivision. [6].

A typical dust explosion is represented through a dust explosion pentagon (Figure 1). Fuel (Dust) forms an integral part of the pentagon and is required along with oxygen, ignition source, dispersion, and confinement, for an explosion to occur. Fuel (dust) forms an integral part of the pentagon and is required along with oxygen, ignition source, dispersion, and confinement, for a dust explosion to occur. Fuel (Dust) is mainly characterized by its size, dispersity, chemical composition and morphology (surface area/shape).



Figure 1: Dust pentagon, Adapted from[7]

Confinement of the dust cloud plays an important role in determining the intensity of the dust explosion. An unconfined dust explosion can cause a flash fire, though not very lethal it may instigate the dust particles, leading to dispersion of dust in the air, resulting in a major second explosion, whereas heat and the explosive pressure generated by a confined dust explosion can cause a detonation with extremely high temperatures. Even the size of the dust particles plays a vital role in the explosion. An explosion due to finely divided dust has more potential of causing high explosion pressure and temperature than little coarse particles as less the particle size, less energy would be required for igniting the dust (greater surface area), thus making the smaller particle more hazardous. Though, this will be true until a point, when the dusts will become so fine that they will start to agglomerate together. Apart from the diameter of the particles the rate of energy release due to the ignition of the dust particles to the degree of heat loss and confinement also plays a very vital role. A journal by KL Cashdollar [8] explained that in a very rare case of events if the reaction caused by the combustion is so fast that pressure generation is more than the pressure dissipated from the cloud, then we have a detonation even in an unconfined dust explosion.

Fuel (dust) + Oxygen = Oxide + heat

Though the above equation is generic, some metals react with atmospheric gases like nitrogen and explode [9]. As mentioned above, various factors affect a dust explosion, which can be shown by the ideal gas equation:

P = nRT / V

Where P is the pressure, V is volume, R is universal gas constant and T is temperature. Thus, we can see that P is directly proportional to T. From above we can say that, more the heat of combustion, per mole of oxygen, greater would be a hazard of a dust explosion. The dust and air mixture falls in the range of explosion or deflagration if combustible dusts are present in such quantities in the air that an explosion/deflagration could happen due to ignition. The following Table 1, depicts the heat of combustion of some common dusts.

Material Oxidation products Heat of combustion			
Substance	Oxidation Product(s)	KJ/mole O ₂	
Calcium	Cao	1270	
Magnesium	MgO	1240	
Aluminum	Al ₂ O ₂	1100	
Silicon	SiO ₂	830	
Chromium	Cr ₂ O ₂	750	
Zinc	ZnO	700	
Iron	Fe ₂ O ₃	530	
Copper	CuO	300	
Sucrose	$CO_2 + H_2O$	470	
Starch	$CO_2 + H_2O$	470	
Polyethylene	$CO_2 + H_2O$	390	
Carbon	CO ₂	400	
Coal	$CO_2 + H_2O$	400	
Sulfur	SO ₂	300	

 Table 1 : Heat of combustion of common dusts, Reprinted from [9]

1.2 Past major dust explosions

One of the earliest records of dust explosion dates back to 1807 when a ship loaded with 37,000 pounds of gunpowder harbored in the docks of Leiden in the Netherland exploded [10]. The blast left 152 people dead and over 200 houses destroyed. It is said that preparation of food on the deck ignited the dust, leading to an explosion equivalent of 9000 kg of TNT explosion. One of the earliest recorded dust explosion in US history was at Peavey terminal elevator at Duluth, the USA in the year 1916. This pre-modern era dust explosion involved grain dust as a source of fuel, which was rarely recorded at that time. After the explosion, the grain storage bins caught on fire and kept burning for a couple of days. [11]. A similar kind of explosion occurred in a corn processing plant in Illinois, killing 42 people. [12]. One of the major dust explosion of the modern era, occurred at 5.2 m³ in a batch mixer in the year 1973, when Aluminum flakes mixed with other chemicals at a slurry explosion factory in Norway got uncontained and formed a dust cloud, which ignited. A secondary explosion happened due to the first explosion which killed 10 workers who were working at the site [6]. One of the most recent recorded dust explosion in the USA, happened at West Pharmaceuticals in the year 2003. The explosion leads to the death of 6 workers working in the plant and injuring the other 38. The massive explosion initiated secondary fires up to 2 miles from the source. The dust somehow got agitated at got released and got in contact with an ignition source leading to a dust explosion, which also killed two Firefighters [13]. There have been several other explosions, though mentioning all here won't be viable.

1.3 Dust Pentagon

A fire triangle have-fuel, oxidant, and a heat source. When these three come together a fire occurs. A dust explosion needs two other parameters in addition to the parameters of a fire

triangle. These are "mixing of dust and air" and "confinement of the dust". Together these five parameters are needed for a dust explosion to happen, and known as "Dust explosion pentagon". As mentioned in Figure 1, the parameters for dust explosion are

- i. Finely divided dust particles.
- ii. Presence of oxygen (oxidizing agent).
- iii. Presence of an ignition source.
- iv. Mixing of dust and air in proportion quantities.
- v. Degree of confinement of the dust.

It is to note that even if there's partial confinement of dusts, it's capable to culminate into a major damaging explosion. In other words, we can say that dusts behave similar to flammable gases [14].

1.4 Details about dusts

The industries which are usually affected by dust explosions are following [15]:

- i. Food Processing Plants- sugar, grains, egg whites, powdered milk, etc
- ii. Synthetic Manufacturing Plants rubbers, plastics, etc
- iii. Wood Processing Plants- sawdust
- iv. Metal Processing Plants aluminum, chromium, iron, magnesium, etc
- v. Pharmaceutical Plants- pharma dusts

In addition to the above mentioned, fine dusts accumulated at shelves, above false ceilings, bins, bags etc in any of the above mentioned industries are prone to dust explosions. A general phenomena for dust explosion is shown at Figure 2.



Figure 2 Events of dust explosion, Reprinted from [16]

1.5 Classification of dusts

All explosible dusts are combustible, but vice versa is not true. A cloud of dust is combustible if it is ignited by an external source, thus generated propagates pressure sufficiently after the external source is taken away [17]. HM Factory Inspectorate of the Department of Employment, UK provides a list of experimentally tested dust, which can be used to know the explosibility of the dust if its concentration is known. The explosibility of a dust at room temperature (25° C) can be divided into two categories:

- i. Group A : dust ignites, and propagates into a flame.
- ii. Group B : do not propagate into a flame.

If the temperature of dusts is increased, Group-B dusts can also explode. The addition of flammable fuel dust can also lead to the conversion of dust which is not capable of exploding to explosive dusts [8]. The combustion class is also one of the factors when we consider the ignitability of a dust layer [18]. This characteristic depends on the nature and reaction of the dust when the ignition source is provided [9] :

i. CC1 : no ignition, no self-sustained combustion.

- ii. CC2 : localized combustion of short duration.
- iii. CC3 : local sustained combustion with no propagation.
- iv. CC4 : propagation smoldering combustion, spreading of fire.
- v. CC5 : propagation of open fire with open flames.
- vi. CC6 : explosive combustion with explosible burning.

The ignition and its impact are hugely dependent on the dust's particle size. Moisture content, ambient temperature, humidity, oxygen availability, the morphology of the dusts and its concentration also affects the ignition. Different dust samples of the same chemical can have different explosive characteristics depending on the factors mentioned above. Apart from the above parameters, another parameter called K_{St} , also called the deflagration index. It is the relative measure of the explosion severity compared to other dusts. More the value of K_{St} more explosive and severe the explosion from the dust would be. Any K_{St} value greater than zero can cause deflagration. Even a small K_{St} value can cause an initial small explosion leading to a gitation to dust, leading to a secondary major explosion [19]. Kst values of dusts based on dust explosion class is depicted in Table 2. The concept of K_{St} was introduced by a German scientist Bartknecht, the so-called cube root law.

The deflagration index shown below gives the maximum pressure generated in a $1m^3$ vessel when the dust is ignited from an external source of ignition.

$$(\frac{dP}{dt})_{max} V^{1/3} = K_{St}$$

$$\frac{dP}{dt} = Rate of rise of pressure$$
$$V = Volume$$

Few of the K_{St} values of the common dusts are mentioned below [19]

Dust	K _{St} (bar.m/s)	Characteristic*	Typical Material
Explosion			
Class			
St 0	0	No explosion	Silica
St 1	>0 and ≤ 200	Weak Explosion	Powdered milk, charcoal, sulfur, sugar etc
St2	$>200 \text{ and } \leq$	Strong	Cellulose, wood flour etc
	300	Explosion	
St3	>300	Very Strong	Aluminum, magnesium etc
		Explosion	
*OSHA CPL 03-00-008 – Combustible Dust National Emphasis Program			

Table 2 Dust class classification

1.6 Factors affecting the explosibility of dust clouds

A lot of factors can influence the potency of the dust cloud explosion. A holistic and comprehensive list of the factors which may affect a dust explosion is below:

1.6.1 Particle size

One of the most important factors affecting the dust explosion is dust particle size. As mentioned above, scores of dust particle sizes are used in industries. It is very often is very easy to create a favorable dust cloud if we use a very small particle size. At the same time, it increases the risk associated with dust cloud explosion. Because smaller particle makes the dust explosion more powerful, as smaller particles would have a more specific area leading to a more uniform distribution of input energy for the explosion.

1.6.2 Moisture content

It has been shown in several studies that more the moisture content in the dust cloud, less the explosibility of the dust cloud [9]. It also remarkably influences the ignition sensitivity of the dust cloud. This may be due to several reason-the vapors generated from the moisture can create an inert environment for the dust inhibiting the strength of the cloud and moisture tends to increase the cohesiveness of the particles leading to more agglomeration as explained at *1.6.4*.

1.6.3 Particle shape/particles distribution

Very recently it was shown that particle shape too plays an important role in effecting the MIE of the dust cloud [7]. Irregular shaped particle virtue of their shape tends to absorb more energy per unit area and explode easily than regular shaped (spherical) shaped particles. Though there is a limiting particle size, below which the strength of dust cloud does not increase or stays stagnant depending on process devolatilization, gas-phase mixing and gas phase combustion [37].

1.6.4 Agglomeration

The degree of particles sticking together too plays a role, as it influences the net exposed surface area of the dust. More the sticking of particles less would the potency of the dust cloud explosion [9].

1.6.5 Turbulence in the cloud

The stochastic nature of the dusts in the cloud influences the explosion as well. More the turbulence in the cloud more strength the explosion will have [9].

1.6.6 Oxygen concentration

Like any explosion, dust explosion too requires oxidation (oxygen) for the explosion. The scarcity of oxygen can inhibit the effectiveness of dust explosion. Thus, inerting is one of the major measures adopted while considering mitigation measures against dust explosions[9].

1.6.7 The shock wave from the initial explosion inducing turbulence in the unburnt part of the dust cloud[9].

Apart from the above-mentioned factors industrial processes like (heat transfer, mass transfers, etc) also influence the dust cloud explosion. Add to the above location of the dust cloud and location of the ignition source can also play a vital role.

1.7 Domino Effect in Dust Explosions

1.7.1 Initial/Primary dust explosion

Dusts required for a full-blown explosion rarely accumulate out of the process of industrial vessels. Most of the recorded dust explosion starts within a piece of process equipment (such as silos, mills, ducts, hoppers, mixers, filters, etc). The initial explosion in any of the process equipment leads to a primary dust explosion. This particular property of a dust explosion differentiates it from a flammable gas explosion, as it is very rare that vapor or gas explosion happens inside the process equipment primarily due to the scarcity of an oxidizing agent. Though if a dust explosion happens, it might cause rupture of the process equipment leading to the introduction of oxygen in the equipment causing vapor/gas explosion [9].

1.7.2 Secondary dust explosion

The shock wave generated from the primary dust explosion can entrain a dust layer nearby which can again cause an explosion due to the heat transfer from the initial explosion(Figure 3). It has been seen that dusts taking tiny space, when disturbed can form a huge dust cloud. It has been observed that a 5m deep cloud of 100g/m³ dust can be generated just from 1mm of dust layer of 500 kg/m³ dust. Usually, the secondary explosion dictates the explosibility of the dust cloud as a secondary explosion is more devastating and causes more damage to life, environment, and equipment [9].



Figure 3: Domino effect in dust explosion, Reprinted from [9]

1.8 US Regulations for dust explosions

The Chemical Safety Board (CSB) is an independent federal agency that investigates industrial accidents, including dust explosions. Major findings of these investigations of dust explosion have dwelled that [20]:

- Over 100 people have died and more than 700 people have gotten injured in nearly 300 dust explosions in the last 25 years
- ii. US safety regulations do not address dusts explosion prevention and mitigation in a comprehensive manner.
- iii. Most of the Safety Data Sheets (SDSs) of dusts do not contain dust explosion hazard information.
- iv. Most of the standards are voluntary unless adopted by state or local jurisdiction as fire codes.

1.8.1 OSHA standards and regulations

OSHA has several mandatory standards, which look the certain parameters of dust hazards. Some of the major OSHA's guidelines which are currently being followed in the US is as under: *1.8.1.1 OSHA 3644-04 2013: Combustible Dust- Firefighting Precaution at Facilities*

This OSHA standard deals with the Firefighting Precautions at facilities with combustible dusts. The primary purpose of this regulation is to protect the emergency responders from harm by giving them necessary prior information about any hazard and safe operating procedures. It helps in identifying the firefighters and other emergency responders who might be called upon an emergency. Though, this regulation does not provide specific strategies of measures to be used in an emergency scenario. The regulation is limited to the fire and the explosive hazard of the combustible dusts [21].

1.8.1.1 OSHA 3674-2013: Combustible Dust- Precaution for Fire Fighters to prevent Dust Explosions

Firefighters can create conducive conditions for a dust explosion to happen by introducing air or by agitating dusts forming dust clouds, bringing an ignition source or applying incompatible extinguishing agents. This regulation helps in addressing the above mentioned issues. It explains a standard operating procedure to be followed by the firefighters in an event of a probable dust explosion [22].

1.8.1.2 OSHA 3878-2009: Combustible Dust: Protecting Workers from Combustible Dust Explosion Hazards

This regulates tells the employees working in a dust explosion prone industry, to mitigate the effects of dust explosion. It tells how to control the dust, responsibilities of the employers to keep workers safe and the worker's right. This regulation regulates the employer to report and contact OSHA in case of a dust explosion [23].

1.8.1.3 OSHA 3371-2009: Hazard Communication Guidance for Combustible Dusts

The hazard communication standard comprehensively addresses the evaluation of the potential hazards of chemicals and the communication of hazard information to the workers. It is a performance-based standard that can be relevant to any chemical industry, in which workers working could be exposed to the hazard of dust explosion. This regulation is intended to help producers and importers of chemicals recognize the potential of dust explosion to identify proper protective measures as a part of their Hazard Communication Standard [19].

1.8.1.4 OSHA Hazard Information: Improper Installation of Wood Dust Collectors

This hazard bulletin provides non-compulsory regulations regarding mitigation of potential fire and explosion hazards in the workplace associated with improperly installed cyclone dust collectors in the wood industry. This deals with location and installation of the walls, vents, ducts, etc[24].

1.8.1.5 OSHA Safety and Health Information: Combustible Dust in Industry: Prevention and Mitigating the effects of Fire and Explosions

This regulation is a non-mandatory regulation that can be implemented in the process industry dealing with combustible dusts. It deals with the hazards associated with the combustible dusts. Work Practices and the guidelines, which shall be implemented and followed which can reduce the hazard of potential for a combustible dust explosion. It provides a training module for the workers, helps them in identifying and protecting themselves from the dust explosion hazards.

1.8.1.6 OSHA Hazard Communication Standard

This OSHA's communicating tool mandates the chemical manufacturers or producers or handlers at their workplace for potential hazards, and to convey required Safety Data Sheets (SDSs) with the chemicals they process, in any form [20].

1.8.1.7 OSHA General Duty Clause

This requires employers to provide a workplace with no potential hazards providing a conducive environment and a healthy workplace.

OSHA issued a Safety and Health Information Bulletin (SHIB-OSHA 2005) on combustible dusts which never got approved and hence didn't developed an outreach program to distribute the SHIB to potential companies which may be impacted by dust explosions [18].

1.8.2 NFPA standards

National Fire Protection Association (NFPA) provides guidelines and regulations/codes which provided measures for preventing and mitigating dust explosion hazards. These codes and regulations are non-mandatory unless it is enforced by the local or state law jurisdiction. Some of the major NFPA standards which deal with the hazards of dusts explosions are mentioned below: *1.8.2.1 NFPA 61* : *Standard for the prevention of fires and dusts in agriculture and food*

processing facilities

The standard is equipped with guidelines applicable for agricultural/or food processing plants for mitigating fire and dust explosion hazards. It provides the minimum guidelines required for mitigation from fire and explosion hazards arising from dusts related to agriculture and food processing[25].

1.8.2.2 NFPA 484: Standard for combustible metals

This standard gives out the requirement of the facilities required for the plants involved in the production, processing, finishing, handling, recycling, storage and use of metals and alloys that are capable of causing combustion or explosion. This standard is strictly meant only for plants doing the above-mentioned activities, and the transportation of metals or alloys susceptible to explosions [26].

1.8.2.3 NFPA 652: The fundamentals of combustible dust

The standard provides the required minimum criteria for the measures to be taken for managing fires, flash fires, explosions which can occur due to combustible dusts. This guideline assists other NFPA standard for dust explosion mitigation. Wherever other NFPA guidelines are not applicable this standard maybe considered as a thumb rule and should be followed. Though, this standard is a bit generic, unless and until specified in NFPA, this shall not be applied [27].

1.8.2.4 NFPA 654: Prevention of fire and dust explosion from combustible particulate solids

The standard provides mitigation measures for all the stages of manufacturing, processing, blending, conveying, repackaging and handling of combustible particulate solids of its hybrid mixture, independent of its concentration or particle size, or if the hazards associated includes a fire, flash fire or an explosion hazards from the dusts [3].

1.8.2.5 NFPA 655: Standard for Prevention of Sulphur Fires and Explosions

The standard deals with the dust hazards associated with the reduction in the size of Sulphur while handling in any form. The purpose of the standard is to minimize and mitigate the inherent fire and explosion hazards associated with the processing and handling of Sulphur. Though, this standard is strictly applied only to the handling of the Sulphur, not to other processes like mining, recovery or transportation. The standard mandates the employer who is dealing with the process to comply with all the required criteria [29].

1.8.2.6 NFPA 664: Standard for Prevention of Fires and Explosions in Wood Processing and Woodworking Facilities

The standard provide fire and explosion prevention and mitigation measures for commercial, industrial and other facilities which process wood or manufactures wood products involving wood products or other materials having cellulose. The standard would provide the design criteria, operation criteria and other maintenance measures for woodworking and wood processing facilities [30].

1.9 Mitigation measures against dust explosions and fires

Mitigation measures against the hazards associated with combustible dusts, is mainly aiming to nullify the possibility of an explosion or a fire or decrease the consequence if it happens. As a general approach of Fire Protection, consists of Passive Fire Protection and Active Fire Protection, same goes with the prevention against dusts hazard. Following detailed measures are adopted in the industries for dust hazard mitigation.

1.9.1 Passive dust explosion measures

Passive dust explosion measures are an integral part of the structure of the building of the plant. The Passive Protection System (PPS) is an inherent system inculcated during the detailed designed stage itself. The PPS helps in reducing the preventing dust explosion or fires and further to reduce its consequence. The PPSs are hugely influenced by the local building code. The system includes compartmentalization of the building with blast/fire-rated walls, using dust hazard resistant materials while construction, using dampers, fireproofing paints, etc. Most common.

PPSs used in industries susceptible to the hazards of Dusts explosions/fires are :-

1.9.1.1 Inherent Safer Designs(ISDs)

Inherent Safer Designs permanently removes or decreases the hazards related to dust explosions/fires. The gist of the inherent safer approach is the avoidance of hazards, rather than their control. It is a very broad philosophy that is applied to the design and the life cycle which includes transport, storage, manufacture, use, and disposal. The ISDs mainly focuses mainly on :

- Eliminating the dust hazard
- Reducing the dust hazard
- Substituting hazardous dust with no or less hazardous dust
- Using less hazardous conditions, conducive for dust explosions

Inherent Safer Designs (ISDs) are relative. The technology of ISDs is always compared with the existing technology considering the task, location and potential consequence. One ISDs may not be viable for a similar kind of industry at a different location. One ISD may be safer than others with respect to a particular hazard, but it may be inherently less safe with respect to others. Technical and economic viability is also one of the major criteria while deciding for ISDs [30].

1.9.1.2 Building design

A thorough risk assessment should be done which should be as per the guidelines laid by Authority Having Jurisdiction(AHJ). This assessment would give the foundation of building design and protection features that needed to be provided. Certain points to be considered while designing should include, but not limited to :

- Building or compartment inside the building where dust explosion/fire hazard may be present shall be protected from the consequence of deflagration or explosion.
- If in a building a dust hazard exists outside the compartment, the concerned area should be provided with explosion/deflagration venting with compliance to NFPA 68.
- A building should be constructed in a way that the consequence of dust hazards like fireball, missile hazards, etc should not expose additional personnel or property damage.
- A building or a part of a building where hazard of dust explosion exists, and the means of egress is enclosed, the egress should be designed to sustain the potential external overpressure, in case of an explosion or deflagration [28].

1.9.1.3 Construction measures to limit the accumulation of dust

Accumulated dusts pose on the major threats in any industry. As per NFPA 654, a dust layer with a thickness of 0.8mm has the potential of causing an explosion. Thus the interiors surfaces/false ceilings where dusts may accumulate shall be designed to facilitate cleaning and to minimize the accumulation of combustible dusts. Those part of the building which remains inaccessible to routine cleaning and housekeeping should be kept sealed to avoid the accumulation of dusts [28].

1.9.1.3.1 Separation of the hazardous areas with one other areas and occupancies

The area within the building or its compartments where the hazards of dust explosion exist, though not the hazards associated with the equipment, shall be separated and segregated from other areas, to reduce consequence if, any dust explosion or fire is to occur. The separation can be done by several means, some are :

- **Segregation**: These are the physical barriers that are constructed with the aim of curtailing the spread of fire. These physical barriers help in preventing a fire from penetrating from the opening of the ceilings, the opening of the floors, stairs, etc, thus these barriers should have a minimum fire-resistance rating based on the duration of fire protection required by the management [28].
- Separation: Separation may be permitted if AHJ accepted the documented engineering measures which act as a blueprint used to limit the dust explosion within a part of the building. The separation distance between the hazardous area and the area to be protected is determined by using engineering techniques by evaluating the properties of the material involved, the quantity of dust processed, type of operation, properties of surroundings, etc. The separation areas should be devoid of any dust accumulation, or the dust accumulated should be easily ascertained by the difference in the color [28].
- **Detachment**: It should be used to reduce the hazard of dust explosion, by physically separating the adjacent buildings. The distance of the detachment to prevent the area having hazards of dust explosion to influence the surrounding area depends on the property of the dust being handled, the quantity of dust, actual processes, equipment design, building designs, etc [28].

1.9.1.4 Equipment design

The equipment used in the industry can also be a housing home for the dusts. These equipment might lead to the accumulation of the dust or breaking the material to the range to be

called dust. A risk assessment should be done, which shall be acceptable to AHJ, to ascertain the level of protection that is provided for dust hazards. Several designs have been into practiced, some of these are discussed below:

• **Dust containment design**: The equipment which deals with combustible dust should be leakproof. It should stop the dust from escaping to the surrounding, except the opening meant for intake and discharge of air material.

1.9.1.5 Dust collection, pneumatic conveying, and vacuum cleaning systems

Where there are dust explosion hazards, and the above-mentioned equipment is used the entire system should be supervised by competent personnel who have required knowledge about the potential dust hazards that may arise. A MOC should be done. If an equipment design has to be changed for making it safer. The air-gas velocity used shall increase the required velocity for the equipment to keep the interiors of the piping of the equipment dust-free [28].

1.9.1.5.1 Requirements of pneumatic conveying systems

The system shall be designed as such, at each collecting point, the system shall attain its minimum velocity for capturing and controlling of the dusts, to facilitate better containment of the dust. The lines branching out shall not be blanked off without providing a mean to maintain the required air balance. If there's a requirement of additional lines, it shall not be connected to the existing system without MOC, and airflow should be balanced. The dust collection system, which is used to remove the dust from the system, and produced sparks or elevated temperature should not be connected to the dust transportation system [28].

1.9.1.6 Requirement for centralized vacuum cleaning system

The system shall be designed to maintain a minimum conveying velocity for the entire time the system is used, to avoid the leftover of the dusts. Wherever the system is to be used for high sensitive dusts, proper precautionary measures should be taken like, using material which does not produce static, keeping the equipment grounded all the time, etc.

1.9.1.7 Duct system

Ducts should be designed as such to prevent the accumulation of dusts by utilizing the science of angled construction. The included angle of the taper shall not be more than 30 degrees. Wherever, duct pass through a physical segregation or separation, it should not spread the dusts in between the segregation or separation. The ducts should be designed as such it's free from any sort of leak of dust it is handling. All doors, opening and portable sections of the ducts should be grounded [28].

1.9.1.8 Storage enclosures

These include silos, tanks, bins, hoppers, etc. The enclosure whether built inside the building or outside, should not increase the fire load, more than the designed fire control capability. If the hazard of an explosion is imminent, the storage system shall be out of the building. The interior of the storage tank should be constructed as such that they facilitate the cleaning of the inside of the tanks. The storage facilities should be designed as such no leak of dusts is allowed. The tanks can be equipped with deflagration vents, to prevent the dangers of deflagration. The storage facilities should be properly grounded [28].

1.9.1.9 Bucket elevators

The casing of the elevator and connecting ducts shall be designed as such it controls the emission of dusts outside. These should be constructed of non-combustible materials. The inlet and the discharge shall be designed as such it is completely accessible for internal/external cleaning as well as inspection. Belt driven bucket elevators shall have no-skid belts, to avoid heat generation due to friction. The belts shall be fire resistant and anti-static. The casing of the elevator should be free from any kind of bearing [28].

There are several other passive techniques, which have been inculcated and being currently used. Other techniques are maintaining particulate transport rate to avoid static, electrostatic safe intermediate containers, designing of vehicles that are to be used inside the plant, dust control measures, etc.

1.9.2 Active dust explosion measures

Active Protection Systems (APSs) are a group of protection that work together but need some kind of action or motion for working efficiently. The action may be manual or automatic (through a sensor). These are usually not inherent with the design of the building and are usually an add on to the existing design. Though, guidelines for implementing APSs are usually drafted at the design stage itself, but inculcated after the construction has been done as per the requirement. APSs require regular maintenance, as they have mechanical parts involved. So APSs are usually expensive than PPSs in the long run. The APSs as discussed above can be manual or automatic. The automatic system shall be provided over the manual system when:

- It is having unacceptable risks for humans to activate the system [28].
- Manual systems are not allowed by the concerned building code [28].

Other points which should be considered while deciding about APSs are:

• The Fire-extinguishers which are to be placed inside the plant posing dust hazards should be compatible with the power being handled [28].

- The wet system involving water system, shall be designed as per NFPA 91, to avoid the accumulation of water in the vessel, pipes, supports, and drains.
- The application rate of the dry extinguishing agent should be as such, it doesn't escape and forms a suspended dust cloud of its own.

Several SPSs measures are used in industries posing dust hazards, some of them are following:

1.9.2.1 Fire extinguishers

Portable Fire-extinguishers shall be provided in entire building, which should be compatible with the dust being processed. The placing of the fire-extinguishers shall be as per NFPA 10. Proper training shall be imparted to the workers working, about when and how to use an extinguisher. They should know the hazard associated with the operation, and they should use extinguisher as such it does not create dust of its own [28]. Agents like water, foam, halons, carbon dioxide shall not be used on combustible dusts, unless it's clearly stated that they are compatible with the concerned dust [27].

1.9.2.2 Automatic water sprinkler system

Automatic water sprinklers shall be allowed to be installed only where the dust being processed is compatible with water. Special consideration shall be given while installing a sprinkler system for combustible dusts. Proper training should be imparted to the workers working for activities to be done, if there's an activation of the automatic sprinkler system like the evacuation of the plant, starting of the redundant safety measure if available. Sprinklers hall not be used is the process areas dealing with alkali metal dusts, as it poses combustible hazards [27].

1.9.2.3 Total Flooding System

The total flooding system involves the flooding of an inert chemical or gas over an enclosed area having a hazard of dust explosion. The isolation devices and separators hall be designed to withstand an external pressure of 100 lb/in², which may arise from the activation of an inert chemical. The discharge of the inert chemical shall be as per NFPA 69. Since total flooding system is usually used in high-risk areas that are not manned by humans. Proper working of these systems is very critical for the overall safety of the plant. Hence, proper maintenance of the system is a must, which raises the cost of the overall system quite significantly. The expensive nature of the system controls the wide usage [29]. A general risk management diagram in association with dust explosion is shown at Figure 4.



Figure 4: Dust explosion risk management, Reprinted from [30]
1.10 Recent development in the field of dust hazard mitigation

1.10.1 Effect of shock wave due to different polydispersity and Particle Size

The Particle size of the dust and its polydispersity have an effect on the dusts moving behind the shock wave produced. The study concluded that the smaller the dust particle with high polydispersity are the major factors which decide the rising of the dust and producing waves. Thus, a particle with the same size, but small polydispersity would have a lower effect [32].

1.10.2 Effect of dust dispersion on particle integrity and the explosion hazard

A well-dispersed dust; which is having a good air mixture ratio will burn well. Moreover, if the particle size is small, less energy is required to excite and ignite it. The research shows that the dispersion concentration and particle breakage are inversely proportional. With an increase in the concentration, there is less particle breakage due to lower in the impact per particle. Though this trend is not linear, thus concentration needs to be determined to know the breakage. This may increase the hazard of dust explosion [33].

1.10.3 Effect of Morphology on the Minimum Ignition Energy of the dust

An irregular shaped dust would be having a more exposed surface to the energy applied than the spherical shaped dust. Thus irregular shaped particles will have different MIE than the spherical shaped dust. This research focuses on the MIE required by different shaped dust particles, and it is shown that the energy which is required for ignition is different [34].

1.10.4 Effect of varying polydispersity on metal dusts

As discussed polydispersity the variability of the dust particle in a crowd. This research shows that the more the polydispersity within a crowd of dust, the more the explosion hazard, and less the polydispersity less the explosion hazard. It states that a fine particle requires less energy to ignite. This initial ignition of the smaller particle provides energy for ignition to the bigger particles, and bigger the particle more energy dissipated during explosion [35].

1.10.5 Risk of dust explosion on combustible nanomaterials

Nanomaterials have valuable properties and are used across various industries. Nanomaterial poses a different kind of hazard due to its unique size. Due to the small size, a very amount of energy would be sufficient in exciting the nanoparticles, resulting in an explosion. The research shows that the explosion risk increases as the particle size decreases and this trend will continue to the Nano-scale. The aggregations of the particles also have an effect on explosion behavior [36].

2. THE EXPERIMENT

Dust explosion is a potent source of destruction. Thus it becomes very important to understand the factors influencing the dust explosion. A lot of studies have focused on the size of the dust particles, dispersity and chemical compositions as fuel parameters affecting dust explosion. However, research on morphology (surface area/shape), as a dust explosion parameter, is scarce and needs more attention.

The current research would try to answer some of the fundamental questions. The entire experiment is divided into two parts, one accessing the role of shape/morphology on Dust explosion and the other explaining the role of density on dust explosion. The experiment would try to explain the role of parameters like particle density and particle shapes effecting the dust cloud dynamics in Minimum ignition device (MIE)- Kühner MIKE3. In this work, a high-speed digital in-line holography (DIH) method is employed to determine the flow field data of the sample particle when dispersed in the MIKE3 MIE apparatus.

For the density experiment, two common industrial dusts were chosen but with contrasting densities and for morphology experiments, two similar samples with different morphology are taken.

2.1 Minimum Ignition Energy Equipment (MIE)

The Minimum Ignition Energy (MIE) device, as shown at figure 5 is primarily used to measure the minimum ignition energy and LFL of the dust sample. The dust dispersion and the ignition take place in a 1.2L Pyrex glass tube. The tube has a height of 30cm, a diameter of 7cm and a wall thickness of 7mm. For the current experiment, this Pyrex glass tube is replaced with a similar quartz tube with two closed windows for the camera lenses. A mushroom-shaped nozzle

is attached to the stainless steel bottom section, where the sample is loaded. The top section of the vessel has a stainless steel flip panel for the venting of combustion products during the explosion. The compressed gas used for the dust dispersion is injected below the nozzle, using a pressure of 7bar (101.5psi) controlled by a suitably ranged regulator on the compressed air cylinder. Once the dust cloud is formed, it is ignited by the electric spark. The spark is generated between two tungsten electrodes located at 1/3 height with a 6 mm gap in-between. The electrode has a diameter of 2.4mm. Compressed air used for pneumatic actuation and dispersion system should not exceed 7bar. Material loading capacity corresponds to dust concentration between 60 and 5000 g/m³ or (0.0072 and 6g). The vessel can stand overpressure around 8 to 12 bar which corresponds to the overpressure generated by most industrial dusts on an explosion. Recognized explosive materials such as gun power or pyrophoric substances should not be tested in the MIE device. Since micro-sized flammable dusts are involved while usage of this apparatus, it poses hazard related to inhalation of micro dust and high voltage due to electrodes, so extra precaution should be taken while dealing with these kinds of dusts. Technical information about the MIE device is shown in Table 3.



Figure 5: Minimum Ignition Energy(MIE) Device, Adapted from [31]

Technical Data of MIE Device		
Dust distribution System	Mushroom shaped nozzle	
Energy Range	1mJ-1J	
Charging Voltage(1mJ-10mJ)	15kV	
Charging Voltage(30mJ-1J)	11kV	
Triggering(1mJ,3mJ)	High-Voltage relay	
Triggering(10mJ-1J)	Moving electrode	
Inductance(with)	1.0mH	
Inductance(without)	0.01mH	
Compressed air supply	7 bar(over pressure)	
Power supply	100-240 VAC/ 180VA/ 50-60Hz	

Table 3 MIE device technical data

2.2 Experiment Apparatus Setup for DIH

The experiment uses MIKE3 along with DIH components to image and record highspeed holographs. MIKE3 is an MIE testing device, with a 1.2L tube, inside which dispersion of the dust is done. The entire setup is partitioned into three parts: the laser, MIKE3, and the imaging and data acquisition system. Laser beams are generated by the laser arrangement, which lights up the dust cloud dispersed in MIKE3. This is then recorded by the imaging and the data acquisition part. A schematic diagram of the experimental setup is shown in Figure 6.



Figure 6: Schematic diagram of the experimental setup

The laser is produced by a solid-state diode-pumped laser (continuous wave, Oxxius 532). The laser beam is attenuated by a neutral density filter and then is reflected by a mirror placed at 45° to the path. The beam is further diverged by two negative lenses and further reflected by a second mirror placed at 45°. The reflected beam is collimated by a positive lens before illuminating dispersed dust inside the Hartman tube. The Hartmann tube, a component of the MIKE3 is a 1.2L cylindrical glass tube. For the sake of convenience, this glass cylindrical tube is replaced by a polycarbonate tube with the same specifications as the glass tube. The polycarbonate tube is fitted with quartz glass windows that provide a clear vision for the highspeed camera. MIKE3 has two electrodes which provided a spark for the ignition. With no ignition desired in the experiment, the electrodes are disconnected. Compressed air at 7 bar is passed through the mushroom nozzle of the MIKE3 which disperses the dust inside the polycarbonate Hartman tube. A stainless steel flapper stops the dust from exiting the tube during the experiment. The DIH consists of a magnification lens and a High-Speed Camera. The lens of the microscope (Infinity K2 DistaMax, Cf-4) is used for the magnification of the dust holograms. Actual pictorial representation of experiment is shown at figure 7.



Figure 7: Pictorial representation of the experimental setup

The attenuated beam from the dust is captured by the high-speed camera which is controlled and recorded by a computer. The 1951 USAF resolution chart is used to calculate the magnification of 225pixel/mm for the experiment. The camera is manually controlled to capture the dispersion at the desired setting. The camera is placed such that it is in the same plane as the electrodes to capture the actual ignition zone. After the holograms of the dispersed dust cloud are recorded, they are saved and further processed. The process omits the position and velocities of out-of-plane dust particles due to uncertainty.

3. DETERMINING THE ROLE OF MORPHOLOGY IN THE DUST CLOUD DYNAMICS IN MINIMUM IGNITION ENERGY (MIE) DEVICE BY USING DIGITAL INLINE HOLOGRAPHY (DIH) TECHNIQUE.

3.1 Introduction

Combustible dusts are almost everywhere around us. They pose potent damage potential, and thus it becomes essential to understand the science behind dust explosions and develop risk assessment and mitigation measures to make industries safer. Scores of industry handled dusts like sugar, milk powder, coal, pharmaceuticals, metal dusts, etc which possess the danger of dust explosion [37]. The information about the risk associated with dust explosion can be inferred by the likely hood of the explosion and the potential damage it can cause. Parameters like Deflagration index -(K_{st}) and Maximum Overpressure-(P_{max}) helps in determining the explosibility whereas parameters like auto-ignition temperature (AIT), Minimum Ignition Energy(MIE), Limited Oxygen Concentration(LOC) and Minimum Explosible Concentration(MEC) influences the ignitability of the dust.

MIE is defined as "electrical energy discharged from a capacitor, which is just sufficient to effect ignition of the most easily ignitable concentration of fuel in air under the specific test conditions" [38]. Though MIE is one of the most important parameters which helps us in understanding the dust explosions, it has been shown that other parameters like $(dp/dt)_{max}$, P_{max} , Flammability limits play an important role as well[6,39]. ASTM standard test methods asks for the generation of a uniform and non-turbulent dust cloud within the testing devices. The usual purpose of the experiment is to mimic an actual dust cloud for precise determination of explosibility and ignitability parameters of the dust particle. However, difficulties are faced to

achieve a quiescent and uniform dispersed suspension which results in variations of local dust concentrations. The majority of the testing devices used in the academic or industrial dwellings (viz MIKE3) use compressed air as a dispersion medium. Thus there is some added degree of freedom in the dust cloud, right before ignition [40]. Particles near the ignition zone of the MIE device face added turbulence due to the shock wave created by the spark, generated by the electrode. Corona discharge is likely to be present if the electrodes have a pointed end. The momentary shock wave will drive out the smaller dust particle, further reducing the dust particles count in the ignition zone [38,41]. Several researchers in the past have studied the pre-ignition turbulence in dust cloud to study its impact on dust parameters like K_{st} , MIE and P_{max} values. The majority of these studies have pointed to high turbulence in the cloud, dust particle collides with each other leading to breakage of a dust particle, forming finer dusts and reducing the MIE of the dust particles and increasing explosion hazards [43].

A recent study conducted by Pranav Bagaria, et al. concluded that the morphology of the dust particle too affects the MIE. Irregular dust particles need less energy or have lower MIE than spherical shaped particle as irregular shaped particles assist more in the process of heat transfer due to greater surface area [7]. These factors make it primitive to understand the role of dust morphology in the dust cloud dynamics. The aim of this work is to study the influence of morphology on dust cloud turbulence during the pre-ignition phase in the MIKE3 device. This experiment uses a high speed digital inline holographic (DIH) technique, taking images of similar aluminum dust particles (irregular and spherical) dispersed in the air inside MIKE3 and obtaining live images of the particles interfaces inside the dust cloud and measuring flow field measurement data. The results obtained from the study will help in understanding the dust cloud

dynamics and factors affecting the dust cloud parameters when similar dust particles are dispersed in MIKE3.

3.2 Material and methods

3.2.1 Sample Preparation

Two different Aluminum, sample was taken for the experiment. The first sample consists of irregular shaped Aluminum particles and the second sample consists of spherically shaped aluminum particles. The samples were prepared by sieving to $< 75 \mu m$ and blending the samples (milling was not done to avoid change in the size and shape of the particles) as per ASTM E2019. Beckman Coulter LS13320 laser diffraction particle size analyzer was used for measuring the particle size distribution (figure 8). Particle size distribution for both the sample was kept as close as possible to avoid any particle size impact on the experiment. After achieving similar particle size distribution for both the samples, the samples were dried in an oven for 8 hours at 60°C to remove any trace of moisture and then stored in a desiccator in sealed containers. Scanning Electron Microscope (SEM) imaging of the two samples (figure 9) was done by JEOL JSM-7500F ultra-high-resolution field emission scanning electron microscope (FE-SCM). The spherical shaped dust particle sample was procured from Henan Yuan Yang Aluminum Industry Co. Ltd, China with chemical composition of greater than 99.5% Al whereas irregular shaped aluminum particle was procured from ECKA Granules Australia Pty Ltd having chemical composition of (99.77% Al, 0.035% Si, 0.108% Fe, and 0.005% Ti).



Figure 8: Particle size distribution of spherical and irregular shaped aluminum particles, Reprinted from [7]



Figure 9: SEM image of irregular (left) and spherical (right) aluminum particles, Reprinted from [7]

3.3 Observation

After the holograms of the dispersed dusts are recorded, they are saved and later sent for further processing. A raw unprocessed hologram is shown in figure 10. The dedicated workplace with codes process the data by selecting a sequence of holograms image by image. The dust particles captured in the hologram are identified and pursued by using a method developed by Jian Gao et al [44] and Daniel R. Guildenbecher et al[45]. There's a well know ambiguity"Depth-of focus problem", which omits the position and velocities of out-of-plane particles. The generated hologram is logged on a greyscale image h (m,n) and stored in the computer.



Figure 10: Unprocessed hologram: spherical (left) and irregular (right) aluminum particles

Daniel R. Guildenbecher et al [45] uses the Reyleigh-Sommerfield diffraction equation, which is numerically calculated and stimulates the analog reconstruction process. In this process, a reference wave illuminates the recorded hologram and further propagates to a form a momentary plane on which a reconstructed image is formed. This reconstruction can be numerically expressed as:

$$E_r(k, l, z_r) = \mathcal{F}^{-1} \{ \mathcal{F} \{ h(m, n) \} G(m', n', z_r) \}$$
(1)

$$G(m', n', z_r) = exp\left(\left(j\frac{2\pi}{\lambda}z_r\sqrt{1 - \left(\frac{\lambda m'}{M\Delta\xi}\right)^2 - \left(\frac{\lambda n'}{N\Delta\eta}\right)^2}\right)circ\left(\sqrt{\left(\frac{\lambda m'}{M\Delta\xi}\right)^2 + \left(\frac{\lambda n'}{N\Delta\eta}\right)^2}\right)\right)$$
(2)

Where E_r is the reconstructed complex amplitude, λ is the wavelength, η is the viscosity of the medium, and m, n, k, 1 = independent variables. The above-mentioned equations are discrete analytical expressions for the Fourier transform of Rayleigh-Sommerfield diffraction kernel [46]. A picture depicting the recording and reconstruction of DIH is at figure 11.



Figure 11: Recording and reconstructing a DIH

3.4 Results

The hologram was recorded approximately 120ms after the dispersion of the aluminum dusts. The time was chosen such that particle concentration would be sufficient enough in the field of view to detect and track the particles which are required for processing of the hologram. An overlay image of the processed hologram of spherical and irregular shaped aluminum is shown at figure 12. The vectors noted to particles in the overlay images explains that particles are still rising inside the tube. On closer look, it can also be seen that few particles have started settling under the action of gravity.



Figure 12 : Overlay image in the field of view- irregular (left) and spherical (right) shaped particles

The processed overlay image helps in explaining the behavior of the particles at the recorded time in an instant.



Figure 13: Planer velocity vs Time for irregular and spherical shaped aluminum particles.

The curve at figure 13 tells us the difference in the velocity of the spherical and the irregular shaped aluminum particles. Though spherical and irregular shaped particles have similar size irregular shaped particles would have more area due to irregular shaped [4]. Owing to more area, the irregular shaped particles will have more contact surface area. After the pressurized air at 7 bar is released for dispersion of the dusts, the interfacial forces acting on the real contact area of the irregular shaped particles would be more than spherical shaped particles. Due to more forces acting on the irregular shaped particles in the upward direction, the irregular shaped particles would attain high velocities than the spherical shaped particles, which is shown in figure 13. The velocity considered is the resulting planer velocity from the x and y-axis. The velocity in the z-axis was not considered as it raises uncertainties. Figure 14 explains the volume occupied by the irregular and spherical shaped aluminum particles after approximately 120ms of dispersion. The curve depicts that the irregular shaped particles are occupying more volume than the spherical shaped aluminum particles. The irregular shaped particles have more velocity than spherical shaped, more irregular particles can reach the line of sight than the spherical particles, forming a cloud of bigger volume. This further explains that more irregular shaped particles are present in the pre-ignition zone.

Past experimental data has shown that irregular shaped particles have a higher coefficient of drag (C_D) than spherical shaped particles [47]. Coefficient of drag influences the terminal fall velocity (TFV). TFV is the velocity a particle attains after it accelerates and reaches a constant velocity. Particles with low TFV can stay suspended in the atmosphere for a longer duration, traveling sizeable distance than particles with high TFV [48].



Figure 14: Volume vs Time for irregular and spherical shaped aluminum particles

TFV is given by Impact Law [49, 50]:

$$TFV = \sqrt{\frac{4gd(\rho_s - \rho)}{3C_D\rho}}$$

Where g is the acceleration due to gravity, d is the diameter of the object and ρ_s and ρ is the density of the particles and the density of the suspended fluid. The sample for the experiment was prepared such that almost all parameters of spherical and irregular shaped particles are similar. Though the coefficient of the drag would be different for spherical and irregular virtue to the difference in the shape of the particle. As irregular shaped particles have a higher coefficient of drag than spherical particles, the TFV for irregular shaped particles would be less (inversely proportional). And since the TFV of the irregular shaped aluminum is low, they would stay in the atmosphere for a longer duration, making irregular dusts potential more hazardous.

4. INFLUENCE OF PARTICLE DENSITY ON DUST CLOUD DYNAMICS IN A MINIMUM IGNITION ENERGY APPARATUS USING DIGITAL IN-LINE HOLOGRAPHY

4.1 Introduction

Industries handling combustible dust face the hazard of dust deflagrations or explosions. The main parameters which aid in the risk estimation of dust explosion are maximum overpressure (P_{max}), deflagration index (K_{st}) and minimum ignition energy (MIE). 20L explosion sphere is commonly used for obtaining P_{max} and K_{st} values while Kühner MIKE3 apparatus is used for MIE values. The testing is done as per ASTM Standards (P_{max} , K_{st} : ASTM E 2016, MIE: ASTM E 2019) by dispersing dust with the help of an air-based system. Turbulence induced due to dust dispersion systems in standard dust testing devices is known to affect the measurement of P_{max} , K_{st} , and MIE. Also, gravitational forces have an impact on particles dispersed in a cloud. However, the influence of dust particle density on dust cloud dynamics is an area that requires investigation.

This study examined the influence of particle density on dust cloud suspension in the MIKE3 MIE apparatus. A digital in-line holographic (DIH) system was employed and captured high-speed images of the dust dispersion at different ignition delays. In this study, two dusts of dissimilar densities were dispersed; no ignition was performed. Flow field measurements of particles in the cloud and concentration of particles in the ignition area were quantified.

Combustible dusts come in a variety of sizes, shapes and particle densities. These properties coupled with a particle size distribution, concentration in dust cloud, and turbulence are important due to their effect on the magnitude and probability of a dust explosion. If not controlled, these properties can result in deviations while determining the explosibility and ignitability parameter values [37]. Besides, the requirements in the ASTM standard test methods call for the generation of a uniform and non-turbulent dust cloud within the testing devices. The intent is to mimic an actual dust cloud for accurate determination of explosibility and ignitability parameters; however, difficulties are found in attempting to achieve a truly quiescent and uniform dispersed suspension resulting in variations of local dust concentrations [40]. This is an artifact of the air-based dust dispersion system found ion most testing devices which causes a significant amount of pre-ignition dust cloud turbulence. Several research studies have been conducted to study the influence of pre-ignition turbulence on cloud dynamics and its impact on P_{max}, K_{st} and MIE measurements. The conclusion of these studies is that high turbulent dust clouds result in increased values of P_{max}, K_{st}, and MIE [40,51-55]. Additionally, particles near the area of ignition in the MIE apparatus are moved away by the shockwaves generated from the spark generation circuit which affects the measurement of the MIE values due to the reduced number of dust particles participating in the ignition [41,56-57]. Such deviations in measured values can cause incorrect estimation of dust explosion risk. Furthermore, dust particles are affected by gravity during suspension [40]. This makes particle density an important dust property that can influence cloud dynamics such as the time of flight of the particles and the maximum height of the dust cloud. High-density dust accelerates slower, settles faster, and reaches a lower height in comparison to low-density dust. The primary reason is the higher inertia for particles with high particle density [58]. Though efforts have been made to characterize the dust cloud flow fields and turbulence using high-speed imaging techniques through experimental studies [59-64], the current analysis is warranted on the impact of particle density on pre-ignition cloud dynamics in an MIE testing device.

The aim of this study is to study the dust cloud. A high speed digital in-line holographic (DIH) system will be used to obtain live imaging of particle interactions and flow field measurements. In addition, particle size distribution and dust concentration near the area of ignition within the dust cloud will also be quantified. The results obtained will better the understanding of dust cloud behavior at various ignition delays when dusts of different bulk densities are dispersed.

4.2 Material and methods

4.2.1 Dust Samples

To study the effect of density, two common industrial dust samples, one with low density and others with high density were taken. The low-density sample was polymethyl methacrylate (PMMA) and the high-density sample was soda-lime glass (SLG)[65-66]. PMMA and SLG both were procured from Cospheric LLC, CA. The chemical composition of these samples are : PMMA(>99%) and SLG(>99.9%). The particle size distribution for the samples was done by Beckman Coulter LS13320 laser diffraction particle size analyzer and the result was reinstated using high-speed DIH, which is explained in figure 15. Once the similarity between particle size distributions was established, the samples were dried in the oven for 08 hrs at 60° C to remove any traces of moisture and then stored in a sealed container in a desiccator. High-resolution images of the samples(figure 16) were taken using the Nikon Eclipse Ti brightfield microscope using a 40x lens and the image was processed by Nikon elements software.



Figure 15: Particle size distribution of PMMA and SLG



Figure 16: High resolution images of PMMA (left) and SLG (right)

The size distribution tells us that D_{50} /mean and the D(3,2)/sauter mean diameter for both the samples are very similar. Moreover, polydispersity (σ_D) which is a vital parameter for affecting dust explosion characteristics [25] is similar for both the samples. Since the basic parameters of these two dusts are similar, any difference in the dynamics of the dust particles in the dust cloud could be attributed to the difference in the density. The size distribution details for PMMA and SLG are mentioned in Table 4.

DUST PARAMETERS				
	PMMA	SLG	Units	
Density	1200	2500	kg/m3	
Mean	51.82	59.3	microns	
D10	47.83	54.06	microns	
D50	51.83	59.29	microns	
D90	56.54	64.45	microns	
D(3,2)	51.63	59.08	microns	
Polydispersity	0.17	0.18		

Table 4: Size distribution statistics of the dusts.

4.3 Experiment Apparatus Setup for Minimum Ignition Energy (MIE) device

The experiment uses MIKE3 along with DIH components to image and record highspeed holographs. The entire setup is partitioned into three parts: the laser, MIKE3, and the imaging and data acquisition system. Laser beams are generated by the laser arrangement, which lights up the dust cloud dispersed in MIKE3. This is then recorded by the imaging and the data acquisition part. A schematic diagram of the experimental setup is shown in figure 17. The laser is produced by a solid-state diode-pumped laser (continuous wave, Oxxius 532). The laser beam is attenuated by a neutral density filter and then is reflected by a mirror placed at 45° to the path. The beam is further diverged by two negative lenses and further reflected by a second mirror placed at 45° . The reflected beam is collimated by a positive lense before illuminating dispersed dust inside the Hartman tube.



Figure 17: Schematic diagram of the experimental setup for density experiment

The Hartmann tube, a component of the MIKE3 is a 1.2L cylindrical glass tube. For the sake of convenience, this glass cylindrical tube is replaced by a polycarbonate tube with the same specifications as the glass tube. The polycarbonate tube is fitted with quartz glass windows that provide a clear vision for the high-speed camera. MIKE3 has two electrodes which provided a spark for the ignition. With no ignition desired in the experiment, the electrodes are disconnected. Compressed air at 7 bar is passed through the mushroom nozzle of the MIKE3 which disperses the dust inside the polycarbonate Hartman tube. A stainless steel flapper stops

the dust from exiting the tube during the experiment. The DIH consists of a magnification lens and a High-Speed Camera. The lens of the microscope (Infinity K2 DistaMax, Cf-4) is used for the magnification of the dust holograms.

The attenuated beam from the dust is captured by the high-speed camera which is controlled and recorded by a computer. The 1951 USAF resolution chart is used to calculate the magnification of 225pixel/mm for the experiment. The camera is manually controlled to capture the dispersion at the desired setting. The camera is placed such that it is in the same plane as the electrodes in order to capture the actual ignition zone. After the holograms of the dispersed dust cloud are recorded, they are saved and further processed. A raw hologram is shown in figure 18 for clarity. The process omits the position and velocities of out-of-plane dust particles due to uncertainty [26].



Figure 18: Unprocessed hologram: PMMA (left) and SLG (right)

4.4 Results and discussion

An overlay image for PMMA and SLG processed to form the hologram approximately 120ms after the dispersion is placed in figure 19. The time was selected such that particle concentration within the field of view was sufficient enough to detect and track particles necessary for the hologram processing routine to operate. A settling dust characteristic can be seen in the overlay image of PMMA and SLG and a small mean particle velocity to the left. The overlay image is giving an idea that more PMMA particles are available in the field of sight than SLG.

After several runs, it was experimentally inferred that 15mg concentration was most viable for generating clear holograms. Since SLG is denser than PMMA, 31.5 mg of SLG was used in another dispersion in order to make the volume comparable between the two dust samples. A total of three dispersions were run: 15mg of PMMA, 15mg of SLG, and 31.5mg of SLG.



Figure 19: Overlay image in the field of view: PMMA (left) and SLG (right)

The overlay image helps in explaining the dust particle flow field at an instant point of time. The density vs velocity curve obtained from the holographs shown in figure 20 gives a holistic view of the dispersed dust characteristics inside the tube. The graph shows that 31.5 mg of SLG has the sharpest rise in the kernel density estimate. This is explained by the fact that 31.5 mg of SLG brings together a large number of particles together and being denser dust itself forms a large dense cloud. The box and whiskers plots show that PMMA has the highest variation in velocity. This suggests PMMA being the less dense dust-gains more velocity and more momentum than the denser SLG [27].



There's a slight increase in the kernel density estimate around the velocity of 15-20m/s for PMMA. This can be attributed to the gained kinetic energy of the small PMMA dust particles

in the due course of time. To validate this result further figure 21 highlights the distinct difference in the volume occupied by 15mg of PMMA and SLG. The higher volume occupied by PMMA can be explained by the higher number of particles being suspended in the air due to its lower density than SLG. PMMA also exhibits higher variation- a phenomenon explained by the longer duration of PMMA particle suspension in the ignition zone.

To make the volume of PMMA and SLG similar, a sample of 31.25mg of SLG was used. Though the volume for 15mg PMMA and 31.25mg SLG should have been similar, there's a clear difference in the volume, as visible in figure 22. The variation in the volume could be explained by a slight difference in the polydispersity of these two dusts. PMMA, being more polydisperse, contains more small PMMA particles in the dust cloud. When these small particles are dispersed inside the Hartmann tube they are suspended for a longer duration which increases the bulk quantity of the PMMA particles. Moreover, due to an increase in the number of SLG particles by using a 31.25mg sample, the collision between the SLG particles would have increased and caused less SLG particles to reach the ignition zone. Also notable, due to the interparticle collision triboelectric effect between the particles cannot be ruled out in this experiment. The triboelectric effect is a phenomenon where particles become charged due to surface contact: sliding, rolling, or impact [67]. A recent laboratory study has shown that smaller particles tend to charge negatively and bigger particles tend to charge positively-forming a polarity between the particles when collided [68]. Due to the developed polarity, smaller particles can conglomerate together forming bigger particles, and thus occupying more volume. The application of DIH at an early dispersion of dust poses a significant challenge for successful

imaging. Usually, for a standard MIE test in Kühner MIKE3, a delay of 120 ms is given between

dispersion and the spark initiation. Due to this short time frame, specific approaches must be developed and implemented for the successful capture and processing of the holograms.

One of the major obstacles for the experiment was to determine the right quantity of the sample for which a clear hologram could be obtained. The most feasible sample quantity was determined after several experimental runs. A balance must be maintained in selecting the concentration of the particle suitable for DIH.



The graph shown in figure 23 provides statistical characteristics regarding the concentration for 15mg of PMMA and SLG dispersed in the MIKE3 right before ignition. There is a distinct difference in the pattern of the error bars for PMMA and SLG. The duration of air

impulse is the same for both dusts; however, because PMMA is less dense the particles remain suspended in the Hartmann tube for a longer duration.





This study examined the influence of particle density on dust cloud suspension in the MIKE3 MIE apparatus. A digital in-line holographic (DIH) system was employed and captured high-speed images of the dust dispersion at different ignition delays. In this study, two dusts of dissimilar densities were dispersed; no ignition was performed. Flow field measurements of particles in the cloud and concentration of particles in the ignition area were quantified.

5. CONCLUSION AND FUTURE WORK

The intent of the morphology experiment was to explain the role of shape in the dust cloud dynamics inside Kühner MIKE3 with the help of high-speed DIH. The two samples, one with spherical shape and other with irregular shaped aluminum dusts were prepared such that almost all the basic parameters remain the same. The current experiment explains that more irregular particles can reach the ignition zone, forming a dense irregular particle cloud, making irregular shaped dust more potent explosion material, than the spherical-shaped particles. Moreover as explained by impact law, the irregular shaped particles would be suspended in the atmosphere for a longer duration, increasing the chance of secondary explosions. As it has already been experimentally shown that, irregular shaped particles have lower MIE [7], the current experiment reiterates that irregular shaped dust particles are more potentially hazardous and extra caution should be taken while handling irregular shaped dusts in the industries.

The second experiment intended to explain the role of particle density in dust cloud dynamics in the Kühner MIKE3 with the help of high-speed DIH. Two widely used industrial dusts with a significant difference in their density were used. As proof of the concept, it could be concluded that less dense dust particles tend to stay in the air for a longer duration occupying larger volumes than denser dust. Less dense particles can be dispersed easily making them more susceptible to dust explosion. Though the results of the experiment were achieved for the preignition stage at a suitably low dust concentration in the field of view, quantitative inference about dust particle flow field data must be obtained prior to application in actual industrial processes.

The current experiments would be continued in the future by studying the organic dusts using the DIH technique. Another study would be conducted to find if the shape or morphology of organic dusts influences MIE of the dust samples. Improvement in the existing DIH techniques could be done by utilizing better optical techniques like using better lasers or better microscopes and the role of other parameters than shape and density affecting the dust cloud dynamics would be ascertained.

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