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The objective of this program was to design and develop a novel hybrid coating technology and related infrastructure for the synthesis of soft phase and hard phase nano particle composite coatings. The project successfully completed design, development and testing of the following two major technologies: (1) Electrostatic coating (ESC) set up for the deposition of solid lubricant as well as hard phase nano particles; (2) Chemical vapor infiltration (CVI) for depositing binder in the porous matrix. The investigators have successfully developed and tested the ESC system for coating lubricant as well as hard nano particles on large areas and complex shapes, and have completed fabrication and testing of the CVI set up. To demonstrate the success of completion of the project, the team has deposited ZnO (0-20 nm) as well as MoS2 (0-700 nm) nano particles as soft lubricant materials and cBN as hard phase material using ESC on complex geometries (carbide insert samples) and has demonstrated TiN infiltration. The research opened many new opportunities to realize practical combinatorial coatings for the next generation of Air Force, Department of Defense (DoD) devices and systems.

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Performance Report

Final Report

COVER SHEET

Research Title: A Novel Technology for the Synthesis and Fabrication of Solid Lubricant and Hard Material Composite Coatings

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Performance Report

Objectives:

The objective of this program was to design and develop a novel hybrid coating technology and related infrastructure for the synthesis of soft phase and hard phase nano particle composite coatings. These objectives are within the scope of the originally proposed research and infrastructure development theme. In particular, the investigators emphasized on developing equipment and process knowledge for the realization of hybrid coating processes, combining electrostatic spray coating (ESC) apparatus along with chemical vapor infiltration (CVI) technology. The goal was to deposit a porous film of nano particles by ESC follow by infiltration using CVI for the vapor phase infiltration. The research opened many new opportunities to realize practical combinatorial coatings for the next generation of Air Force, Department of Defense (DoD) devices with a clear-cut emphasis on homeland security.

Status of effort:

The project successfully completed design, development and testing of the following two major technologies:

(1) Electrostatic coating (ESC) set up for the deposition of solid lubricant as well as hard phase nano particles.

Appendix I is a complete overview of their establishment.

(2) Chemical vapor infiltration (CVI) for depositing binder in the porous matrix.

"Development of Titanium Nitride Chemical Vapor Deposition System for Infiltration and Related Deposition Applications," a thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Micro-Electronics-Photonics, Gilbert Nyandoto, University of Arkansas (2003; *thesis is available in the University library*).

Accomplishments/New Findings:

The investigators emphasized on developing equipment for the realization of hybrid coating processes, combining electrostatic spray coating (ESC) apparatus along with chemical vapor infiltration (CVI) technology. The goal was to deposit a porous film of nano particles by ESC followed by infiltration using CVI for the vapor phase infiltration. The investigators have successfully developed and tested the ESC system for coating lubricant nano particles on large areas and complex shapes, and have completed fabrication and testing of the CVI set up. To demonstrate the success of completion of the project, the team has deposited ZnO (0-20 nm) as well as MoS_2 (0-700 nm) nano particles as soft lubricant materials and cBN as hard phase material using ESC on complex geometries (carbide insert samples). Concurrently, the team chose the appropriate ratio of ZnO:MoS_2, through (in) discussion with Dr. Zabinski at WPAFB, for identifying solid lubricant material phases pertinent to the Air Force's applications. The scientific merit of the project was in the development of a unique technology which breaks the paradigm of 2D multilayer coatings and allows deposition of the material coating phase with a dispersed, infiltrated 3D configuration.

Experimental

The solid lubricants (MoS_2 and ZnO) used for the deposition were procured from Alfa Aesar® in powder form. In particular, average sizes of 2 μm for microsized ZnO (<10 μm), 700 nm for submicron MoS_2, and of 50 nm for nanosized ZnO, respectively, were applied. Scanning electron microscope (SEM) examinations showed that nanosized ZnO powder has clusters of uniform ZnO particles, while MoS_2 powder is in aggregates of particles of various sizes, micronsized ZnO powder is mostly in discrete particle form. The substrates were ground blocks of 1045 steel and WC-Co with a size of 12.5×12.5 mm^2. The small sample size was chosen to facilitate the characterizations. Before deposition, the substrates were pre-treated by degreasing and ultrasonic cleaning.

A schematic of the ESC (corona charging) system is illustrated in Fig. 1. Typically, the substrates are electrically grounded. A high-voltage (negative) potential of several tens of kV is applied to the pointed electrode, then a highly non-uniform electric field is created between the electrode and the substrate, with the density of field lines at different spots implying the field strength. This electric field excites ions and electrons present in the air, which later charge powder particles through their kinetic interactions. With the present configuration of one-gun setup, the sprayed powder particles can easily cover an area of 500×500 mm^2. This setup can be scaled up with multiple guns for large area deposition requirement. In the deposition process, besides the physical properties of powder materials, processing parameters including main air pressure (for powder transport), distance between electrode and substrate as well as electrical potential have influential effects on the deposition quality.

At a main air pressure of 207 kPa (30 psi), electrical potential of –60 kV, and a distance between the substrate and electrode of 150mm, depositions with nanosized ZnO, submicron MoS_2, and microsized ZnO powders were carried out to examine the effects of particle size on deposition. For the understanding of the effect of processing parameters, especially electrical potential on coating formation, powder deposition at potential from –33 to -100kV was performed with nanosized ZnO powder (p=207 kPa, d=150 mm). Weighing method was used to determine the powder transfer efficiency. The deposited samples were characterized for coating thickness, qualitative surface roughness, clusters, and pores with a Hitachi scanning electron microscope (SEM, S-2300).

Results and discussions

3.1 Coating formation based on micron, submicron, and nanoparticles by ESC

Fig. 2 shows the typical deposition of submicron MoS_2 (~15 μm) on 1045 steel substrate, and of microsized ZnO (~20 μm) and nanosized ZnO (~50 μm) on WC-Co substrate, respectively. The deposition time was a few seconds, indicating a high deposition rate (~5g/min) at the selected process parameters. The change of the substrates under the same deposition condition did not yield discernible effect on the coatings. The deposited particles are bonded to the substrates by the combined action of electrostatic force and Van der Waal force, forming the coatings. SEM examination displays that submicron MoS_2 and microsized ZnO coating, in many cases, consists of discrete particles and aggregates of average size less than 5 μm [Figures 2(b) and (d)], while nanosized ZnO coating is made of "netted" clusters of varied sizes [Figure 2(f)], depending on the deposition conditions. This observation is consistent with that obtained in the analysis of the respective powders, which can be explained by the cohesive actions among ultrafine particles, and the deposition mechanism of electrostatic spraying coating.

Both MoS_2 and ZnO powders used in this investigation are ultrafine particles that have very high surface energy, and thus tend to agglomerate to minimize the energy. It is a common phenomenon to form clusters due to the strong cohesive forces among discrete particles. Because of this feature, the powders would be very difficult to delivery by conventional techniques. Even with a fluidization technique, such types of ultrafine particles (Geldart-C) are still difficult to fluidize. ESC is promising to address this problem. It works on the interactions between charged particles and grounded substrates. In ESC process, the movement of powders is initialized by aerodynamic force, which may not be adequate to break all the agglomerations or clusters. The following charging process does help to reduce chances for further agglomeration, but the clusters formed before particles being charged may have few chances to be broken completely. Thus, under the resultant action of air drag forces, electrostatic forces, gravity force, and interfacial forces, the charged particles or clusters migrate to the substrate surface along the electric field lines forming the deposition. Depositing nanoparticles, just like their counterpart, microsized particles, is not only affected by the physical properties of powder particles, but also by the ESC processing parameters.

3.2 Effect of the physical properties of the lubricant powders on the formation of coatings

According to previous studies on the deposition of microsized particles, powder properties including electrical resistivity, powder particle size and distribution, particle shape, and fluidity significantly affect the performance of powder coating. This is especially true for nanoparticles due to their high ratio of surface area to volume. As expected, the fluidity of nanosized ZnO powder is not as high as that of submicron MoS_2 and microsized ZnO powder, however, it does not influentially affect the nanosized powder delivery in ESC process as it does in many other conventional techniques. Under the same deposition conditions and with the same amount of powder, nanosized ZnO powders displayed smoother coating and higher transfer efficiency than submicron MoS_2 and micron ZnO powders, though none of them had transfer efficiency higher than 30%. Compared to the deposition behavior of its counterpart, microsized ZnO particles (< 10 μm), ZnO nanoparticles provided coating of more uniform thickness and density [Figure 2(c)-(f)]. It can be explained by that nanosized ZnO powder has smaller particle size and narrower size distribution than submicron MoS_2 and microsized ZnO. Based on previous studies, powder coating uniformity and transfer efficiency are improved as its average particle size drops down and its distribution is more uniform. In accordance with the measurements of electrical resistivity, both MoS_2 and ZnO can be categorized as semi-conductors, which have a typical value of resistivity of a few 10^6 Ω.m. Though this value is much lower than that of dielectric materials ($>10^{13}$Ω.m), it did not significantly affect their transfer efficiency because particles with this resistivity can still be effectively charged in a corona ESC process.

3.3 Effect of deposition parameters on coating thickness, surface roughness, pore size, and cluster size

In depositing solid lubricants, appropriate deposition thickness and uniform surface morphology are functionally important. The direct response to this requirement is coating thickness, smooth coating surface, and evenly distributed lubricants or pores, which are essential in the follow-up bonding process. In addition to the physical properties discussed above, the ESC processing parameters also have an important role in the coating formation, because these parameters not only affect particles flow, particles charge, but also the adhesion to the substrates. Fig. 3 shows the variation of ZnO nanoparticles deposition thickness with electrical potential change. When other processing parameters are constant, as the absolute value of the potential applied to the electrode increases, the deposition becomes thicker. However, the deposition became saturated at a potential of -60 kV. This is mainly due to the occurrence of back ionization, which limits the further accumulation of charged particles. The onset of the saturation may be marginally adjusted by ball milling the powder. Ball milling induces physical changes in powder particle shape, particle size and its distribution. These changes, in turn, affect particles surface charge and their deposition behavior, and further the deposition thickness. Powder over-coating is the other option to shift the onset of the saturation by changing the electrical resistivity and particle size of the powders.

The maximum peak-to-valley roughness height of the depositions showed slightly decrease as the absolute value of electrical potential was increased to 75 kV, but significantly at 100 kV (Fig.4). This trend is probably due to the quick breakdown of agglomerates by the effective charge from the increased voltages. This observation can be partially proved from the SEM images as shown in Fig. 5, which shows the clusters formed at –100 kV are more uniform than any other depositions in this experiment. The formation of uniform clusters is beneficial to the application because it provides more contact points for high efficient lubrication. According to electrostatic theory, the electrostatic field is directly proportional to the potential, which also affects the charging of the powder particles or clusters in the migration process from spray gun outlet to the grounded substrate. High voltage reduces the time for particles charging to their Pauthenier limit, also results in high ion-wind speed, and expedites the particle movement toward the substrate. This helps to prevent the charged particles from grouping. During a typical deposition time of a few seconds, majority of large clusters can be effectively broken down under the action of high voltage before their deposition to the substrate.

Conclusion

Electrical spray coating was successfully employed to deposit micron, submicron, and nanosized solid lubricant particles on flat samples. According to SEM characterizations of the deposited microsized ZnO, submicron MoS_2, and nanosized ZnO, the following conclusions can be drawn:

(1) Particle size plays an important role in affecting the performance (coating uniformity and powder transfer efficiency) of powder deposition. Being prone to aggregate in powder source, nanosized ZnO powders form the deposition more in clusters than in discrete particles. The clusters in the deposited submicron MoS_2 and micron ZnO are not as many as that in nanosized ZnO.

(2) When other processing parameters are held constant, deposition of nanosized ZnO particles saturates at electrical potential of –60 kV. Prior to –60 kV, the deposition thickness increases as the potentials go up. The maximum peak-to-valley surface roughness height decreases slightly with increase of electrical potential from 30 to 75 kV, but significantly with further increase of the potential to 100kV due to the effective breakdown of the clusters from high voltage;

(3) The size of the clusters, and thus the pores, becomes small, and the distribution of the clusters is more uniform with the increase of the potentials.

List of figure captions

Figure 1. A schematic of the ESC system (corona charging).

Figure 2. SEM images of: (a) coating of submicron MoS_2 on 1045 steel; (b) submicron MoS_2 particles in coating formation; (c) coating of micron ZnO on WC-Co; (d) micron ZnO in coating formation; (e) coating of nano ZnO on WC-Co; and (f) ZnO clusters in coating formation.

Figure 3. Deposition thickness variation with electrical potential applied at the electrode.

Figure 4. Variation of maximum peak-to-valley roughness height with electrical potential.

Figure 5. SEM pictures showing the clusters of deposited nano ZnO powder at (a) -33kV; (b) –45 kV; (c) –60 kV; (d) –75 kV; and (e) –100kV.

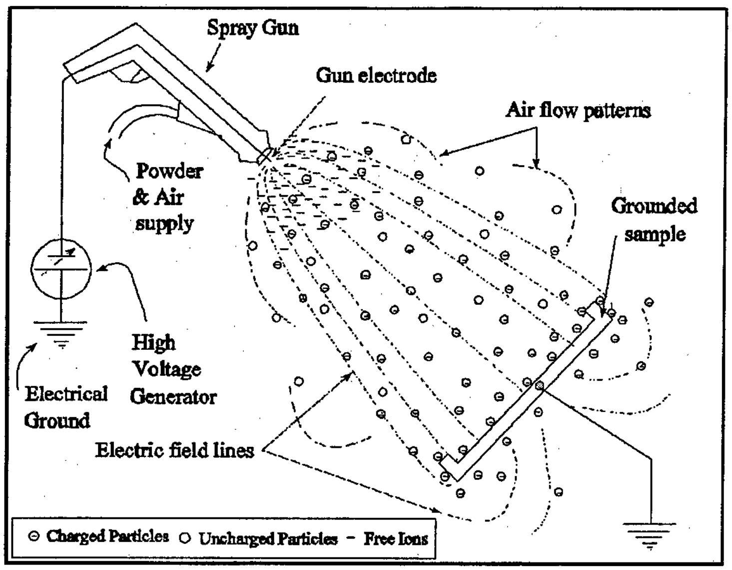

Spray Gun
Gun electrode
Air flow patterns
Powder & Air supply
Grounded sample
High Voltage Generator
Electrical Ground
Electric field lines
Charged Particles
Uncharged Particles
Free Ions

Figure 1

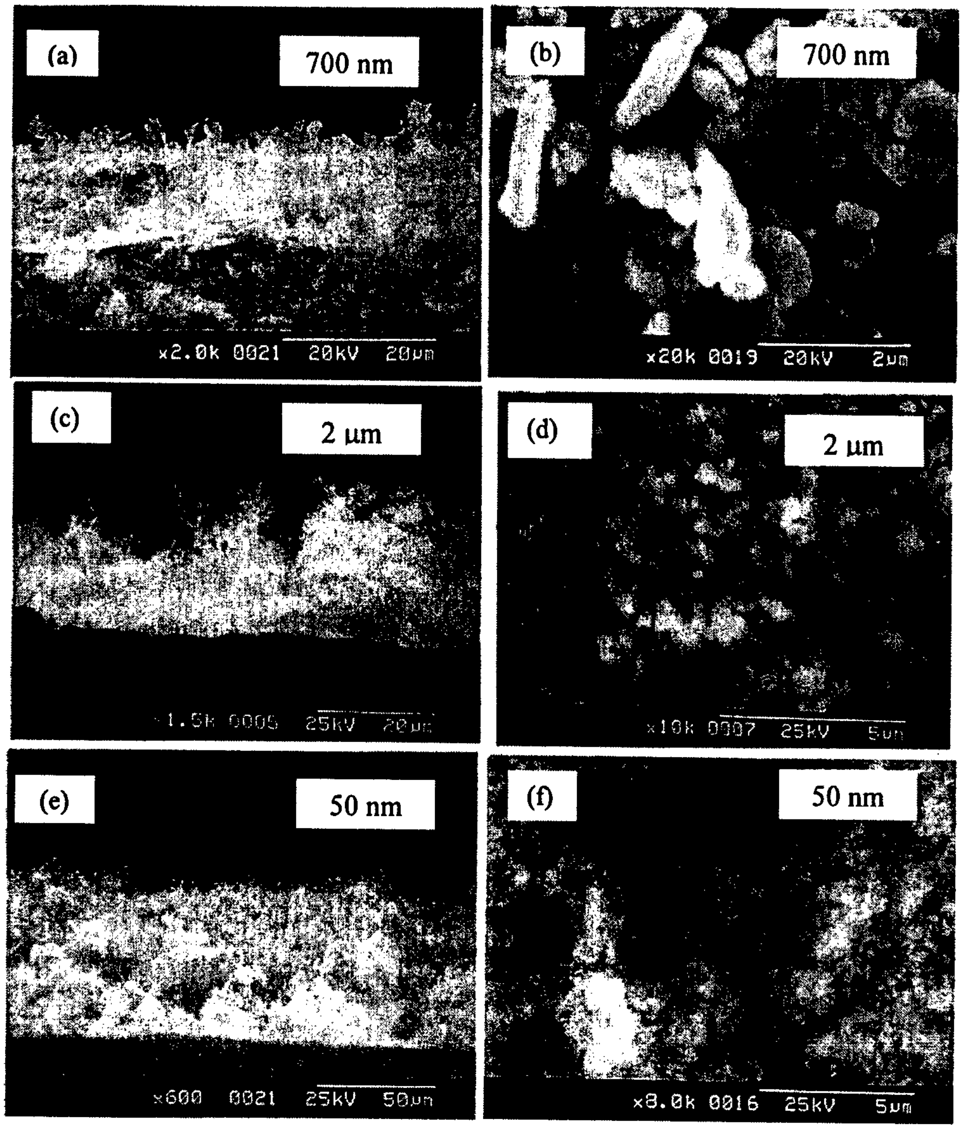

(a)
700 nm
x2.0k 0021 20kV 20μm
(b)
700 nm
x20k 0019 20kV 2μm
(c)
2 μm
25kV 20μm
(d)
2 μm
x10k 0007 25kV 5μm
(e)
50 nm
x600 0021 25kV 50μm
(f)
50 nm
x8.0k 0016 25kV 5μm

Figure 2

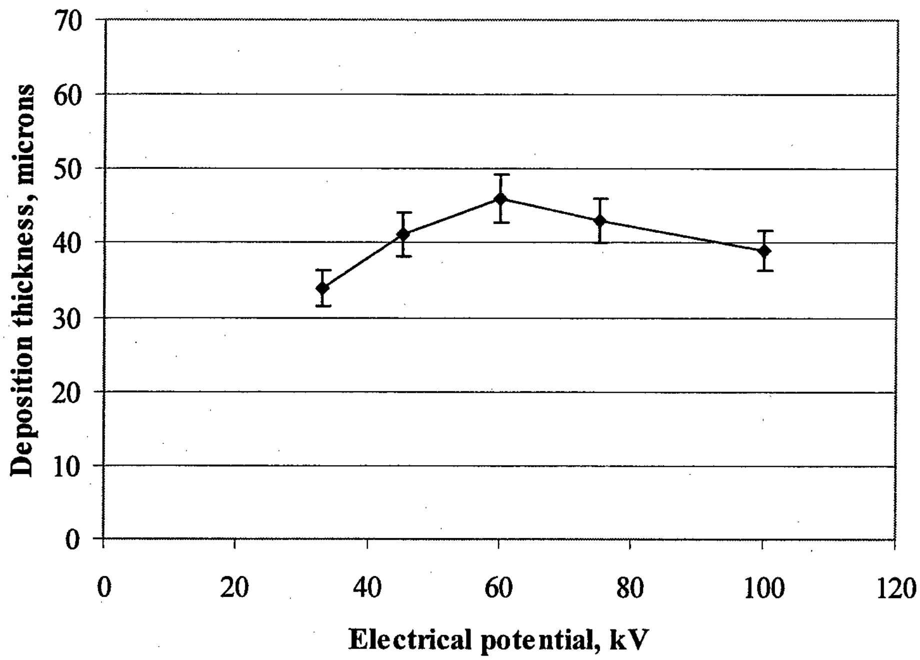

70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
Deposition thickness, microns
Electrical potential, kV

Figure 3

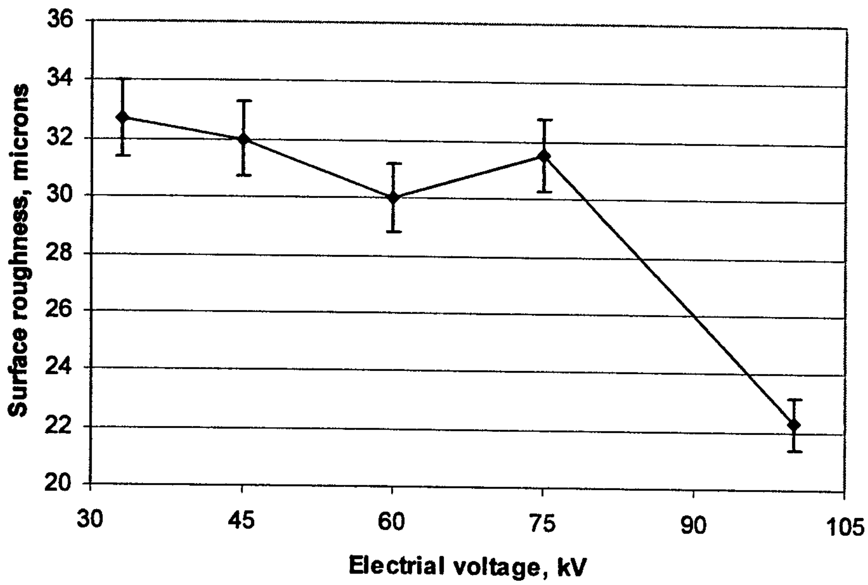
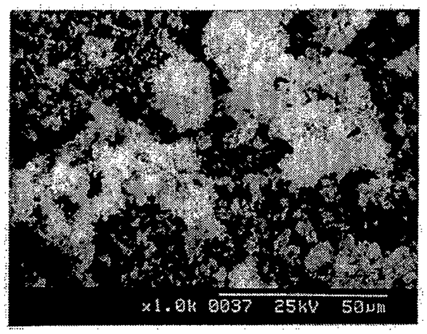
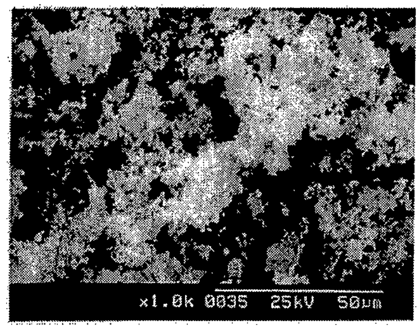

36
34
32
30
28
26
24
22
20
Surface roughness, microns
30
45
60
75
90
105
Electrial voltage, kV

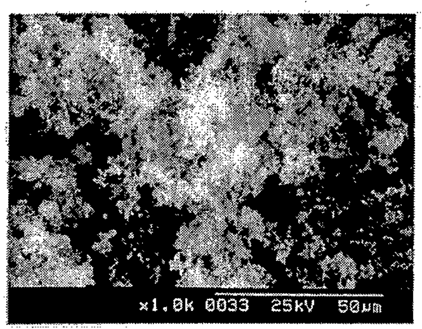
Figure 4



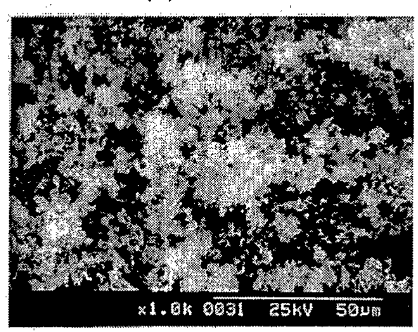
(a)



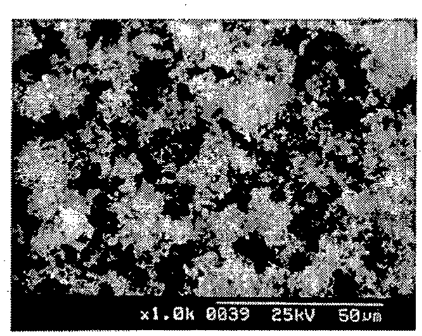
(b)



(c)



(d)



(e)

Figure 5

Performance Report

Personal Supported:

Dr. Bhat, MEEG, U of A (for CVI development)
Mr. Gilbert Nyandoto, MEPH, U of A (M.S., complete support; currently in Ph.D. program*)
Dr. Jiang Wenping, MEEG, U of A (Post-doctoral fellow)
A. More, MEEG, U of A (M.S., partial support)
Ajit D., MEEG, U of A (Apprenticeship, partial support; currently in the PhD. Program*)

* The experience and education achieved through this project have encouraged these students to go for higher education (Ph.D.).

Publications:

"Nano particle Solid Lubricant Composite Coating using ESC Technique", Wenping Jiang, Ajay P. Malshe and W.D. Brown, Surface Coating and Technologies (2004, in press).

"Nano structured Coatings for Machining and Wear-Resistant Applications," Ajay P.Malshe, Wenping Jiang and Ajit R.Dhamdhere, Journal of Materials, pp. 28, September 2002.

This project extensively established coating infrastructure for the above described novel technologies and thus, there are a relatively moderate number of publications.

Interactions/Transitions:

a. Participation/presentations at meetings, conferences, seminar, etc.

"Nano particle Solid Lubricant Composite Coating using ESC Technique", Wenping Jiang, Ajay P. Malshe and W.D. Brown, International Conference on Thin Films and Metallurgical Coatings, San Diego, CA (April 2003; presentation).

b. Consultation and advisory functions to other agencies and laboratories

Periodic technical meetings and telephonic consultations (APM, point-of-contact, U of A) are held with Dr. Jeff Zabinski, Director, Surface Science, who is our point-of-collaboration at WPAFB. Multiple conversations and consultations have further expanded the finds of this project to (1) develop a multi-axes nanoparticle coating system and (2) application of this technology for self-healing corrosion resistant coating for aging aircrafts (proposal pending).

c. Transitions

The inventions in this project are partially at the foundation of the realization of a spin off company and related technology scale-up transfer with this new company, NanoMech LLC in Arkansas. This company is currently working on commercialization of this technology.

New Discoveries, inventions, or patent disclosures:

"Over coating of particles and deposition of single and multiphase combinatorial electrostatic coating (ESC) and follow up processing," Ajay P. Malshe and W.D. Brown (patent disclosure pending, 2001-2003).

Honors/Awards:

♦ Service recognition, Electrical and Communication Systems (ECS), National Science Foundation (NSF), 2002.

♦ College of Engineering, 2001-2002 University of Arkansas, Outstanding Researcher Award, Mechanical Engineering, UA.

Appendix

(ESC coating process guidelines using hard cBN powder as a testbed)

ESC Operation Guidelines

Content

1. Chapter 1 1

Electrostatic Spray Coating : Basic Process

2. Chapter 2 5

Electrostatic Spray Coating : Experimental Setup

3. Chapter 3 9

ESC cBN : Coating Characteristics and Process Parameters

4. Chapter 4 12

ESC cBN : Coating Procedure

5. Chapter 5 13

ESC cBN : Process Optimization - Prior Results

Chapter 1

Electrostatic Spray Coating : Basic Process

1.1 Introduction :

The set goal of this was successfully achieved by way of design and development of a novel combinatorial technology to form first ever cBN-TiN composite coating. The hybrid technology is a two-step process comprising of electrostatic spray coating (ESC) and chemical vapor infiltration (CVI). The ESC methodology, which is borrowed from paint technology, was researched at MRL as per the requirements of first ever cBN composite coating. The process of chemical vapor infiltration was investigated in the other past of this project.

Electrostatic spray coating (ESC) is a widely used method in paint industry for applying polymer coatings either to protect the surface or promote aesthetics. The method is commonly applied to various consumer goods, however, is unexplored for applications in ceramic and metal coating industry. Over the time, the worldwide popularity of powder coating technology has enjoyed a steady growth. There are numerous parts and products being powder coated. For example, the appliance industry benefits from powder coating on front and side panels of refrigerators, washer tops and lids, dryer drums, air conditioner cabinets, water heaters, dishwasher racks and cavities of microwave ovens. Powder coating has also replaced porcelain enamel on many washer and dryer parts. One of the driving forces in the massive success has been the continuous improvements in equipments used. The details of ESC process and setup is discussed in the following sections.

1.2 ESC Process :

The electrostatic spray coating involves physical spraying of powder particulate matter on the desired substrates. The powder particles commonly used in ESC are electrically insulating in nature and can carry the static charge over a distance of a few tens of centimeters. Also, the particles can be provided in the form of powder which is easily available from powder industry.

The desired powder is fed to the electrostatic spray gun by common means like powder hopper, powder feeder etc. The electrostatic charge is transferred or generated on powder particles by applying high voltage to spray gun electrode (typically a few tens of kilo volts - kV) with respect to the electrically grounded objects. The quality of the ESC coating depends on the effective charging of the sprayed particles. Depending on the available equipment and the powder material, there exist two most common methods for particle charging in ESC technology, namely corona charging and tribo electric charging. The charged powder particles thus generated follow the electric field lines toward the grounded objects and gets coated. Here, the electric field generated acts as a guiding agent for the charged particles onto their journey to the grounded substrate surface. Since the electric field lines terminate over the entire surface area of the grounded sample, particles of a given powder material can be dispersed uniformly on a given surface. Thus, the spray coating is formed all over the object surface.

1.3 Basics of ESC :

The basic process in electrostatic coating technique is the process of corona charging. Corona charging is a high-voltage process in which gaseous ions transfer their charge from the charging electrode in the electrostatic gun to the powder particles. The schematic explaining the charging phenomena and electrostatic spray coating process is shown in the figure 1. If a particle is caused to travel through a region of ionized gas in which the charge is predominantly of one polarity or the other, the particle acquires a net nonzero charge. This would depend on the following factors : approaching velocity of the ions, the ion mass, ion charge, particle radius and particle charge. An electric field, E, will be created as a result of the surface charge, the field at the interface between the particle and its surroundings. With continued increase in charge collection, E will increase until

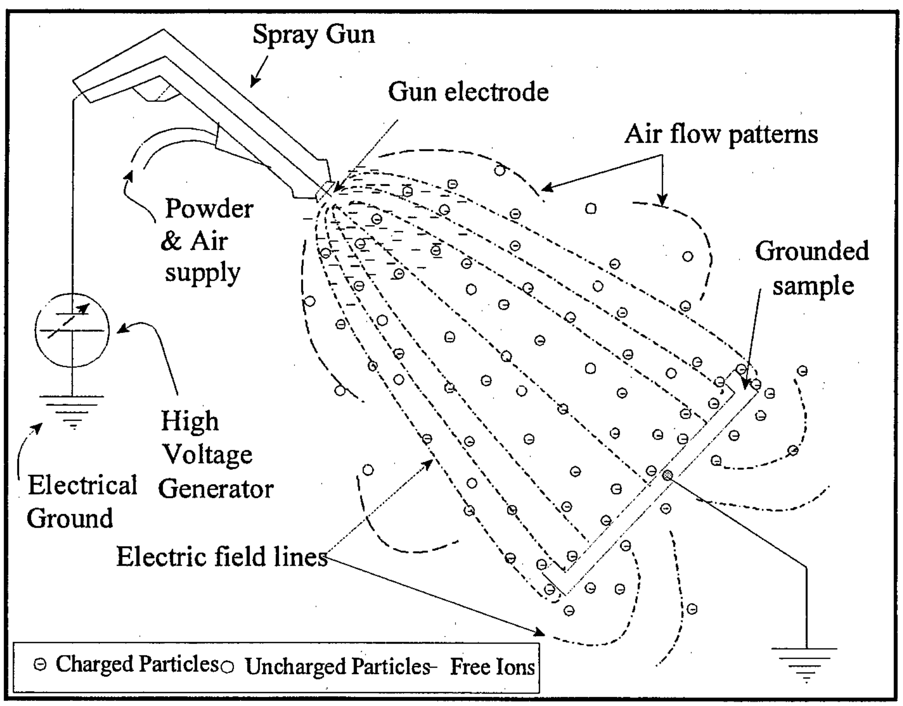


Figure 1: Principle of corona charging and electrostatic spray coating.

a value is reached which corresponds to a situation where no further transport of ions onto the particle will be possible. This limiting value of surface charge, known as Pauthenier limit, can be expressed mathematically as,

$Q = 4 \pi \epsilon_o a^2 B E$ where $B = 1 + 2(\epsilon_r - 1)/(\epsilon_r + 1)$

ϵ_o = permittivity of free space

$= 8.854 \times 10^{-12}$ F/m,

ϵ_r = relative permittivity of powder particle,

a = radius of particle, and

E = electric field for which particle is subjected to;

For a spherical particle of radius "a", its mass "m" can be calculated as,

$m = (4/3) \pi a^3 \rho$, where ρ = particle density.

The ratio of charge to mass i.e. q/m will be calculated from the following relation

$$q/m = Q/m$$
$$= (4 \pi \epsilon_o a^2 B E)/((4/3) \pi a^3 \rho)$$
$$= 3 \epsilon_o B E / a\rho$$

The charging electrode is held at a very high negative potential, requiring a power supply rated from 80 to 100 kV. At these high voltages, the electric field is strong enough to cause the breakdown of the air molecules into anions and electrons and to form a corona - a glowing blue region the size of a fine match head. Electrons are accelerated from the corona region by the strong field and collide with oxygen atoms outside the corona, forming oxygen ions. These ions in turn collide with the powder particles, transferring one electron to the particle during each collision. Thousands of collisions take place when a particle is charged. A fully charged particle with a diameter of 40 μm will carry approximately 500,000 electrons. As the particle charges, however, it generates a stronger and stronger field of it own. This field acts to repel ions from the particle, making each successive charge harder to acquire. It is easiest to charge a particle in the region of highest field strength near the electrode. Since the particles are passing through this region rather quickly, however, there is not enough time to charge a particle to its theoretical limit. It is therefore desirable to keep the velocities in the charging region as low as possible; this allows more time for particles to be charged. Also, it is important that particles be well spread out in the charging region so that ions have an opportunity to reach the individual particles. Further, the geometry of the charging electrode and the nozzle of the gun must be arranged so that the ion current passes through the powder cloud as the current makes its way to the grounded substrates. Finally, the electrode must be in the direct line of sight of the ground to maintain the ion current, the electric field between the charging electrode and the ground must not be impeded or blocked by any structure in the gun.

The strong field promotes effective charging and helps to direct the charged powder particles efficiently to the substrates. Generally, the strong field and excess ion current produced by corona guns work to create a self-limiting action that helps produce thin (~ 25 μm) and highly uniform coating on the substrates.

Chapter 2

Electrostatic Spray Coating : Experimental Setup

2.1 Introduction :

The ESC setup has been through different stages of development in order to achieve better coating quality, as well as the ability to handle a larger number of samples with ease of operation. The existing electrostatic coating setup used for powder coating consists of the following parts: spray gun and its controller, powder delivery / feeder, air supply, coating chamber, and most importantly the powder recovery unit. A photograph and a line diagram of the electrostatic coating setup are shown in Figures 2 and 3, respectively, displaying the listed setup parts. The brief description and operation of each of the components in electrostatic coating setup are detailed in the following sections.

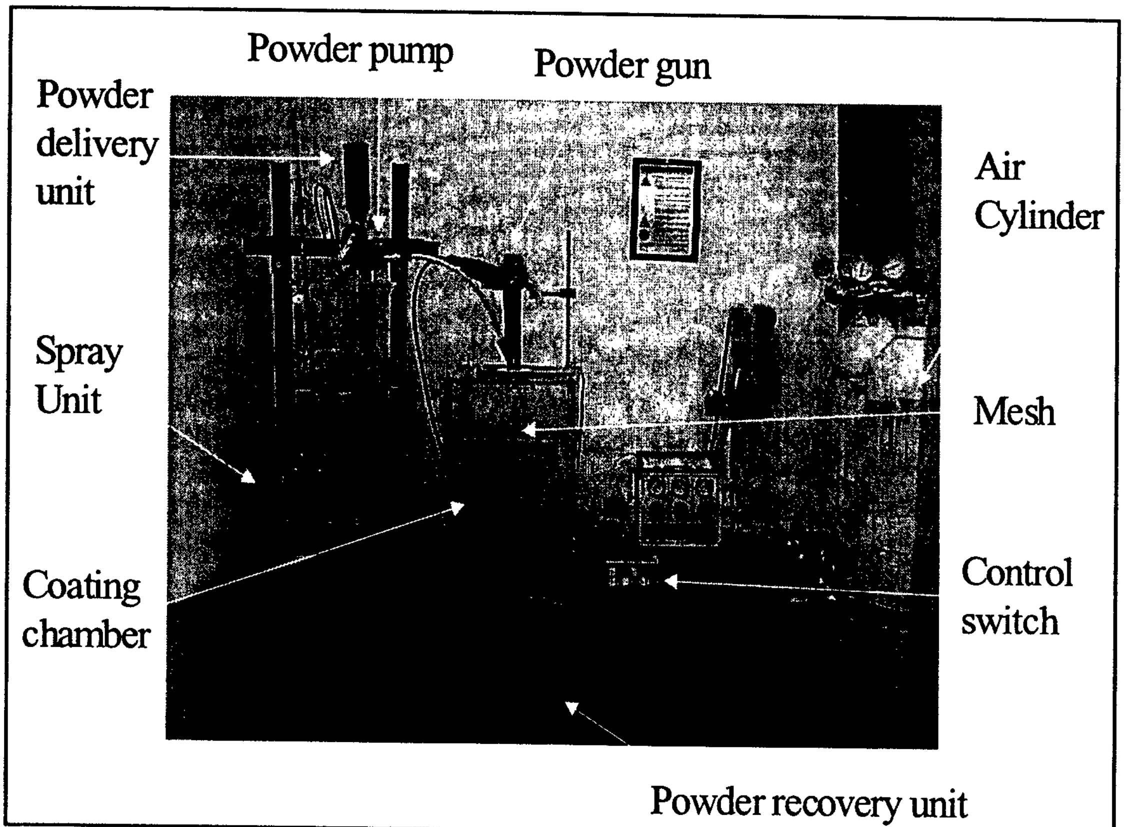


Figure 2 : Electrostatic spray coating setup showing the individual parts.

2.2 ESC setup parts :

2.2a Powder delivery unit :

The meaningful ESC particles coating formation is incomplete without having an efficient delivery system and appropriate quantification. Thus, it was necessary to set up suitable equipment capable of efficiently delivering the particles powder to the spray gun. Different methods with varied gadgets were searched and experimented for the required delivery system without much success in consistency. A simple device known as Uniflow powder measure (UPM) is found to be useful for consistent delivery of powder in ESC. It consists of a powder hopper and an adjustable cylinder which houses an adjustment screw for setting the right quantity of powder. The consistency of the powder delivery from this device has been tested by conducting a series of consistency trials.

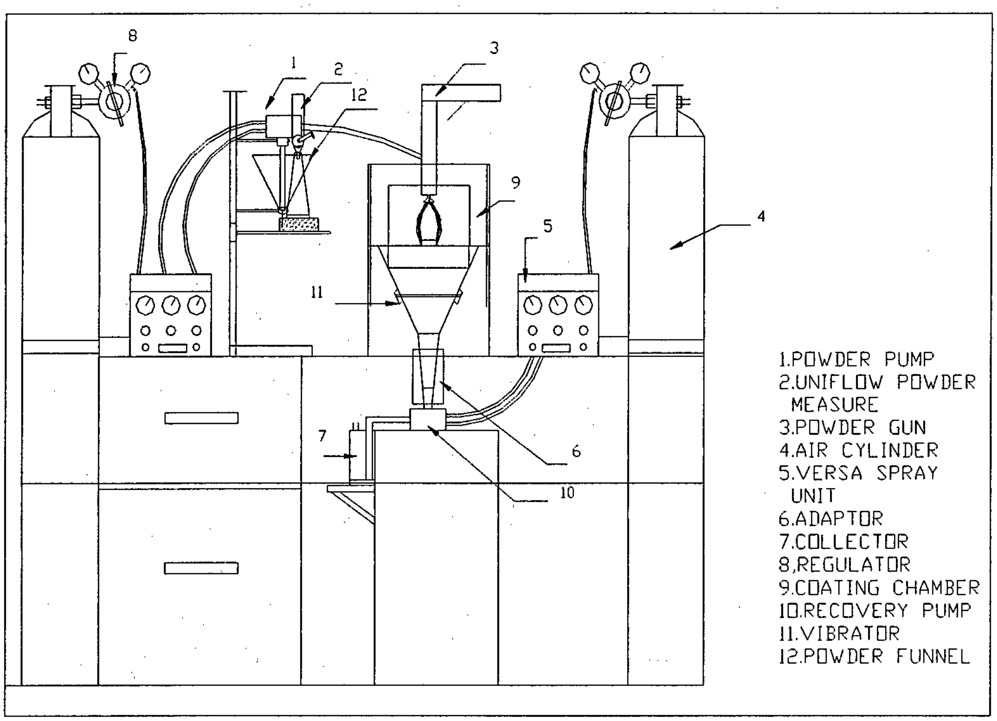


Figure 3 : Schematic of ESC setup.

The desired quantity of powder discharge is calibrated separately with respect the adjustment screw on UPM. Once the desired quantity of powder is set, it can be coupled to the powder funnel from where the powder can be fed to the system. During each operation of the UPM, the set quantity of powder is delivered and is collected in the powder feed funnel. The set amount of powder is then fed to the electrostatic gun for coating, using the venturi effect from powder pump.

2.2b Coating chamber :

The coating chamber is a ESC process enclosure housing the samples to be coated along with sample holding mesh / fixture and powder recovery components. It is made of plexiglass, which is electrically insulating in nature. Typically, the samples are placed on the mesh inside the coating chamber or sample holding fixture. The mesh / sample holding fixture is supported by four metal rods placed at four corners of the coating chamber so that the mesh height can be adjusted in vertical direction. The distance between the samples and the spray gun electrode can be adjusted to the desired distance by sliding the mesh. The coating chamber exhaust (bottom end) is in the form of a powder funnel to expunge the unused powder into the powder recovery system. The walls of the chamber serve as a boundary for the powder spray cloud limited to the region covering the tool samples placed on the mesh support.

2.2c Powder feed pump :

For ESC coating, the set amount of powder is discharged by UPM into the powder funnel. This powder is then sprayed onto the carbide samples in the presence of electrostatic field. Here, the powder feed pump serves as the means for delivering the powder from powder funnel to the powder spray gun. It comprises of a aluminum metal housing with ports for air flow, powder suction, and powder delivery. An adaptor / plastic pick up tube assembly is connected to the powder suction port, which sucks the powder from the powder feeder funnel. The powder pump require two types of air flow for it to perform its function namely, flow rate air and atomizing air. The flow rate air sucks the fluidized powder from the feeder funnel by creating a venturi effect, and forces the powder and air mixture through the feed tube to the ESC spray gun. The atomizing air helps in dilution and atomizing the powder stream as it exits the powder pump. The operating range used for the respective air flows is given in the operating manual for the Versa Spray equipment.

2.2d ESC spray gun :

The ESC powder spray gun is the heart of electrostatic coating set up. The prime function of the gun is to induce electrostatic charge on powder particles as they exist from the gun, and to spray these charged particles onto the grounded substrate with the help of air flow. The spray gun accommodates the charging electronics and the charging electrode at the exit end. The charging electrode is surrounded by a conical nozzle and a deflector. The spatial arrangement of nozzle and deflector allow the air flowing through nozzle to go around the spray electrode to prevent powder from collecting on the electrode itself. The powder pump pumps the powder and air mixture through the spray gun and around the spray nozzle, where the powder particles are charged. The operation of ESC spray gun is controlled by the control unit, which is assisted by the powder feed pump.

2.2e Control unit :

The control unit provides user with a control over the air pressures to the powder feed pump as well as the spray gun electrode voltage. The electrode voltage, which is required for charging the powder particles, is one of the important control factors determining the ESC coating quality. A push-pull rotary switch and potentiometer allow the operator to choose between two different modes (kV / AFC) and to set the output voltage. The speed and manner in which the powder flows through the gun can be controlled by adjusting the settings of the flow rate and atomizing air.

2.2f Powder recovery unit :

The powder recovery unit helps in collecting the unused powder during ESC operation. This unit consists of a separate control unit, powder collecting funnels, and a separate low feed powder pump. The powder particles which are sprayed but not collected on any grounded surface, so called "unused powder", is collected in the powder funnel just below the sample holding fixture in the coating chamber. By using the inverse principle of a powder feed pump, the powder particles are forced in a powder collector. Application of this unit ensures the economical use of powder. Several trials conducted on the laboratory scale unit have shown about 95% recovery of the powder.

Chapter 3

ESC Coating Characteristics and Process Parameters

3.1 ESC coating characteristics and control :

The electrostatic spray coating has found to be the efficient process to form a uniform powder coating of cubic boron nitride (cBN) on carbide tool inserts. cBN is an example, the process is also tested for solid lubricant nano particles. An ESC cBN powder coating can be characterized by various parameters namely coating thickness, uniformity, porosity, and the particle clusters. Coating thickness is the thickness of cBN particle layer that is coated on all the edges as well as the corners of the sample (refer figure 4). Coating uniformity can be defined in terms of the coating thickness over the entire surface of the substrate. Higher the variation in coating thickness, lower is the uniformity of the coating. Cluster of particles is defined as a large group of individual cBN particles adhering together in the form a clump. Clump formation is mainly due to the difference in the average size of individual particles. With a differing size of particles, the effective charge on it varies thereby leading to the formation of large chunk of particles.

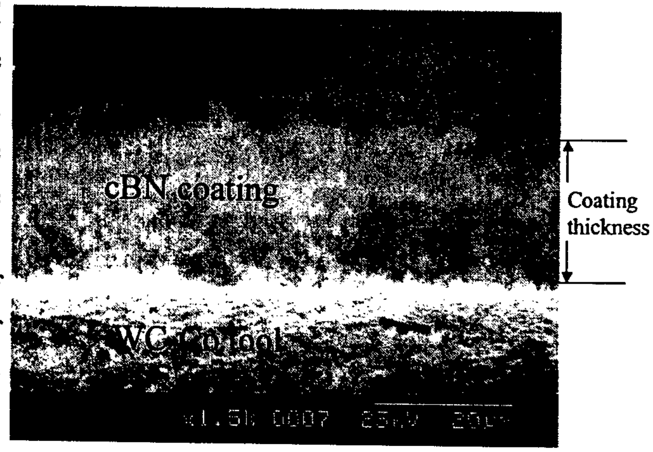


Figure 4: ESC cBN coating.

In order to obtain a good quality cBN coating, it is essential to set ESC process parameters at the optimum levels. The important ones in the list are spray gun electrode-sample distance, spray gun potential and the air flows. For example, the particle clustering tendency can be minimized by setting optimum for air pressure as well gun electrode-substrate distance. When the electrode-sample distance is higher, the particles have to travel more and hence tend to separate out by the influence of air pressure. The quality of the ESC coating will differ drastically depending upon the settings of the operating parameters.

3.2 ESC operating parameters :

3.2a Spray gun electrode-substrate distance :

This is an important parameter and plays a crucial role in achieving a uniform and cluster less powder coating. It is the separation distance between the spray gun pin electrode and the tool insert surface. When the powder is sprayed from the ESC gun, the sprayed charged particles exist the gun in the form of an envelope, which is approximately in the form of a divergent torch light. The coating span is defined as the distance between extreme points of the coating spray envelope at a given separation of electrode-substrate distance. As with a divergent torch light, for smaller electrode-substrate distances, the coating span is smaller. The coating span definition can be restated as the maximum distance on the tool holding mesh within which the extremely separated substrates displays a desired coating thickness.

For a smaller distance of separation, the resistance of the surrounding air as seen by the applied gun potential is less, which leads to the increase in the discharge current for the gun electrode. When the discharge current is high, the discharge voltage will be low, which, in turn, affects the effective charging of the particles. Hence, a higher separation distance is preferred, typically ranging between 80 mm - 180 mm, depending upon the application.

3.2b Gun voltage :

The applied spray gun voltage results in the effective charging of the particles. When an electrostatic potential of the order typically used in ESC process is applied to the gun electrode, an electrical breakdown of air occurs surrounding the spray gun pin electrode resulting in the formation of ions. Under the effect of applied electric potential, these ions drifts away from the electrode, forming a corona region. The flow of free ions from the corona needle is called as the discharge current and it is this current that is responsible for the charging of the powder particles. The discharge current is be of the order of few μA. The level of the discharge current is controlled by the discharge voltage and the distance between the substrate and the electrode. As explained before, if the discharge current is high, the discharge voltage will be low, which, in turn, affects the effective charging of the particles. Hence the discharge current should be kept at a normal level. The typical value of the spray gun voltage is 60 kV. If the gun voltage is too low, it results in the poor charging of the particles, and if the gun voltage is set at a very high value, it leads to coating defects. So the gun voltage values in the range between 40- 60 kV are useful.

3.2c Air pressure :

Powder spray guns use air to assist powder transport during spray coating. The main air pressure at the cylinder pressure regulator is distributed inside the spray gun control unit into flow rate and atomizing air. The details of the control unit and related information can be found in the manufacturers operating manual. The main air pressure is responsible for the travel of powder particles from the powder feed funnel, through the powder pump and out to the electrostatic spray gun nozzle. When the main air pressure is changed, there is a change in the flow pattern of the particles affecting the coating uniformity. The flow pattern being either laminar or turbulent, depending upon the regulated air pressure. The air pressure should be maintained at a value so as to cause the breakage of clumps into smaller particles and to provide free flow of powder from the powder feed pump to the gun. The recommended main air pressure is 30 psi. This value is also found to be optimum in earlier experiments.

The effect of these important parameters on the cBN coating quality can be studied, as discussed above, by varying one of the factors keeping the rest constant. However, it is important to note that these factors may interact with each other, providing a different result than expected. For example, it is possible that the variation in main air pressure for a given value of spray gun voltage, may lead to the variation in the gun voltage. This may result through the change in discharge current which itself is affected by the changes in the amount of flowing powder and the availability of gaseous matter around the spray gun exit, thereby affecting the gun voltage.

Chapter 4

ESC of cBN : Coating Procedure

4.1 Electrostatic spray coating procedure :

The working principle of ESC process as well as its operation is simple.

The distance of sample holding mesh or fixture is set at the required value from the spray gun electrode. The samples are thoroughly cleaned just before the actual coating. It is done in steps, using ultrasonic bath, by first cleaning the inserts with the help of soap water and followed by cleaning in acetone. The cleaning procedure helps to remove any surface impurities like dust particles or oil residue on the inert surfaces. The cleaned substrates are then placed on an electrically-grounded sample holding metal mesh platform or metal fixture.

Before the actual spray process, the powder is heated under IR lamp for about 2 - 5 minutes to avoid any clustering due to moisture in the powder. This powder is transferred to the powder delivery device, UPM. UPM device must have been set to deliver the required amount of cBN powder. One cycle of UPM delivers the set amount of powder in powder feed funnel. This is the basic preparation of ESC process.

Switching the gun control unit forces the air to pass in the powder pump along with the high voltage applied to spray gun electrode. When the air is passed through the low feed powder pump, powder particles are sucked from the powder feeding funnel due to venturi effect and are fed to the electrostatic spray gun. These particles, upon their passage by the high potential spray gun electrode, are charged and sprayed onto the samples placed on the mesh. The unused powder is collected in the powder collector with the help of the powder recovery system. The actual process of the spray coating process lasts for about few seconds.

The cBN coating thickness is analyzed using the scanning electron microscope. One of the sides of the sample is carefully scrapped with a sharp knife edge blade so as to expose the interface between the ESC cBN particle overlayer and the carbide tool insert. A representative SEM picture of this interface is shown in figure 4.

Chapter 5

ESC of cBN : Process optimization - Prior Results

5.1 ESC Process Optimization :

As discussed earlier, there exists a number of available operating parameters and the powder coating characteristics. An optimization technique was employed to find out an optimum condition for these different parameters amongst the various possible values. Taguchi's statistical method was exercised in ESC process to determine the optimum condition which will reliably give desired cBN coating qualities. The availability of such a condition will help in obtaining desired quality ESC cBN powder coating as well as batch process it in manufacturing. The results of optimization experiments are given below.

5.1a Optimum set of conditions -

Following table gives the set of optimum values of the parameters. The other parameters which were held constant are the particle size (< 2 um), batch size (4), and orientation of the sample tools. The conditions given in the table are found to achieve an average coating thickness of approximately 22 um.

Sr.No.	Operating parameter	Optimum Value
1	Spray gun electrode substrate	150 mm
2	Spray gun voltage	60 kV
3	Main air pressure	30 psi

5.1b Coating uniformity -

Under the optimum conditions, the maximum variation in coating thickness on a given sample was found to be 4 microns whereas it was about 5 microns between two different samples. The variation can be attributed to the inherent variation as well as the noise factors, like humidity, of the process.

5.1c Pore Size -

This is a void space between adjacent particles. The pore size was found at 4 different locations of the given coating, under optimum conditions, with an average pore size of about 2 um. The measurements were done on using a SEM picture (refer figure 5) of the coating at 8000x (area of 15 um X 12 um).

5.1d Particle Cluster -

The average cluster size was determined to be about 15 microns measured over an area of 100 µm X 75 um.

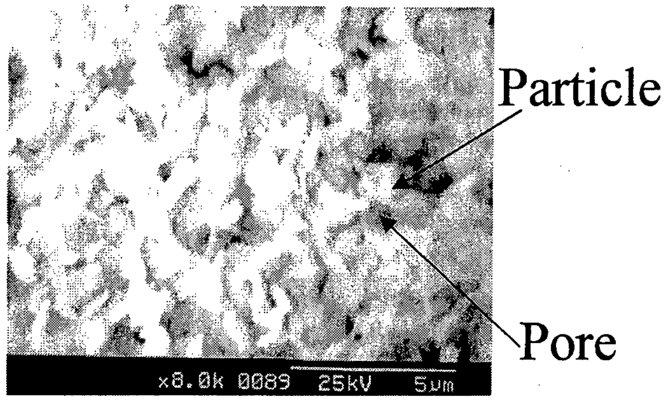


Figure 5: Particulate and Pores in an ESC cBN coating.