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THE DEVELOPMENT AND EVALUATION OF REMOVABLE THIN FILM
COATING TECHNOLOGY FOR THE ABATEMENT AND MITIGATION OF
HAZARDOUS PARTICULATES IN AN OCCUPATIONAL SETTING

by

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A dissertation submitted to the School of Public Health

University of Medicine and Dentistry of New Jersey and the

Graduate School-New Brunswick

Rutgers, The State University of New Jersey

in partial fulfillment of the requirements

for the degree of

Doctor of Philosophy

Graduate Program in Public Health

written under the direction of

Mark Gregory Robson, Ph.D., M.P.H.

And approved by

New Brunswick, New Jersey

October, 2007

ABSTRACT OF THE DISSERTATION

The Development and Evaluation of Removable Thin Film Coating Technology for the Abatement and Mitigation of Hazardous Particulates in an Occupational Setting

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This dissertation evaluates a new decontamination technique for the mitigation and abatement of hazardous particulates. Currently, traditional decontamination methods, such as, the wet method and the use of vacuums and brooms are used to clean facilities and equipment. These are time consuming, resulting in prolong exposure to the contaminant and may generate airborne hazards. A new technique using removable thin film coating technology, a loosely adhered paint-like coating was tested as a viable alternative to traditional methods. Tests conducted at three different sites on different hazardous metals resulted in reducing the initial levels of the metals by 90 percent and had an average reduction of one magnitude after one application of the coating. The paired t-tests performed for each metal demonstrated that there was a statistically significant reduction in concentration after the use of the removable thin film coating: lead ($p = 0.03$), beryllium ($p = 0.05$) aluminum ($p = 0.006$), iron ($p=0.0001$), and copper ($p=0.004$). A Kendall Tau correlation coefficient confirmed that there was a positive

correlation between the initial levels of contamination and the removal efficiency for all of the metals at each of the three sites.

Qualitative tests demonstrated that the coating reduced the amount of visible luminescent dust from various surfaces and that it worked well as a preventative method, protecting clean areas from becoming contaminated. These tests also exposed a limitation of the coating. It could not migrate into the minute scratches on the surface substrates. The use of a scanning electron microscope (SEM) and calibrated carbon dust supported the previous findings with a statistically significant ($p=0.00007$) removal of carbon dust from the surfaces substrates. The SEM also revealed that wherever there were large clusters of carbon dust, the coating would tear and remain on the sample surface.

To eliminate these issues two different methods were tested. First, Kevlar™ fibers were added to improve the strength of the coating. Next, the use of an engineered textile, saturated with the coating, was tested. This appeared to eliminate the issue of removing contaminants from minute surface scratches and improved the removal process of the coating.

Acknowledgement

Though only my name appears on the cover of this dissertation, a great many people have contributed to its production. I owe my gratitude to all the people who have made this dissertation possible.

I would like to acknowledge many people for helping me during my doctoral work. I would especially like to thank my advisor, Mark Robson, PhD for his generous time and commitment. Not only was he readily available to me, as he so generously is for all of his students, but he always read and responded, with great patience, to all of my questions, concerns, and numerous emails.

I am also very grateful for having an exceptional doctoral committee and wish to thank Philip Efthimion, PhD, Michael Gochfeld, MD, PhD, and Jim Zhang, PhD, M.S. for their continual support and encouragement.

I owe a special note of gratitude to Charlie Gentile, Princeton University, for supporting and believing in my ideas about a new way to clean up hazardous particulates and for assisting and helping me find ways around, through, and over the numerous bureaucratic obstacles that I continuously encountered.

I am extremely grateful for the assistance, generosity, and advice I received from Lloyd Ciebiera and the Princeton Plasma Physics Laboratory managed and run by Princeton

University. Their assistance made it possible for me to complete the research and development of the Removable Thin Film Coating Technology.

Finally, I'd like to thank my family. To my parents, who now know more than they ever wanted to about cleaning up hazardous particulates, thank you so much for your continuous support and belief in me and that one day I would actually finish. I'm grateful to my brother for his encouragement and enthusiasm.

This research was funded by Princeton Plasma Physics Laboratory managed and run by Princeton University: Laboratory Program Directors Account and by the University of Medicine and Dentistry. I extend thanks to Lew Meixler for doing an excellent job with getting the two universities and the Department of Energy to agree on a contract.

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Chapter 1

Introduction

Background

There are many industrial processes that contaminate facilities and equipment with hazardous particulates. This can be an expensive problem, usually necessitating extensive decontamination procedures and discarding and replacing equipment. The presence of hazardous materials, in fine ($<10\mu\text{m}$) or coarse ($>10\mu\text{m}$) particulate matter, also presents industrial hygiene issues in the work place. The fine dusts are of respirable size and may result in upper respiratory and pulmonary issues. The coarser dust particles have the potential to cause irritation to the skin, eyes, nose, and throat. There is the possibility of employees being over exposed to these hazardous particulates resulting in illness and even death. Most industrial processes that include machining of materials, such as, beryllium and beryllium compounds require that the areas be decontaminated on a regular basis.

Removable thin film coating technology has been used to decontaminate commercial nuclear facilities since the early 1980s^[1]. The removable thin film coating is a loosely adhered paint-like coating that decontaminates radioactive equipment, prevents contamination and fixes contamination in place^[2]. Industry has limited the use of removable thin film coating for radioactive contamination only, therefore ignoring the possibility of using this coating on other particulate contaminants, such as heavy metals.

The removable thin film coating has the potential to act as a proactive measure in protecting areas that are designated as clean from becoming contaminated.

Currently the methods of decontamination used for hazardous particulates include the wet method, the high efficiency particulate vacuum, or sticky cloths^[3]. These methods of decontamination generate a lot of hazardous waste which has to be specially disposed of. Not only is this process expensive and time consuming, but it must be done on a regular basis, increasing the potential for over exposure of employees to the hazardous particulate. While conducting the research for this dissertation to prove that this method was a more efficient technique for the abatement of hazardous contaminants, an opportunity arose that also demonstrated that this technique was more effective than the traditional methods. Effectiveness was defined as removing enough lead dust to bring the levels below the HUD guidelines, using the least amount of person- hours, and producing the least amount of waste to be disposed. Initially during the decontamination and decommissioning of the Tokamak Fusion Test Reactor at the Princeton Plasma Physics Laboratory the workers decontaminated the lead oxide dust from the different surfaces by using a high efficiency vacuum followed by numerous cleanings with water. This process took over 3,000 person hours, the surfaces were still above the HUD guidelines, and there were numerous 55 gallon drums of hazardous liquid waste to be disposed of. Next the removable thin film coating technique was tested. This process took 128 person hours and after two applications of the coatings the lead levels were below the HUD guidelines. This technique only generated one 55 gallon drum of solid waste.

A study conducted by the Department of Energy found the removable thin film coating to be a more effective decontamination method for radioactive contamination than the traditional steam vacuum cleaning method that is normally used, supporting the findings of this dissertation. The results of this study demonstrated that the removable thin film coating technology was cost efficient in labor^[4]. It only took two mechanics and one health physicist 4.35 hours to complete the job using the removable thin film coating technology versus the 70.4 hours it took for the same number of workers to complete the job using the steam vacuum cleaning technology. The steam vacuum cleaning technology was less expensive in the equipment and waste disposal areas. However, in the demobilization area the removable thin film coating technology cost \$139 with only one hour of clean up versus \$5,503 total cost for 48 hours of clean up^[4]. Overall the removable thin film coating technology had a 33% cost saving over the steam vacuum cleaning technology. This study along with the study conducted at the Princeton Plasma Physics Laboratory further demonstrates the advantages of using the removable thin film technology, not only for radioactive decontamination but for any hazardous particulates.

Overview of the Study

This dissertation is an attempt to address the issue that the Removable Thin Film Coating Technique is more efficient than the traditional methods for hazardous particulate abatement and mitigation. The null hypothesis (H_0) is that the Removable Thin Film Coating Technique is no more efficient than the current wet method in the abatement of hazardous particulates. The alpha level range will be set at 0.05.

The steps that were conducted to reject the null hypothesis include:

1. Develop removable thin film coating technology to adequately decontaminate hazardous particulates from contaminated areas including lead contamination from a nuclear fusion reactor building, beryllium dust from an existing beryllium facility and various metal particulates from an active machine shop.
2. Develop removable thin film coating technology to be used as an engineering control to prevent hazardous particulates from contaminating clean areas.
 - a. Two proof of principle tests were conducted to test the hypothesis. First, the removable thin film coating process was applied to various surfaces that were contaminated with a luminescent dust. These surfaces included: stainless steel, aluminum, galvanized steel, PVC, and Lexan™. The luminescent dust was used as a substitute for any hazardous particulates. To determine the presence of the luminescent dust an ultraviolet light was used. Two sets of each surface sample were made. For the first set, the samples were coated initially with the removable thin film coating, and then the luminescent dust was applied. Next, another layer of the removable thin film coating was applied, sandwiching the dust between the two layers. The second set of samples was first coated with the luminescent dust; then the removable thin film coating was applied. After the coating cured, it was removed from the surface samples. The removable thin film coating peeled off as one entity. Once the coating was removed, the ultraviolet light was used to determine if any luminescent dust remained on the samples.

- b. The second proof of principle test was conducted at a Los Alamos National Laboratory Beryllium Facility. This facility was in the process of being decontaminated and reclassified as a non-beryllium area. The same application method that was used for the luminescent dust was applied to various surfaces. The coating was applied to various surfaces, such as, flat smooth surfaces or non-conforming porous surfaces, to remove the hazard. For each location two areas were blocked, the first block was used to determine the initial beryllium level. The removable thin film coating was applied to the second block and left to cure. After twenty-four hours the coating was removed and the areas tested to determine what beryllium concentration was still present. The effectiveness of this method of removal was assessed.
3. A new process to improve the application and removal of the removable thin film coating was developed. This new process incorporates an engineered textile, which is absorbent and highly porous. First, the textile was saturated with the coating. The thickness of the textile can vary so that it may hold different amounts of the removable thin films. Also, the dimensions of the textile can be chosen so that it may cover part or all of the contaminated area. Once the coated textile was applied to the contaminated area, it was allowed to cure into a solidified mass. After curing, the coated textile containing the hazardous particulates was peeled off the surface for disposal.
4. Test different applications of removable thin film coating to various surfaces and determine if there was any adverse reaction of the coating to surfaces.

5. Determine the overall efficiency of the removable thin film coating as a decontamination technique.

Chapter by Chapter Overview

The next few pages will provide a chapter by chapter summary of this research project. Chapter II; entitled “Evaluation of an Innovative Use of Removable Thin Film Coating Technology for the Abatement of Hazardous Contaminants”, evaluates a new decontamination technique for the mitigation and abatement of hazardous particulates. This chapter discusses how the use of removable thin film coating as a decontamination technique for surface contamination was a more efficient method of decontamination when compared to the traditional decontamination methods used to clean facilities and equipment. To prove this theory the method was tested at three different sites, TFTR Test Cell Basement, Beryllium Machine Shop, and a Metal Machine Shop on different hazardous metals including lead, beryllium, aluminum, iron, and copper. The different tests resulted in demonstrating that one application of the coating reduced the levels of these metals 90% and had an average reduction of one magnitude. The paired t-tests that were performed for each metal demonstrated that there was a statistically significant reduction of the metal after the use of the removable thin film coating as compared to the initial contamination levels. A Kendall Tau correlation coefficient was computed and demonstrated that there was a positive correlation between the initial levels of contamination and the removal efficiency for all the samples taken from different locations on the floor for each of the three sites.

Chapter III entitled “The Use of the Removable Thin Film Coating Technique as an Alternative to Traditional Decontamination Methods to Mitigate and Abate Hazardous Particulates” discusses how using the removable thin film coating technique as an alternative abatement method will increase efficiency, will not generate airborne hazards, decrease costs, and with one application bring the hazardous dust concentrations to acceptable levels. Qualitative tests were performed and demonstrated that the removable thin film coating reduced the amount of visible luminescent dust (a surrogate for hazardous dust) from various surfaces. These tests also indicated that wherever there were minute scratches, the coating did not remove all of the dust. The qualitative tests demonstrated that the coatings worked well as a preventative method. They protected the clean areas from becoming contaminated when exposure to the luminescent dust. Further investigation was conducted using a scanning electron microscope and carbon dust. Overall, the SEM experiment demonstrated that there was a statistically significant ($p=0.00007$) removal of carbon dust (less than 10 μ m in size) from surfaces with crevasses larger than 3 μ m.

Chapter III also discusses some of the limitations of the removable thin film coating technique that was revealed during the use of the SEM and the attempts that were made to resolve these issues. Some of the limitations discovered are, where there were large clusters of carbon dust, the coating would tear and remain on the sample surface. One method to resolve this limitation involved adding Kevlar™ fibers to the removable thin film coating. It was thought that this would increase the strength of the coating and eliminate the coating from tearing when removing large clusters of a contaminant.

Another method tested to improve the removable thin film coating technique was the use of an engineered textile, saturated with the coating. These tests were used to determine if the addition of a textile would increase the removal efficiency, alleviate the tearing problem and improve the ease of removal of the coating after it cured.

Chapter IV entitled “Conclusions and Limitations” provides conclusions regarding the stated hypothesis, discussion of the limitations of the study, as well as recommendations for future research.

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Chapter 2

Evaluation of an Innovative Use of Removable Thin Film Coating Technology for the Abatement of Hazardous Contaminants

Abstract

This study evaluates a new decontamination technique for the mitigation and abatement of hazardous particulates. The traditional decontamination methods used to clean facilities and equipment are time consuming, prolonging workers exposure time, may generate airborne hazards and can be expensive. The use of removable thin film coating as a decontamination technique for surface contamination proved to be a more efficient method of decontamination. This method was tested at three different sites on different hazardous metals. One application of the coating reduced the levels of these metals 90% and had an average reduction of one magnitude. The paired t-tests that were performed for each metal demonstrated that there was a statistically significant reduction of the metal after the use of the removable thin film coating: lead ($p = 0.03$), beryllium ($p = 0.05$) aluminum ($p = 0.006$), iron ($p=0.0001$), and copper ($p=0.004$). The Kendall Tau correlation coefficient demonstrates that there was a positive correlation between the initial levels of contamination and the removal efficiency for all the samples taken from different locations on the floor for each of the three sites. This new decontamination technique worked efficiently, requiring only one application, therefore decreasing exposure time to the workers and did not generate any airborne dust.

Introduction

There are many industrial processes that contaminate facilities and equipment with hazardous particulates. This can be an expensive problem, usually necessitating in extensive decontamination procedures and discarding and replacing equipment. The presence of hazardous materials, in fine ($<10\mu\text{m}$) or coarse ($>10\mu\text{m}$) particulate matter, also presents industrial hygiene issues in the work place. There is the possibility of employees being over exposed during the decontamination process to these hazardous particulates resulting in illness and even death. Most industrial processes that include machining of hazardous metals, such as, beryllium and lead require that these areas be decontaminated on a regular basis. Due to the potentially hazardous properties of metal dust and particulates a decontamination technique that is efficient, does not generate airborne dust, and is safe, is desirable.

Currently the methods of decontamination used for beryllium and other metals include the wet method, a high efficiency particulate vacuum, or sticky cloths. ⁽¹⁾ Also, many machinists clean their shops with brooms, generating airborne dusts. Not only are these processes expensive and time consuming, but they must be done on a regular basis, increasing the potential for over exposure to the employees. Removable thin film coating technology will decrease the amount of time it takes to decontaminate an area, reducing the exposure time to the workers; it will not generate airborne dust during the process and will reduce the amount of waste generated from the decontamination process.

Removable thin film coating technology has been used to decontaminate commercial nuclear facilities since the early 1980s.^[2] The removable thin film coating is a loosely adhered paint-like coating that decontaminates radioactive equipment, prevents contamination and fixes contamination in place.^[2] However, arbitrarily limiting the use of removable thin film coating for radioactive contamination eliminates the possibility of using this coating on other particulate contaminants, such as heavy metals.

In this study three different locations, the Tokamak Fusion Test Reactor Basement, Beryllium Machine Shop, and an Active Machine Shop, contaminated with different hazardous metals were chosen to analyze the efficiency of the removable thin film coating as a decontamination method. The first location was the Tokamak Fusion Test Reactor (TFTR) at the Princeton Plasma Physics Laboratory (PPPL). The Laboratory is managed by Princeton University and is funded by the U.S. Department of Energy, Office of Science. The Tokamak Fusion Test Reactor (TFTR) operated at PPPL from 1982 to 1997.^[3] TFTR set a number of world records, including a plasma temperature of 510 million degrees centigrade. In 1999, PPPL commenced the TFTR Decontamination and Decommissioning (D&D) Project. The objective of the D&D project was to completely remove TFTR in a safe, efficient and cost effective manner.^[4]

The safe removal of lead shielding at Princeton Plasma Physics Laboratory was a major component of work, during the Tokamak Fusion Test Reactor (TFTR) Decontamination and Decommissioning (D&D) Project.^[5] The physical aspects of this project started in the beginning of October 1999. Throughout the years of 1999, 2000, and 2001

approximately 250,000 pounds of lead were safely removed from the TFTR test cell and test cell basement. Typically, the lead was in the form of bricks each weighing approximately 27 pounds. The lead bricks were used as radiation shielding around TFTR's diagnostics. After years of use, many of the bricks were coated with a layer of white powder. Analysis of this powder revealed inorganic lead oxide. During the removal of the bricks, this powder had a tendency to become airborne and resettle on other surfaces throughout the work area. This re-deposition was a serious concern in the TFTR Test Cell and Test Cell Basement where there was a high number of workers performing collateral tasks associated with TFTR D&D.^[5]

The existence of lead oxide presented both airborne exposure and surface contamination issues for the workers in the field who were removing this material.^[5] The workers were required to wear disposable protective clothing, which was discarded before leaving the contamination area, and full face air purifying respirators. The workers were also entered into a lead blood level medical surveillance program. Lead toxicity targets the nervous system.^[6] Chronic exposure to lead can result in neurological effects, such as peripheral neuropathy, fatigue, wrist and foot drop, and seizures. It can also cause gastrointestinal and reproductive effects in both men and women.^[6] The presence of the surface lead dust contamination also created issues with the equipment and structural elements, such as metal beams and wooden shelves. These objects, which were coated with the lead dust, had to be disposed of as hazardous waste, substantially increasing disposal costs.

The second location was at the Old Beryllium Shop at Los Alamos National Laboratory. In 1953 all machines and equipment were moved from an old facility into the shops at building SM-39.^[7] Operations in the new shops included lathes, a mill, a surface grinder, and drill press all within a hood enclosure.^[8] The beryllium machine shops were washed down weekly and sampled to ensure that loose beryllium dust levels were below 15 $\mu\text{g}/\text{ft}^2$.^[8] In 2002 the shops in building SM-39 began the process of being decommissioned and decontaminated. Once the project is completed the area will be free released and reclassified as a non-beryllium area.

Beryllium has been used for various operations related to weapons production at LANL since 1943.^[7] Machining and firing tests resulted in beryllium being released not only in the work area but also into the environment. Machining, grinding, sanding, and other general handling of beryllium and beryllium components occurs in the machine shop as well as experimental areas. There are industrial hygiene records from 1943 to 1980 that indicate beryllium metal was processed in shops and metallurgical labs. Soluble beryllium salts were used in chemical labs at twenty different technical areas within LANL.

Inhalation is considered the primary route of exposure for workers.^[9] However, there is no human data available on the deposition or absorption of inhaled beryllium and beryllium compounds. It is assumed that beryllium and beryllium compounds are governed by the same factors as other particulates; dose, size, and solubility.^[9] A non-cancerous health endpoint is Chronic Beryllium Disease (CBD), also known as

berylliosis.^[9] Inhalation exposure to beryllium and beryllium compounds can result in CBD, which is an inflammatory lung disease. Symptoms associated with CBD include chest pain, cough, and dyspnea.^[10] It is evident that there is an exposure-response relationship to beryllium (EPA, 1998). Several studies have shown that workers chronically exposed long enough to low levels of beryllium and beryllium compounds, even at the beryllium Permissible Exposure Limit (PEL) of $2\mu\text{g}/\text{m}^3$ did develop CBD.^[9]

There is epidemiological data available that suggests that lung cancer is a health endpoint for workers with inhalation exposure to beryllium and beryllium compounds. For airborne particles, the lung is the target organ for both humans and animals.^[11] The safe human chronic air concentration (RfC) is $2\text{E}-2\mu\text{g}/\text{m}^3$. This is 1/10 of the adjusted adverse effect level for beryllium sensitization and CBD in workers. It is estimated that the human lung cancer risk is $2.4\text{E}-3$ for exposure to $1\mu\text{g}/\text{m}^3$ of beryllium.^[9] The relationship between the quantitative cancer and non-cancer risk estimates can be determined by calculating the cancer risks to people who are hypothetically exposed through inhalation to the RfC of $2\text{E}-2\mu\text{g}/\text{m}^3$. The lifetime cancer risk would be $5\text{E}-6$ for inhalation. This risk level is normally considered to be negligible.^[9]

The final site chosen was an active machine shop at the Princeton Plasma Physics Laboratory (PPPL). During machining operations, such as, grinding, polishing, cutting, and drilling, and during clean up, the workers have inhalational exposure to metal particulates and dusts. The usual methods for cleaning a machine shop, sweeping with a

broom and vacuuming, stir up the metal dust causing airborne contamination. This dust resettles onto the machinery, floors and other surfaces.

Occupational exposure to metal dusts and particulates can induce a variety of lung disorders and disease including parenchymal diseases, airway disorders, and cancer.^[12]

Pneumoconiosis is an example of a parenchymal disease that can occur because of occupational exposure to metal dusts. The machine shop at PPPL most frequently machined aluminum, copper, and iron.

There are a number of pulmonary effects associated with aluminum exposure, such as chronic bronchitis, pulmonary fibrosis, granulomatous lung disease, pneumonitis, and pulmonary edema.^[12] The occupational health hazards that are of most concern in a machine shop are those associated with grinding and polishing aluminum. There have been cases of alveolar proteinosis and fibrosis in workers in machine shops. Inhalation of copper dusts and fumes may cause irritation to the upper respiratory tract and ulceration or perforation of the nasal septum.^[12] Copper dust may also produce metal fume fever in workers resulting in symptoms such as chills, muscle aches, nausea, fever, coughing, and weakness. Occupational inhalational exposure to iron can result in pulmonary disease, also referred to as siderosis. Siderosis for the most part is a benign disease with minimal or no symptoms.^[12]

Methods

This study employed quantitative methods to determine if the efficiency of the removable thin film coating was statistically significant. Due to the hazardous properties of the different metals, a decontamination technique that is safe to the workers, efficient, and economical is desirable. One such technique is the use of removable thin film coating. To determine the efficiency of the removable thin film coating a sampling plan was developed that consisted of using wipe samples to get an initial level of the contaminant present at each location and also using wipe sampling after the removal of the thin films coating to determine the amount of contaminant left from various locations within each site.

Wipe Sampling

Individuals trained in surface wipe sampling techniques collected samples of the contaminant before and after the use of the removable thin film coating that were subsequently analyzed and recorded as initial and after levels. The method used to collect the samples was based on the NIOSH 9100 “Lead in Surface Wipe Sample” of the NIOSH Manual of Analytical Methods. Two sample areas were marked out in each location adjacent to each other. There were 17 locations at the PPPL TFTR basement sampled for lead, 9 locations at the Old Beryllium Machine shop were sampled for beryllium, and 21 locations at the PPPL active machine shop were sampled for aluminum, copper, and iron. The first area in each location was used to determine the initial level of contaminant. The other area was sampled after the thin film coating was removed to determine how much contaminant was left. The two adjacent sample areas

for each location were assumed to have the same concentration and distribution of the contaminant. Only having one sample area at each location was not practical as it could not be determined how much of the contaminant was removed during the initial wipe sampling.

Removable thin film coating

In the second sample area of each location a layer of the removable thin film coating was applied using a sponge brush. The removable thin film coating that was used for decontamination was a water-based organic polymer.^[2] Removable thin film coating technology is designed to trap and fix particulates in the coating's matrix by adhesion. As the liquid polymer is spread over the contaminated area, it migrates into the micro-voids of the surface. Once the polymer starts to cure, it attracts, absorbs, and chemically binds to the contaminants.^[2] This coating technology can be applied to an existing contaminated area to fix and capture the particulates for removal. Once the curing process is completed, the removable thin film coating traps the contaminant into the polymer matrix.^[2] The nature of the removable thin film coating, after sufficient cure time, is such that it can typically be removed as one continuous entity. The removable thin film coating can be applied to almost any surface type and have a statistically significant removal rate.

Laboratory Analysis of the Wipe Samples

Evaluation protocols required that the wipe samples be analyzed by laboratories recognized by the American Industrial Hygiene Association's Industrial Laboratory

Accreditation Program (IHLAP ISO/IEC 17025 Accreditation). All wipe samples were analyzed for beryllium, lead, iron, copper, or aluminum by inductively coupled plasma mass spectrometry using the Environmental Protection Agency modified SW 846 6010B;ICP;LEADWP. Quality control procedures in the laboratory included analyzing blanks that were submitted with each sample batch.

Some of the metal samples were reported as below the laboratory detection limits and the authors were concerned that this would affect the statistical significance of the removal efficiency of the thin film coating. For the samples that were below the limit of detection, a value one-half of the detection limit was substituted.

Statistical Analysis

The data was entered into a Microsoft ® Excel spreadsheet and a one tailed paired t-test was used to determine if there was a significant reduction in amount of metal particulates after the use of the removable thin film coating. The removal was considered significant if $p < 0.05$. Data was also analyzed using SAS version 9.1 (SAS Institute Inc., Cary, N.C.). Kendall tau-b was used to measure the strength of association between the initial surface concentration and the percentage of removal efficiency.

Results

Surface Wipe Samples

The lead surface dust levels were measured by wipe sampling at seventeen different locations on the floor in the TFTR basement. The means for the initial levels were

2687.35 ug/ft² and 111.24 ug/ft² for the final levels after the use of the removable thin films. The largest concentration of lead dust was located where the columns of lead bricks were used to shield the diagnostic equipment. A one-tail paired t-test was computed to determine if the use of the removable thin film coating significantly reduced the lead surface dust contamination. The results ($p = 0.03$) demonstrated that there was a statistically significant removal of the lead surface dust. However, the power of this test was small due to the small number of samples measured. The average removal efficiency was 82%. The data, summarized in Figure 1, also showed that there was an average reduction of lead dust in the order of one magnitude.

The beryllium surface dust levels were measured by wipe sampling at nine different locations throughout the beryllium machine shop. The sample areas fell into two categories: accessible occupied areas and limited access mechanical spaces. The accessible occupied areas were the floors, walls, milling machines, and countertops. The limited access mechanical space included the baghouse, ventilation ductwork, and light fixtures. The means for the initial levels were 0.1540 ug/cm² and 0.575 ug/cm² for the final levels after the use of the removable thin films. The largest concentration of beryllium dust was located on the Bridgeport milling machine. The milling machine having the largest concentration of beryllium is obviously due to the fact that this is where the contaminant was being machined. The grinder table top samples were removed from the data set as an outlier. This outlying data may have been caused by cutting oil or lubricant that was present interfering with the removable thin film coating's ability to adhere to the surface. A one-tail paired t-test was computed to determine if the

use of the removable thin film coating significantly reduced the beryllium surface dust contamination. The results ($p = 0.05$) demonstrated that there was a statistically significant removal of the beryllium dust. However, the power of this test was small due to the small number of samples measured. The average removal efficiency was 88%. The data (Figure 2) also showed that there was an average reduction of beryllium dust in the order of two magnitudes.

The final facility where wipe samples were measured was from the active machine shop at PPPL. Twenty-one samples for each of the metals of interest, aluminum, iron, and copper were taken from the floor around the various machinery throughout the shop. The means for aluminum were 32.07 ug/cm² for the initial levels and 1.64 ug/cm² for the final levels after the use of the removable thin films, for iron initial levels were 25.46 ug/cm² and 1.97 ug/cm² for the final levels, and copper had an initial level of 3.98 ug/cm² and 0.12 ug/cm² for the final levels. A one-tail paired t-test was computed for each of the three metals to determine if the use of the removable thin film coating significantly reduced the metal surface dust contamination. The results for aluminum ($p = 0.006$), iron ($p=0.0001$), and copper ($p=0.004$) demonstrated that there was a statistically significant removal for each of the different metals. The average removal efficiency for all three metals were nearly the same; copper 90%, iron 88%, and aluminum 89%. The data also showed that there was an average reduction of each metal dust in the order of one magnitude. This is summarized in Figure 3 for aluminum, Figure 4 for iron and Figure 5 for copper.

Kendall Tau Correlation Coefficients

Kendall Tau correlation coefficients were computed to investigate the probability of removal of the contaminant with the removable thin film coating. All of the wipe samples, except for the ones collected at LANL, were taken from various locations on the floor. The LANL samples were taken from different surface locations throughout the machine shop. All of the floor samples from the different locations and for the different contaminants indicated that there was a significant positive correlation between the initial contamination concentration and the percentage of removal efficiency as shown in Table I for the metals at the PPPL Machine Shop and in Table II for the metals at PPPL TFTR Basement.

The results of the Kendall Tau b correlation coefficient for the beryllium wipe samples at LANL (Table III) did not produce a statistically significant correlation between the initial contamination concentration and the percent of removal efficiency. These samples were taken from different surface substrates within the machine shop. These areas include the floors, walls, light fixtures, milling machines, and from the ventilation.

Discussion

The first objective behind using the removable thin film coating technique at the different locations and on the different contaminants was to decontaminate the areas below the required levels to be considered cleaned. During the decontamination of the TFTR basement, the removable thin film coating worked in reducing the lead contamination below the HUD guideline of 50ug/ft². The beryllium machine shop was

to be decontaminated below the Department of Energy standard of $0.2\mu\text{g}/100\text{cm}^2$.⁽¹⁾

After one application of the coating in the various accessible and non-accessible locations, the mean beryllium level was $0.06\mu\text{g}/100\text{cm}^2$. This level was low enough for the area to be considered clean of beryllium contamination and could be free released, meaning used for any non-beryllium activities. The removable thin film coating also worked very well in decontaminating the metal dust from the PPPL machine shop areas, with an average of 89% removal rate after one application.

Another objective was to decrease the amount of time that it takes to decontaminate the areas, therefore decreasing the workers exposure time and preventing airborne dust from generating during the process. During the initial decontamination of the TFTR basement, multiple cleanings with water and Windex were done. This process took a long period of time, generated a large amount of liquid waste to be disposed of as hazardous waste, and did not reduce the levels of lead below the HUD guidelines. Only one application of the removable thin film coating technology was needed to reduce the amount of lead to a safe level. Overall, this process took less time and did not generate as much waste. When this decontamination technology was applied to the PPPL machine shop it did not generate any airborne dust, eliminating a health concern associated with the traditional methods of cleaning with a broom or vacuum.

The use of the Kendall correlation coefficient demonstrated that the wipe samples at all the different locations where the removable thin film coating was used on the floor had a significant positive correlation between the initial levels and the removal efficiency

percentage. However, when the coating was used on different substrates, such as, the milling machine, grinder table, and light fixtures, there was no correlation between the initial levels of contamination and the removal efficiency percentage. This could be due to cutting oils and lubricants that were present on the machines interfering with removable thin film coating's adhesion to the surface. Also these substrates did not have a continuous flat surface like the floor, and the coating may not have been able to get into all the cracks and crevasses.

There were a few limitations with this study. One limitation of this study is the assumption that the contaminants are evenly distributed across each sample location. The locations of the sample areas used to determine the initial concentration of the contaminants were adjacent to locations where the removable thin film coating was applied. This was done because there was no way to determine how much of the contaminant was removed during the wipe sampling process and it was thought that this was the best way to get the most accurate data. Another limitation was the small number of samples and the lack of repeat samples. Despite these limitations our results demonstrated that the removable thin film coating can be used successfully as a decontamination technique for hazardous particulates. It was less time consuming and less labor intensive than the traditional decontamination methods.

Conclusion

The results presented in this research indicate that the removable thin film coating technique performed well at decontaminating three different facilities contaminated with

various hazardous particulates. The results indicated that there is an average of 90% removal efficiency with one application of the coating. The paired t-tests that were performed for each metal demonstrated that there was a statistically significant reduction of the metal after the use of the removable thin film coating. This new decontamination technique worked efficiently, requiring only one application therefore decreasing exposure time to the workers and preventing airborne dust from generating.

Figure 1. Lead surface dust reduction after the use of the removable thin film coating.

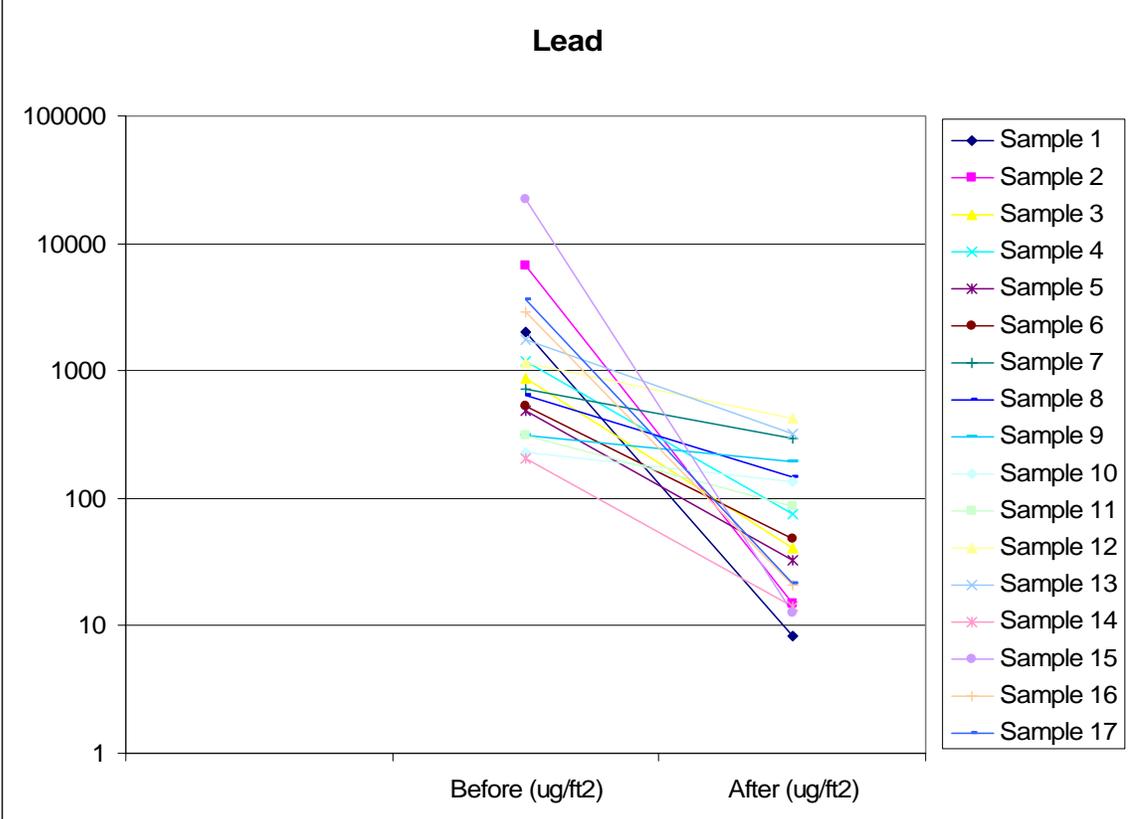


Figure 2. Reduction of beryllium contamination after the use of the removable thin film coating.

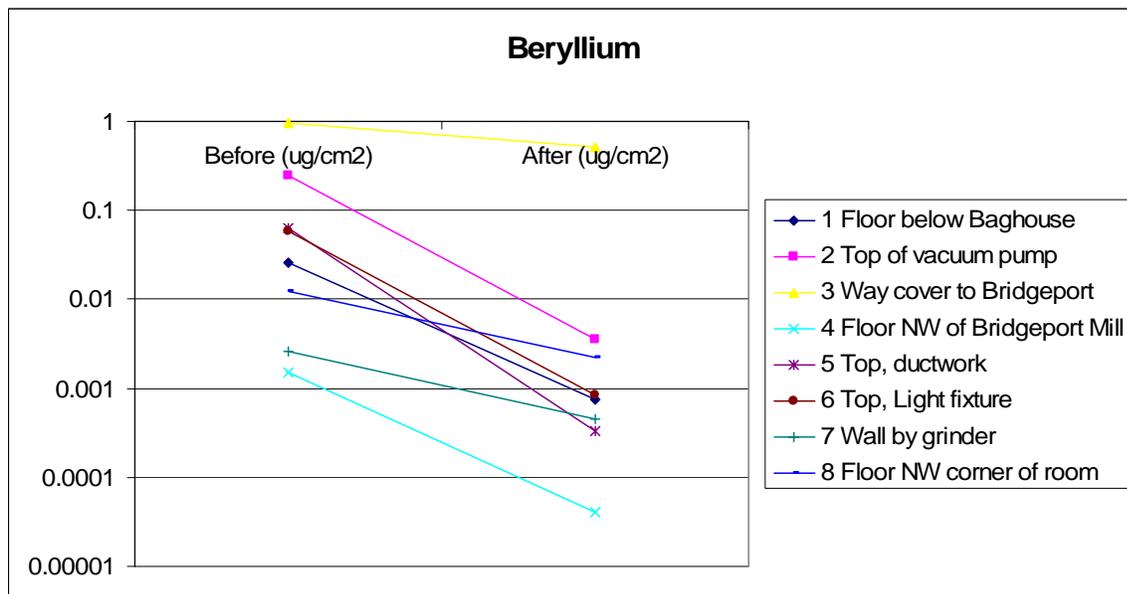


Figure 3. Reduction of aluminum contamination after the use of the removable thin film coating.

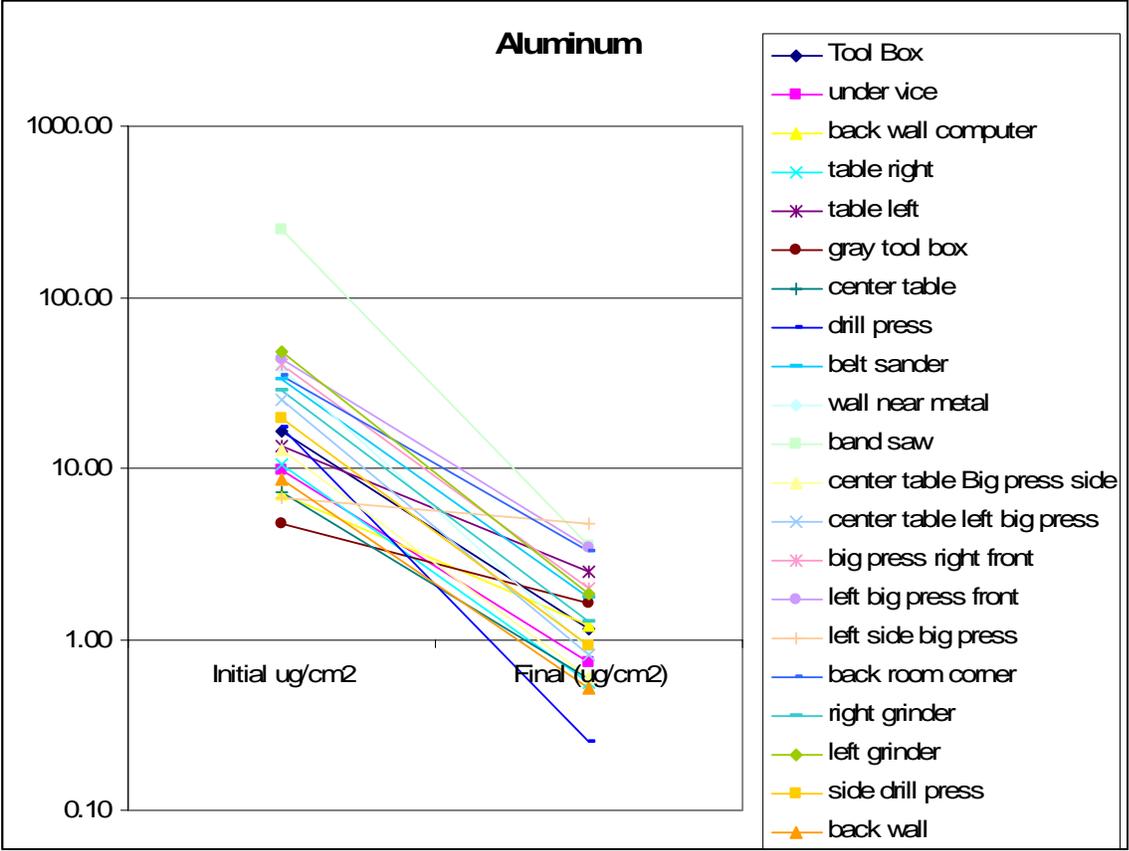


Figure 4. Reduction of iron contamination after the use of the removable thin film coating.

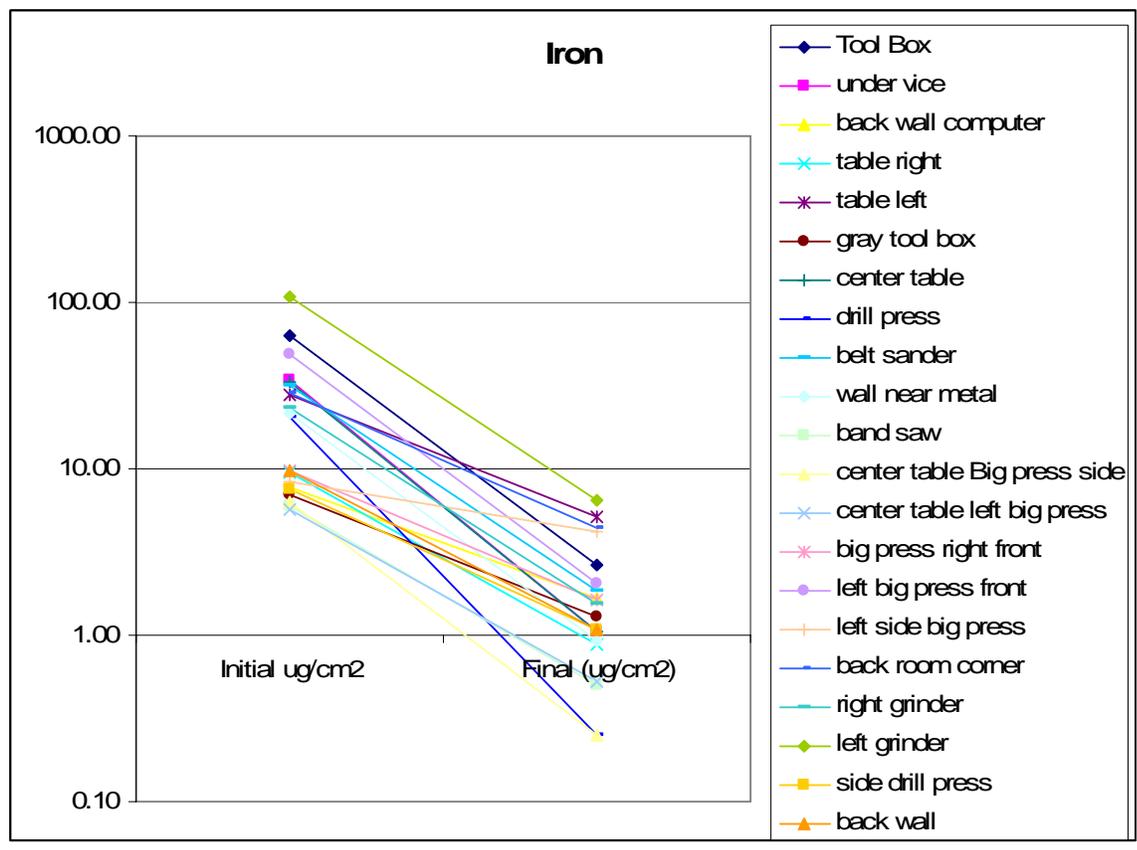


Figure 5. Reduction of copper contamination after the use of the removable thin film coating.

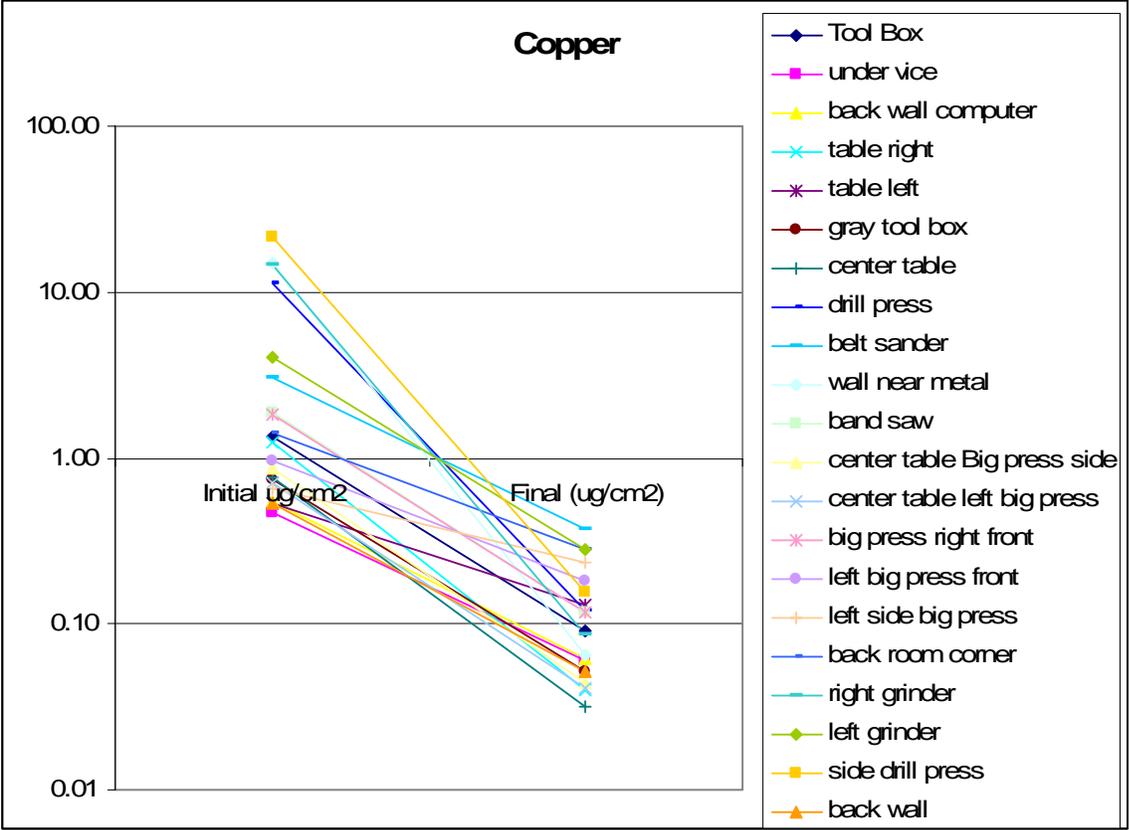


Table I. Kendall Tau b Correlation Coefficients for the metals at the PPPL Machine Shop

Variable	N	Cu Initial	Cu Percent Removed	Fe Initial	Fe Percent Removed	Al Initial	Al Percent Removed
Cu Initial	21	1.00	0.45 (p=0.004)	0.04 (p=0.79)	0.15 (p=0.35)	0.43 (p=0.006)	0.46 (p=0.003)
Cu Percent Removed	21	0.45 (p=0.004)	1.00	-0.16 (p=0.30)	0.28 (p=0.07)	0.13 (p=0.40)	0.55 (p=0.0005)
Fe Initial	21	0.04 (p=0.79)	-0.16 (p=0.30)	1.00	0.30 (p=0.06)	0.17 (p=0.28)	-0.11 (p=0.47)
Fe Percent Removed	21	0.15 (p=0.35)	0.28 (p=0.07)	0.30 (p=0.06)	1.00	0.15 (p=0.33)	0.34 (p=0.03)
Al Initial	21	0.43 (p=0.006)	0.13 (p=0.40)	0.17 (p=0.28)	0.15 (p=0.33)	1.00	0.47 (p=0.003)
Al Percent Removed	21	0.46 (p=0.003)	0.55 (p=0.0005)	-0.11 (p=0.47)	0.34 (p=0.03)	0.47 (p=0.003)	1.00

Table II. Kendall Tau b Correlation Coefficients for the metals at PPPL TFTR Basement

Variable	N	Pb Initial	Pb Percent Removed
Pb Initial	17	1.00	0.59 (p=0.0010)
Pb Percent Removed	17	0.59 (p=0.001)	1.00

Table III. Kendall Tau b Correlation Coefficients for the metals at LANL Beryllium Machine Shop

Variable	N	Be Initial	Be Percent Removed
Be Initial	8	1.00	0.22 (p=0.40)
Be Percent Removed	8	0.22 (p=0.40)	1.00

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Chapter 3

The Use of the Removable Thin Film Coating Technique as an Alternative to Traditional Decontamination Methods to Mitigate and Abate Hazardous Particulates

Abstract

This study evaluates a new decontamination technique for the mitigation and abatement of hazardous dust and particulates. Traditional decontamination methods are time consuming, expensive, can create airborne hazards, and do not always bring the concentration of the contaminant to acceptable levels. The use of the removable thin film coating will increase efficiency, will not generate airborne hazards, will decrease costs, and with one application will bring the hazardous dust concentrations to acceptable levels. Qualitative tests demonstrated that the removable thin film coating reduced the amount of visible luminescent dust (a surrogate for hazardous dust) from various surfaces. It also indicated that wherever there were minute scratches, the coating did not remove all of the dust. However, the qualitative tests showed that this decontamination method worked well as a preventative method, protecting clean areas from becoming contaminated when exposed to the luminescent dust. Further investigation was conducted using a scanning electron microscope and carbon dust. Overall, the SEM experiment demonstrated that there was a statistically significant ($p=0.00007$) removal of carbon dust (less than 10 μ m in size) from surfaces with crevasses larger than 3 μ m. The

SEM also revealed that there were some limitations, where there were large clusters of carbon dust, the coating would tear and remain on the sample surface. One method to resolve this limitation involved adding Kevlar™ fibers to the removable thin film coating. It was thought that this would increase the strength of the coating and eliminate the coating from tearing when removing large clusters of a contaminant. Unfortunately this did not alleviate the issue. The use of an engineered textile, saturated with the coating, appeared to eliminate the problem with the coating not being able to remove the contaminant from the minute surface scratches and improved the removal process of the coating.

Introduction

Machine shops present unique occupational hazards, which are potentially dangerous if not controlled. During machining operations, such as, grinding, polishing, cutting, and drilling, and during clean up, the workers have inhalational exposure to metal particulates and dusts. The usual methods for cleaning a machine shop, sweeping with a broom and vacuuming, stir up the metal dust causing airborne contamination. This dust resettles onto the machinery, floors and other surface. These methods of cleaning are not efficient at lowering and maintaining the metal dusts to acceptable levels. The use of the removable thin film coating will eliminate the generation of airborne dust during the cleaning process and is a more efficient method of abatement, bringing the dust levels down to acceptable levels.

Occupational exposure to metal dusts and particulates can induce a variety of lung disorders and disease including parenchymal diseases, airway disorders, and cancer^[1].

Pneumoconiosis is an example of a parenchymal disease that can occur because of occupational exposure to metal dusts.

One of the major issues in industry involves contamination of equipment and facilities due to different processes. This can be an expensive problem, usually resulting in extensive decontamination procedures and the need to discard and replace equipment. In a previous study the use of a removable thin film coating was field tested as a decontamination technique for particulate surface contamination. The coating was tested at three different facilities on different hazardous particulates. The results of that study demonstrated that the removable thin film coating technique performed well at decontaminating the three different facilities contaminated with various hazardous particulates. The results indicated that there was an average of 90% removal efficiency with one application of the coating. Paired t-tests were performed for each metal and there was a statistically significant reduction of the metal after the use of the removable thin film coating. This new decontamination technique worked efficiently, requiring only one application, therefore decreasing exposure time to the workers and did not generate any airborne dust. A Kendall Tau correlation coefficient was performed and the results demonstrated that there was a positive correlation between the initial levels of contamination and the removal efficiency for all the samples taken from different locations on the floor for each of the three sites.

Due to the positive results from the field testing of the removable thin film coating, the authors felt it was important to further investigate this technique as a viable alternative to traditional methods of decontamination.

Removable Thin Film Coating

The removable thin film coating is a water-based organic polymer ^[2]. It is designed to trap and fix particulates in the coating by adhesion. As the liquid polymer is spread over the contaminated area, it migrates into the micro-voids of the surface. After about eight hours the polymer starts to cure. As it cures it attracts, absorbs, and binds to the contaminants into the polymer matrix ^[2]. This coating technology can be applied to an existing contaminated area to fix and capture the particulates for removal ^[2]. After the coating has completely cured, it can typically be removed as one continuous entity.

Methods

Qualitative Tests and Data

Initially, qualitative tests were conducted to determine if the removable thin film coating visibly reduced the amount of dust present on a surface. The removable thin film coating was applied to various surfaces that were contaminated with luminescent dust with particle sizes less than 5 μm . Luminescent dust was used as a safe way to represent the presence of hazardous particulates. Two different tests were conducted. The first test was done to evaluate the efficiency of the coating for the use of abating surfaces contaminated with hazardous particulates. Five different surfaces samples: stainless steel, aluminum, galvanized steel, PVC, and Lexan, were coated with a layer of the luminescent dust. Then a layer of the removable thin film coating was painted onto each sample. After 24 hours the coating was removed (Figure 1) and an ultraviolet lamp was used to detect any remaining luminescent dust.



Figure 1. The removable thin film coating is removed after it has cured

The results from qualitative tests did show that there was a reduction in the amount of luminescent dust still on the surface after the coating was removed. However, wherever there were minute scratches in the surface of the samples, such as scratches left from the milling process, the removable thin film coating did not remove the luminescent dust from these areas. It also appeared that occasionally as the coating was being removed from the sample, some of the luminescent dust would become loose and redeposit onto the sample surface. This loosening also allows the hazardous particulate to be re-introduced into the air.

A second test was conducted to determine if the removable thin film coating could be utilized as a protective coating for different surfaces. This test was used to determine if a clean area (ready for free release, and not considered a contamination area) is painted with the coating, is still clean after being exposed to the contaminant when the coating is removed. To test this theory the surface samples were cleaned and then coated with the removable thin film coating and left to cure overnight. Luminescent dust was then applied to each surface and another layer of the coating was placed on top of that. After

this second layer cured, both were removed and the area was qualitatively analyzed using the ultraviolet light. Trapping the luminescent dust between two layers of removable thin film coating did protect the sample surfaces from contamination.

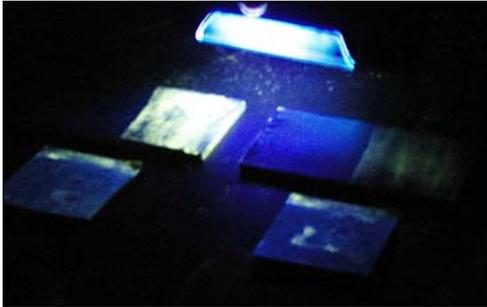


Figure 2. Samples under UV light. The sample on the left is the sample that was contaminated first and then painted with the removable thin film coating. This sample reveals the luminescent dust is still present after the removal of the thin film coating. The sample on the right, where the contaminant was sandwiched between two layers of the coating, shows that very little luminescent dust remains after removal of the thin film coating.

This method of using the coating as a protective measure for keeping the clean surface from becoming contaminated worked well (Figure 2). The contaminant became trapped between the two layers of the coating and did not contaminate the clean surface. The dust did not migrate through the layers, contaminating the surface. This test reinforces the idea of using the removable thin film as a proactive measure in many manufacturing situations where equipment is reused.

SEM Sampling and Data

The results from qualitative tests showed that there was a reduction in the amount of luminescent dust still on the surface after the coating was removed. However, these tests also showed that wherever there were minute scratches in the surface of the samples, the removable thin film coating did not remove the luminescent dust from these areas. To

learn how the coating worked at the microscopic level and to determine why the coating was not removing the contaminant from the minute scratches a scanning electron microscope (SEM) was used.

Different substrate material was used to simulate the different types of surfaces on which the removable thin film coating could be used. The surface substrate samples included stainless steel, aluminum, galvanized stainless steel, Lexan® Polycarbonate, G-10, Phenolic, Gray PVC, and vinyl floor tile. G-10 is a laminate material made of continuous filament glass cloth with an epoxy resin binder. This material has excellent electrical properties, chemical resistance, and high strength. It is used in electrical equipment, cryogenic insulation and aerospace conditions^[3]. Phenolic is also a laminate material that is made of a continuous cotton woven cloth that is impregnated with a phenolic resin binder. This type of material is hard and dense and will not shrink or warp when heated up to 250 degrees F. Phenolic is used for electrical insulators, washers, pulleys, gaskets, etc.^[4]. Three sets of each surface substrate samples were needed to test if the removable thin film coating was efficient at removing contaminants that were 10µm or less from the surface and from inside any micro-voids. The first set was used to determine what the surface of the substrate looked like under the SEM without any contamination on it. The second sets of samples were coated with calibrated carbon dust. Carbon dust was chosen as a surrogate for the hazardous particulates because it was safer to work with and had similar properties. The carbon dust was calibrated to be with in the range of 1 to 10µm. This size was chosen to represent respirable particles, as this is what is most dangerous to the workers. Each sample had the carbon dust brushed directly onto

the surface. This was done to insure that the dust got into all the micro-voids. The non-metallic samples were placed in a sputter coater and coated with either 30 or 40 nm of iridium.

The non-metallic samples had to be coated with a thin layer of iridium to make them conductive and able to be viewed with the SEM. Iridium was chosen as the coating instead of gold due to the fact that the grain size is finer and smaller, it is good for high resolution work, and it does not oxidize ^[5]. The sputter coater uses an ion source that emits a fine beam of ions and an energetic neutral beam onto a target of iridium. The iridium is then sputtered off the target surface and onto the sample. The ion beam does not interact with the sample, minimizing the damage that it may cause the sample ^[5]. This process takes place under high vacuum. During the coating process the samples undergo a complex rotating planetary motion. This experiment used a rotational speed of 3 with a tilt speed of 50%, a tilt angle of 50 degrees, and two ion sources with a beam energy of 7kv and a beam flux of 6mÅ. The third set of samples was coated in the same manner as with the carbon. Then each sample was coated with the removable thin film coating and left to cure overnight. After the coating cured, it was removed and all the non-metallic samples were sputtered in the same manner as before.

Once all the samples were prepared they were then viewed in a scanning electron microscope. The SEM microscope uses electrons instead of light to form an image ^[6]. Optical microscopes have a limited resolution of about 1000 diameters magnification. The SEM has a limited resolution of magnification of about 1,000,000 diameters ^[6]. An electron beam is generated in a vacuum. The beam is then scanned across the surface of

the sample by electromagnetic deflection coils and generates secondary electrons, backscattered electrons, and x-rays^[7]. A detector collects these signals and forms an image of the sample that is displayed on a cathode ray tube screen^[6]. By the detector correlating the sample scan position with the resulting signal, the image that is formed is very similar to an image that would be seen with an optical microscope. The lighting and shadowing provide a natural looking surface topography^[6]. A copy of the image generated by the SEM (Figure 3 and 4) for each sample was printed and viewed. For each set of before and after samples a $300\ \mu\text{m}^2$ and $1200\ \mu\text{m}^2$ area was marked off. Each carbon particle present within the marked area was counted to determine the concentration for each sample.

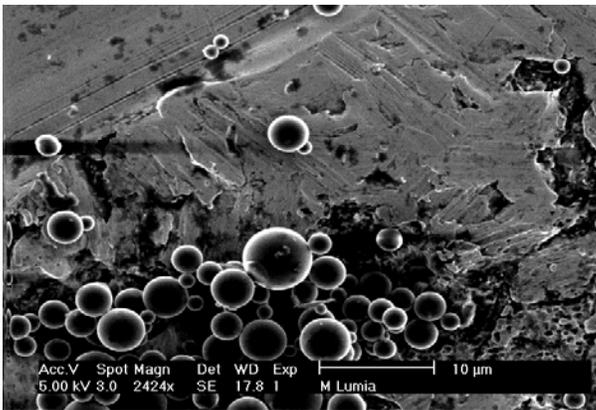


Figure 3. SEM image of stainless steel with carbon dust

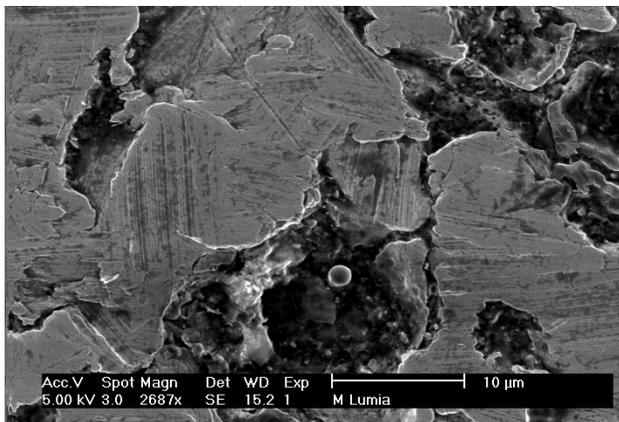


Figure 4. SEM image of stainless steel after use of removable thin film

The carbon surface dust levels were measured by determining how many particulates were present before the use of the removable thin film coating compared to how many were present after the use of the coating for each surface. The means for the initial levels were 0.042 ug/cm² and 0.006 ug/cm² for the final levels after the use of the removable thin films. A one-tail paired t-test was computed to determine if the use of the removable thin film coating significantly reduced the carbon surface dust contamination. The results ($p = 0.00007$) demonstrated that there was a statistically significant removal of the carbon surface dust. However, the power of this test was small due to the small number of samples measured. The average removal efficiency was 81%. The data also showed that there was an average reduction of carbon dust of less than one magnitude which is summarized in Figure 5.

The SEM was used to determine how the removable thin film coating worked at getting into micro-voids and at removing particulates less than 10 micrometers. It was discovered that there were a few areas where the coating failed. The coating could not get into crevasses less than 3 μm to remove the particles that were wedged there. In some

areas where there were large clusters of carbon particles, it was observed that when the coating was removed, the weight of the cluster caused the coating to tear. The cluster, covered with a layer of the removable thin film coating, remained on the surface of the sample substrate as seen in Figure 6. However, the overall SEM experiment did show that there was a statistically significant removal of the carbon dust from the micro-voids that were found on the various substrate samples.

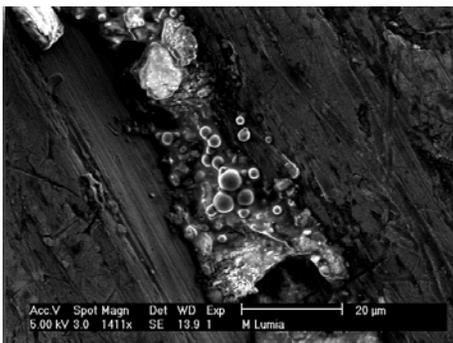


Figure 6. A cluster of carbon particles covered in the removable thin film coating that remained on the surface after the coating was removed

Improvement of the formula and the application

Enhancement of the Removable Thin Film Coating Formula

Due to the issues of the coatings tearing wherever there were large clusters of carbon an attempt was made to increase the strength of the coating and therefore increase the efficiency of removal process. Kevlar™ fibers were added to the removable thin film coating. Kevlar™ fibers were chosen because it was thought that the fibers would overlap each other forming a web like structure that would increase the overall strength of the coating. Into 93.2 grams of the removable thin film coating, 0.7 grams of Kevlar™ fibers were added and mixed thoroughly to distribute the fibers evenly.

For this experiment the two different removable thin film coatings were used to decontaminate a machine shop. Product 1 is the original removable thin film coating and product 2 is the removable thin film coating with the incorporation of Kevlar™ fibers. Individuals trained in surface wipe sampling techniques collected samples of the contaminant before and after the use of both products and were subsequently analyzed and recorded as initial and after levels. The method used to collect the samples was based on the NIOSH 9100 “Lead in Surface Wipe Sample” of the NIOSH Manual of Analytical Methods. Three sample areas adjacent to each other were marked out in each of the 21 locations. The first area in each location was used to determine the initial level of contaminant. The second and third areas were sampled after the product 1 and product 2, respectively, were applied and removed to determine how much contaminant was left. The three adjacent sample areas for each location were assumed to have the same concentration and distribution of the contaminant. Only having one sample area at each location was not practical as it could not be determined how much of the contaminant was removed during the initial wipe sampling.

After the samples were collected they were sent to an independent laboratory for analysis. Evaluation protocols required that the wipe samples be analyzed by laboratories recognized by the American Industrial Hygiene Association’s Industrial Laboratory Accreditation Program (IHLAP ISO/IEC 17025 Accreditation). All wipe samples were analyzed for iron, copper, or aluminum by inductively coupled plasma mass spectrometry using the Environmental Protection Agency modified SW 846 6010B;ICP;LEADWP.

Quality control procedures in the laboratory included analyzing blanks that were submitted with each sample batch.

Some of the metal samples were reported as below the laboratory detection limits and the authors were concerned that this would effect the statistical signification of the removal efficiency of the removable thin film coating. For the samples that were below the limit of detection, a value one-half of the detection limit was substituted.

Twenty-one samples for each of the metals of interest, aluminum, iron, and copper were taken from the floor around the various machinery throughout the shop. The results for product 1 are the following: The means for aluminum were 32.07 ug/cm² for the initial levels and 1.64 ug/cm² for the final levels after the use of the removable thin films, for iron initial levels were 25.46 ug/cm² and 1.97 ug/cm² for the final levels, and copper had an initial level of 3.98 ug/cm² and 0.12 ug/cm² for the final levels. A one-tail paired t-test was computed for each of the three metals to determine if the use of the removable thin film coating significantly reduced the metal surface dust contamination. The results for aluminum ($p = 0.006$), iron ($p=0.0001$), and copper ($p=0.004$) demonstrated that there was a statistically significant removal for each of the different metals. The average removal efficiency for all three metals were nearly the same; copper 90%, iron 88%, and aluminum 89%. The data, summarized in Figures 7,9, and 10, also showed that there was an average reduction of each metal dust in the order of one magnitude.

For product 2 the results are: The means for aluminum were 32.07 ug/cm² for the initial levels and 1.47 ug/cm² for the final levels after the use of the removable thin films, for iron initial levels were 25.46 ug/cm² and 1.22 ug/cm² for the final levels, and copper had an initial level of 3.98 ug/cm² and 0.35 ug/cm² for the final levels. A one-tail paired t-test was computed for each of the three metals to determine if the use of the removable thin film coating significantly reduced the metal surface dust contamination. The results for aluminum ($p = 0.006$), iron ($p=0.0001$), and copper ($p=0.004$) demonstrated that there was a statistically significant removal for each of the different metals. The average removal efficiency for all three metals were nearly the same; copper 86%, iron 92%, and aluminum 90%. The data, as shown in Figures 8, 10, and 12, revealed that there was an average reduction of each metal dust in the order of one magnitude.

Table 1. Friedman test

P value	P<0.0001
Exact or approximate P value?	Gaussian Approximation
Are means signif. different? ($P < 0.05$)	Yes
Number of groups	3
Friedman statistic	97.01

Because the results obtained by the wipe samples were non-parametric, paired, continuous measures, a Friedman's test (Table 1) was used to determine if at least one of the groups' means was statistically different from the others. Based on the p-value ($p<0.0001$) obtained from this test, one of the groups' mean was statistically different.

Finally an unpaired t-test was done to compare the two different coatings to each other to determine if one was statistically different from the other. There was no statistically

significant difference between the two products ($p=.197$). Unfortunately, this test demonstrated that the addition of the Kevlar™ fiber neither increased nor decreased the efficiency or the strength of the coating.

In the past studies, one of the issues with the removable thin film coating occurred in areas where there were large clusters of the contaminant. When the coating was removed, the weight of the cluster caused the coating to tear. The tear would leave the contaminants, covered with a layer of the removable thin film coating, on the surface. For this study it was proposed that the addition of the Kevlar™ fibers into the removable thin film coating would increase the strength of the coating. The coating could then more completely remove the metal dust from the surface, thereby increasing the efficiency of the removal process. However, based on the results of this study, the addition of Kevlar™ fibers neither solved the problem nor increased the efficiency of the coating.

Engineered textile

To increase the efficiency of the thin film in the removal of hazardous particulate contamination a new process was tested that incorporated an engineered textile. The engineered textile is absorbent and highly porous; the thickness of the textile can vary so it is able to hold various amounts of the removable thin film coating. The dimensions of the textile can be altered to cover part or all of the contaminated area. First, each sample was coated with the luminescent dust. Then the textiles, saturated with the coating, were placed onto the sample surfaces. After allowing each sample to cure overnight, the coating with the textile was removed. An ultraviolet light qualitatively determined

(Figure 13) that the addition of the textile did increase the efficiency of the removal of the hazardous particulates from the surface of the samples.

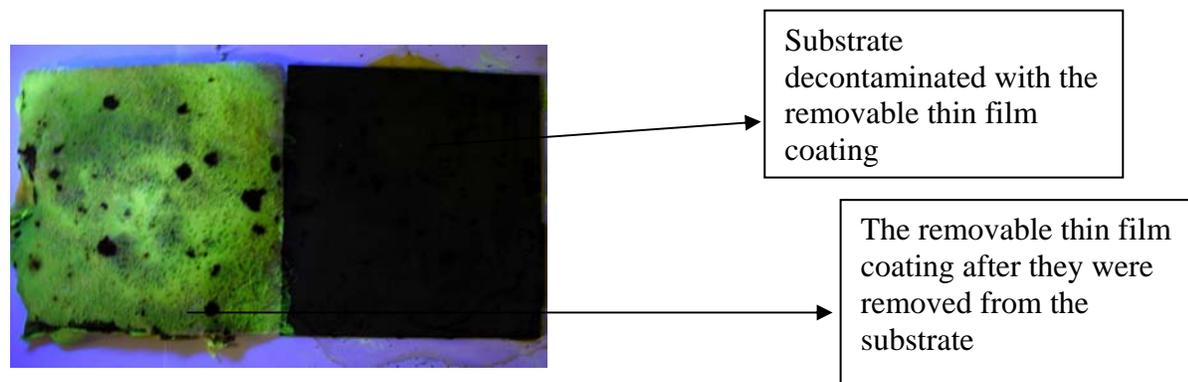


Figure 13. Use of the removable thin film coating with engineered textile. The yellow is the luminescent dust shown under the ultraviolet light that has adhered to the removable thin film coating.

Discussion

The qualitative tests did demonstrate a reduction in the amount of dust that was removed with one application of the removable thin film coating. The tests also revealed some limitations with the products and indicated that more research is required. Wherever there were minute scratches in the surface of the samples, the luminescent dust remained after the coating was removed. Also, the authors occasionally witness some of the luminescent dust becoming loose from the coating and redepositing onto the sample surface as the coating was being removed from the sample. These are issues that may be eliminated with manipulation of the formula for the removable thin film coating. A product that is less viscous, while maintaining its integrity to bind to particulates, may be more efficient at getting into the minute areas. More research is needed to develop a stronger bond between the contaminant and the cured removable thin film coating to prevent redeposition of the contaminant onto the surface.

The use of the removable thin film coating as a proactive protective coating for surfaces that are already clean worked very well during the luminescent dust test. Trapping the luminescent dust between two layers of coating did protect the sample surfaces from contamination. This is an encouraging finding as one of the uses of this technique is to prevent the contamination of equipment, such as, glove boxes, ventilation systems, and machinery. The protective qualities of this technique could potentially save a significant amount of the money that is presently spent replacing all of these contaminated items.

Overall, the SEM experiment did show that there was a statistically significant ($p = 0.00007$) removal of the carbon dust from the micro-voids that were found on the various substrate samples. The use of the SEM revealed that the removable thin film coating worked well removing particulates less than 10 micrometers and getting into spaces that were larger than $3\mu\text{m}$. The SEM visualized what the qualitative tests demonstrated with the dust not being removed from the minute scratches. The SEM showed that the coating could not get into crevasses less than $3\mu\text{m}$ to remove the particles that were wedged there. Another issue seen with the use of the SEM was that in some areas where there were large clusters of carbon particles, when the coating was removed, the weight of the cluster caused the coating to tear. The cluster, covered with a layer of the removable thin film coating, remained on the surface of the sample substrate.

It was thought that the addition of the Kevlar™ fibers would increase the overall strength of the coating. However, in practice this did not occur. The fibers were thoroughly mixed into the coating, however they had a tendency to clump together and did not form

the web-like structure that was imagined. A larger amount of fibers and a different application may help eliminate these problems and produce the desired results. One thought is that the removable thin film coating be applied to the surface first, then, using a sprayer, a larger amount of Kevlar™ fibers be sprayed onto the coating. This would eliminate the clumping and more evenly distribute the fibers across the coating.

The use of the engineered textile did show a visible reduction in the amount of dust present on the sample surface. The textile also had an added bonus of improving the ease of removal of the coating. It was much easier to pull the coating off the surface, after it cured, by pulling on the textile. Quantitative tests are needed to definitively prove that the addition of the textile increased the efficiency of removal of the hazardous contaminant.

Conclusion

The use of a removable thin film coating technique for the decontamination of a machine shop proved to work well in a previous field test. Therefore, more tests were conducted to further investigate if this technique was a viable alternative to traditional methods of decontamination. Overall, all the qualitative tests and the SEM demonstrated that this was an efficient technique for decontamination and could be used as a preventative measure to protect facilities and equipment from becoming contaminated. More research is needed in improving the strength of the coating, to prevent it from tearing when removing large clusters of particulates and improving its ability of getting into scratches in surfaces that are less than 3µm.

Figure 5. Logarithmic graph of the level before and after the use of the removable thin film coating on carbon particles.

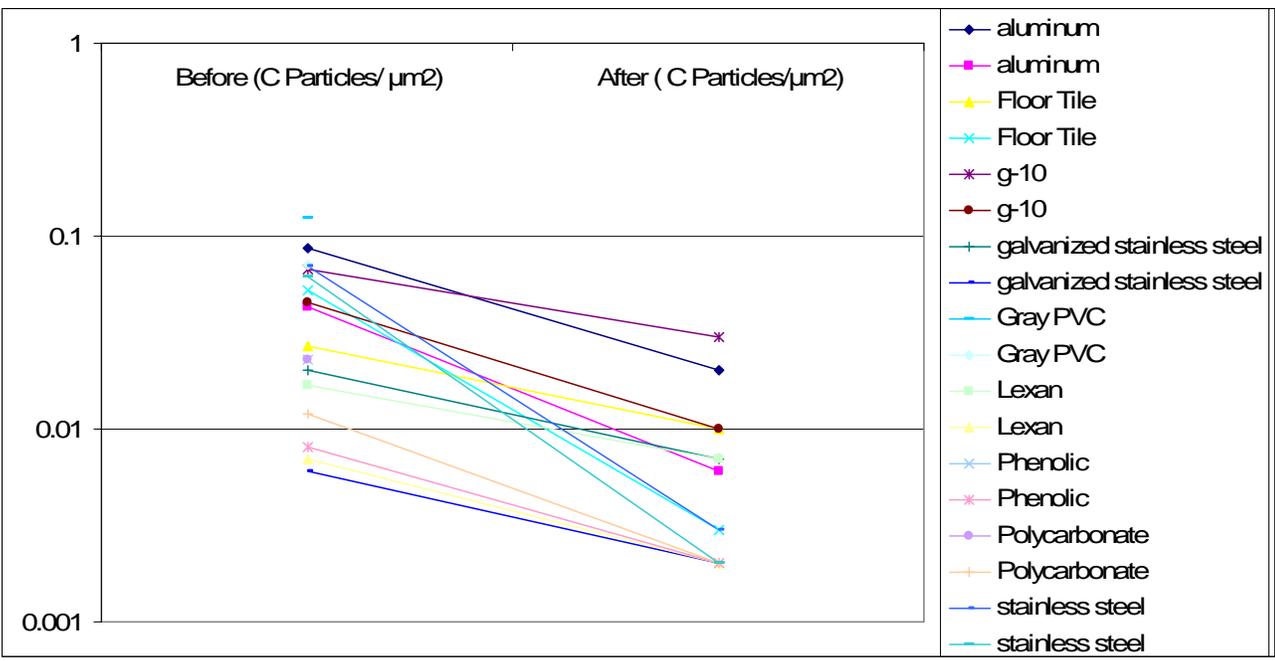


Figure 7. Before and after the use of Product 1 on aluminum at the 21 different locations

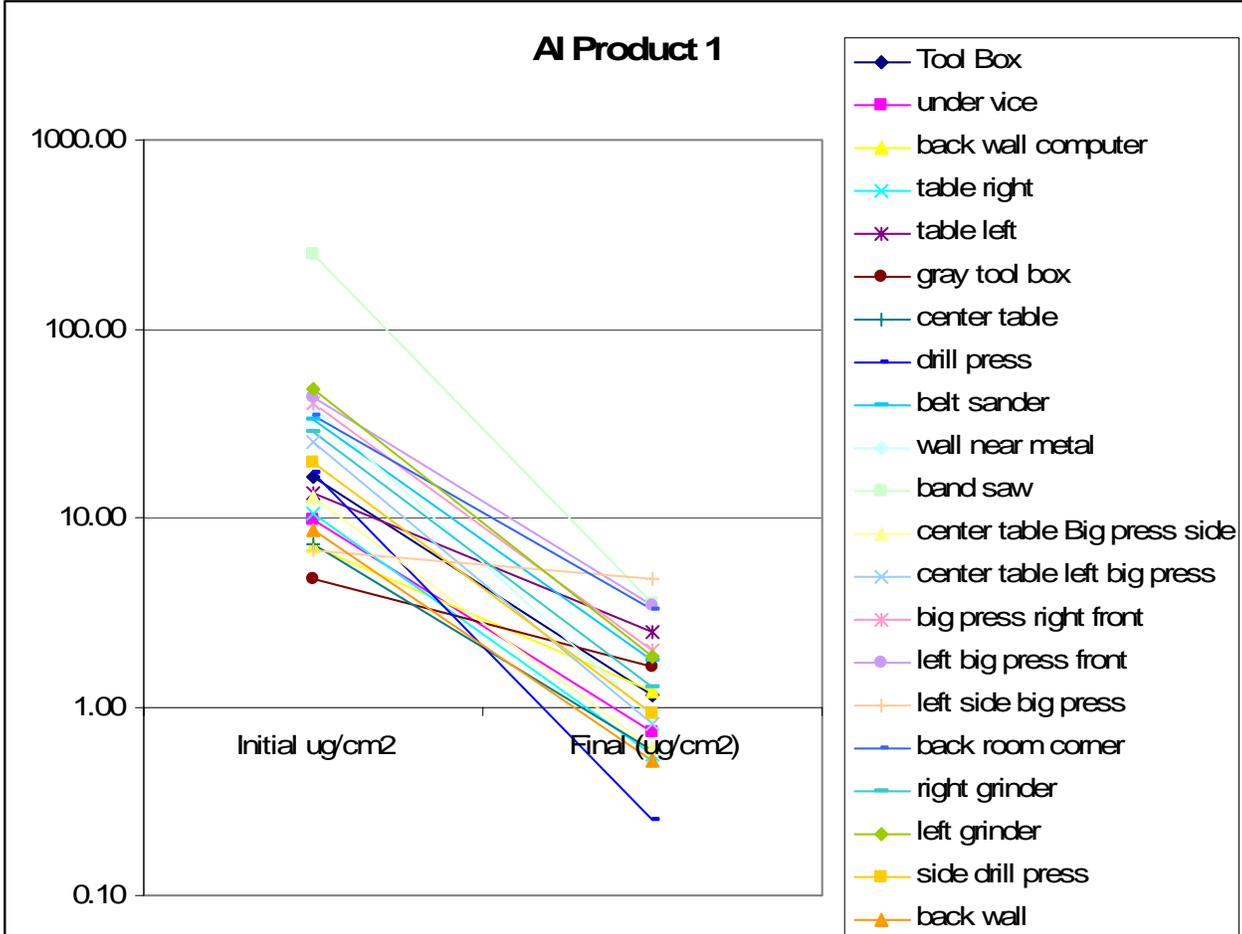


Figure 8. Before and after the use of Product 2 on aluminum at the 21 different locations

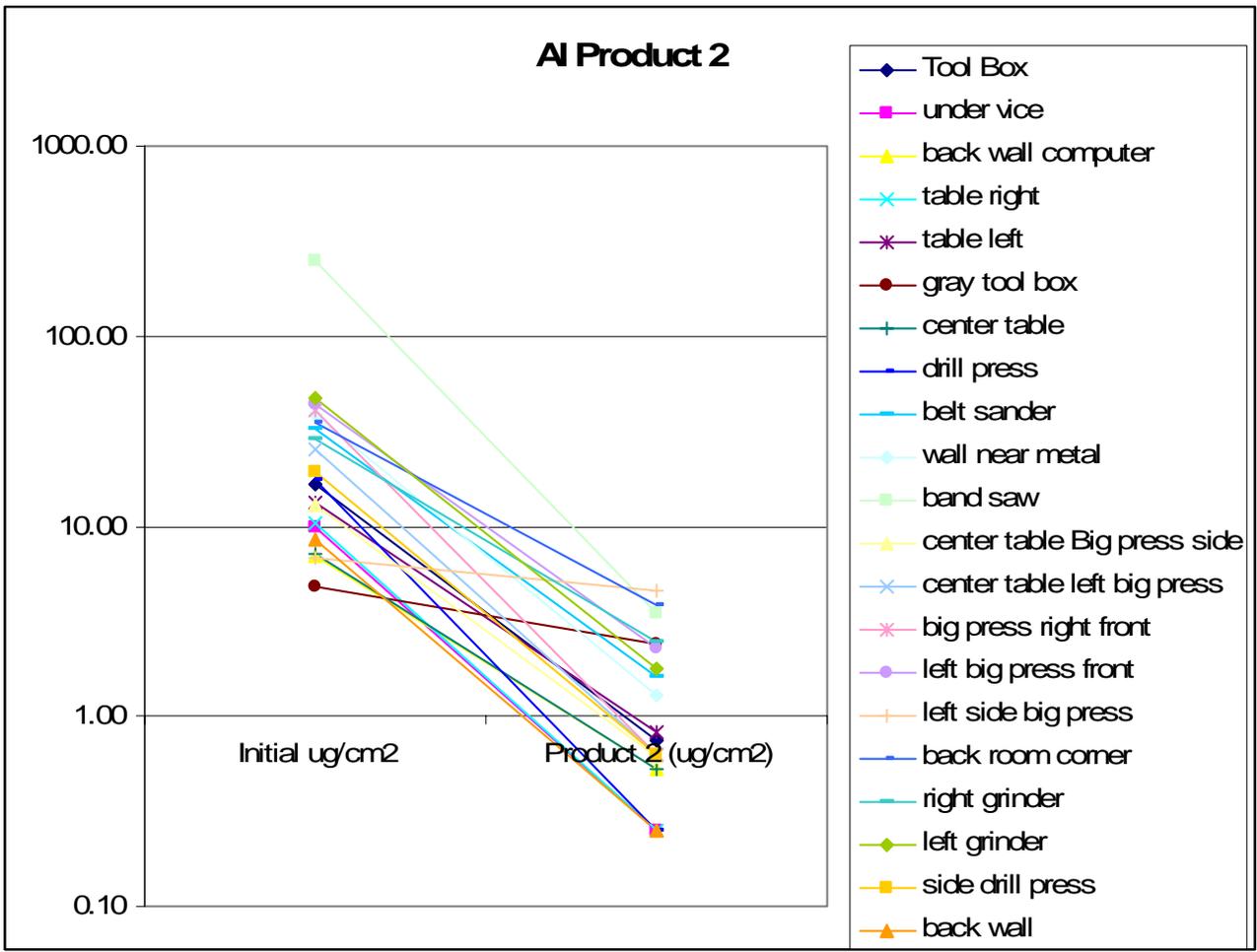


Figure 9. Before and after the use of Product 1 on iron at the 21 different locations

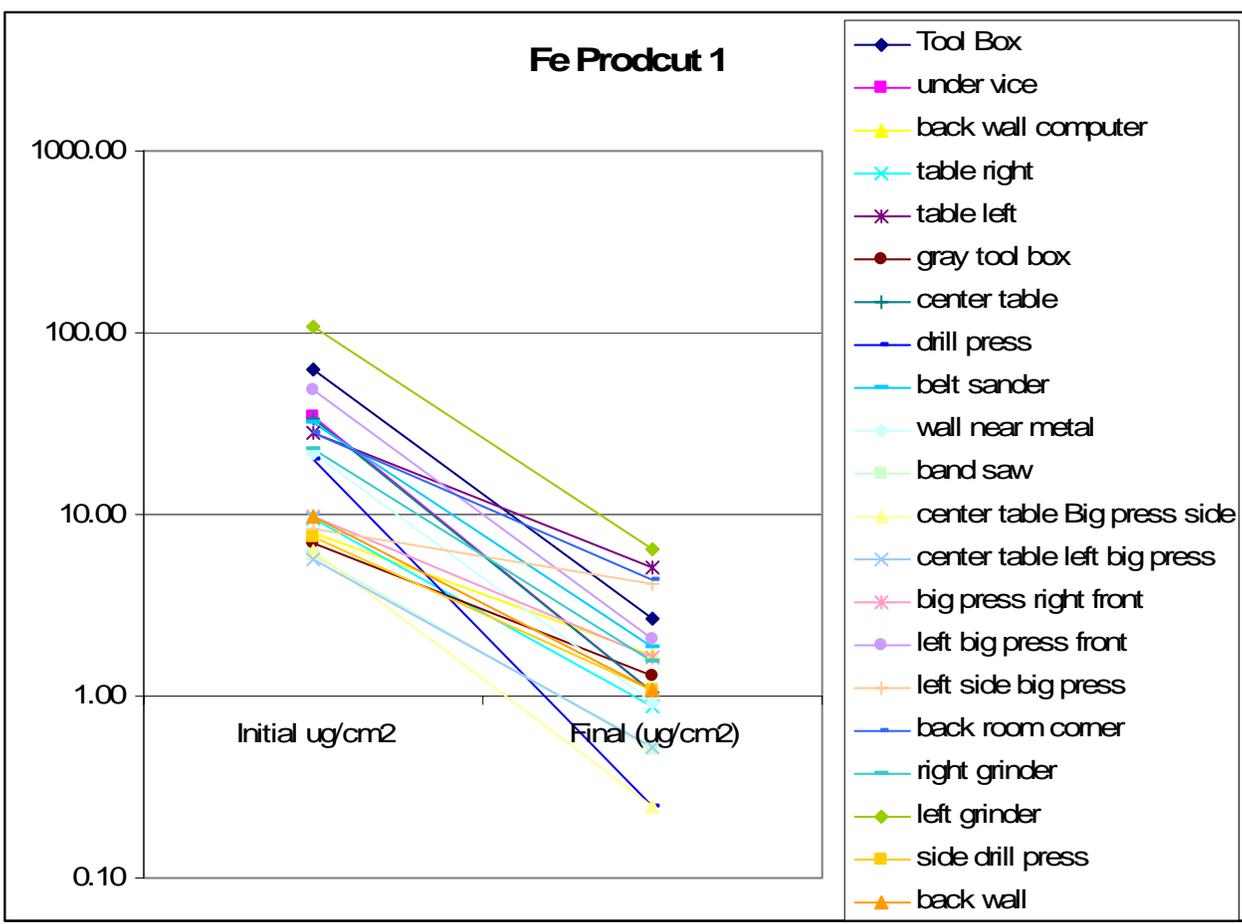


Figure 10. Before and after the use of Product 2 on iron at the 21 different locations

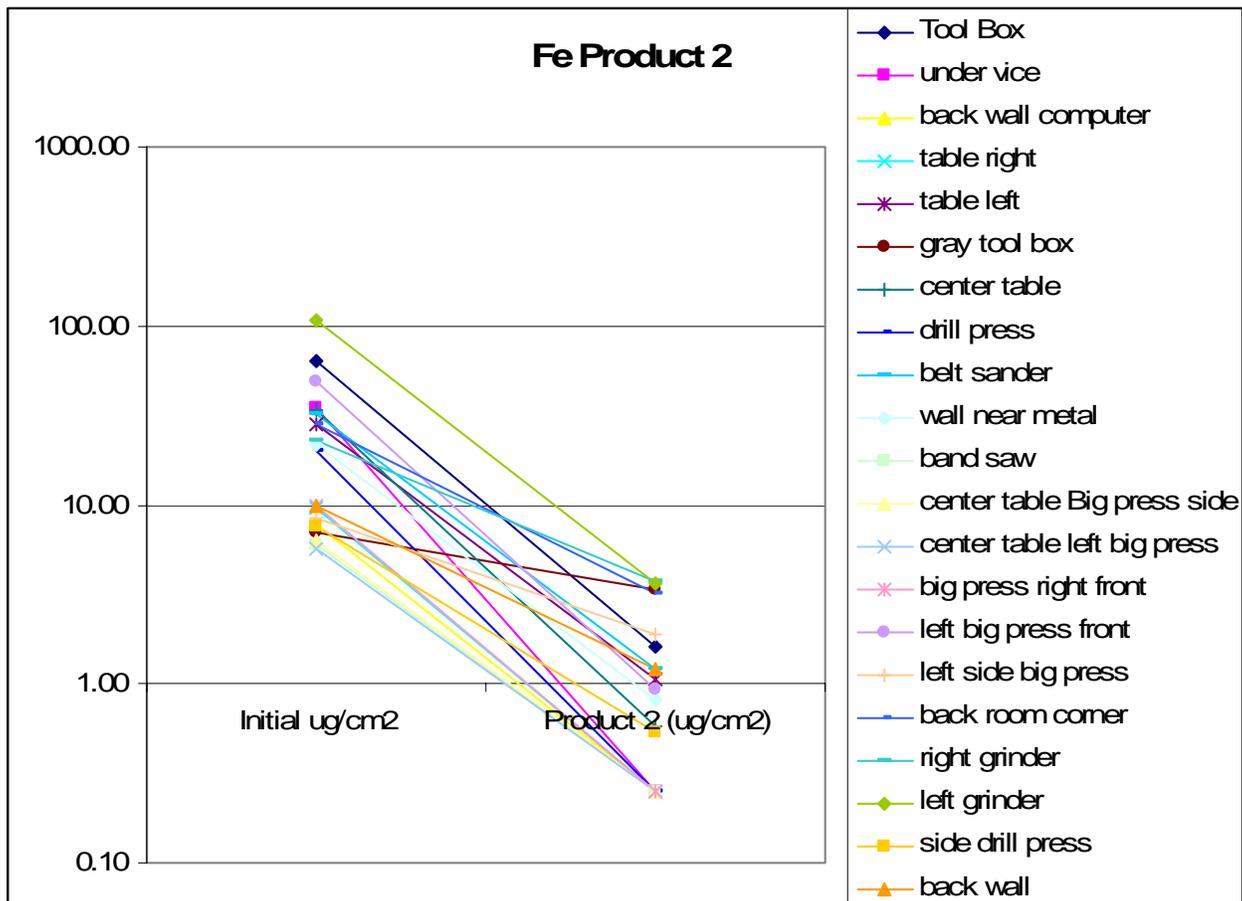


Figure 11. Before and after the use of Product 1 on copper at the 21 different locations

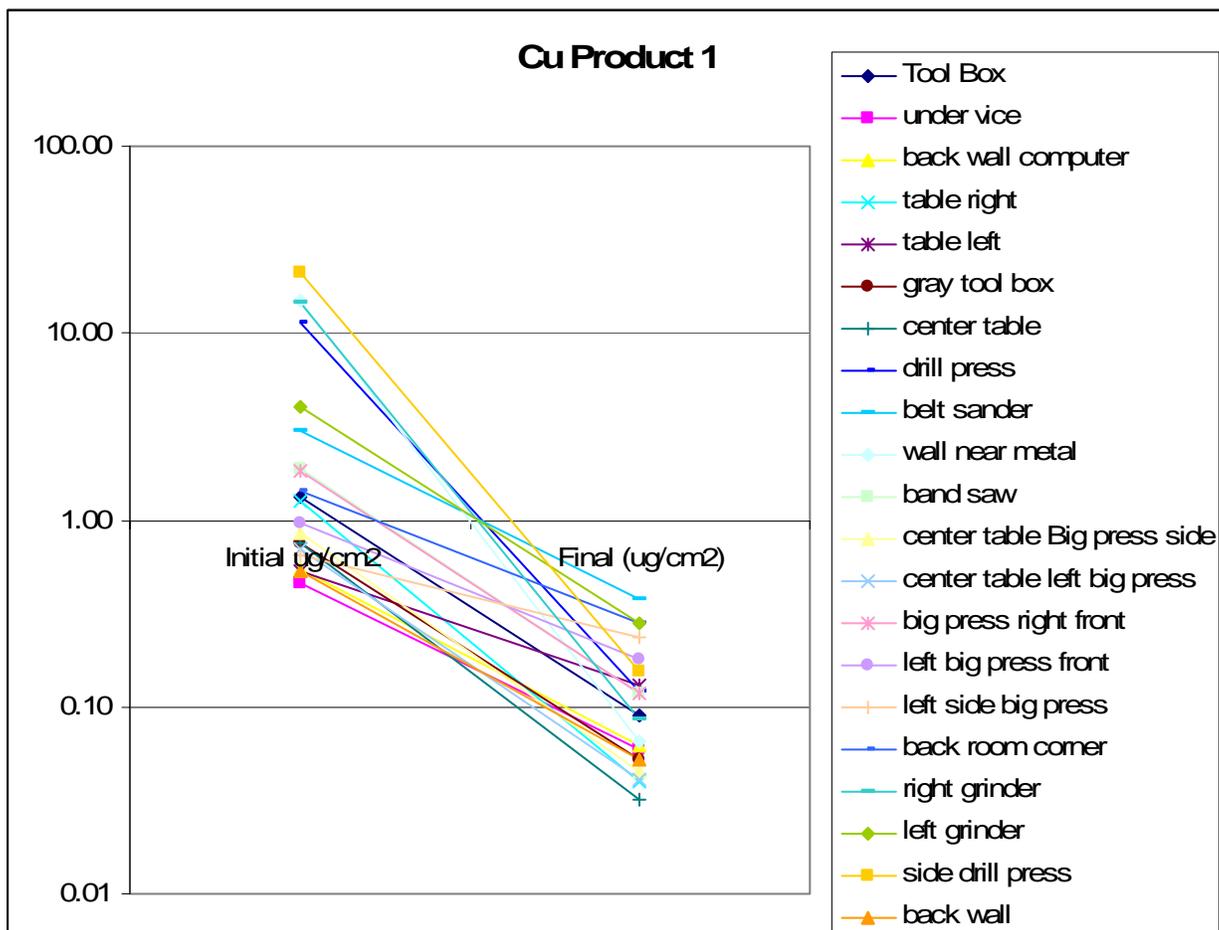
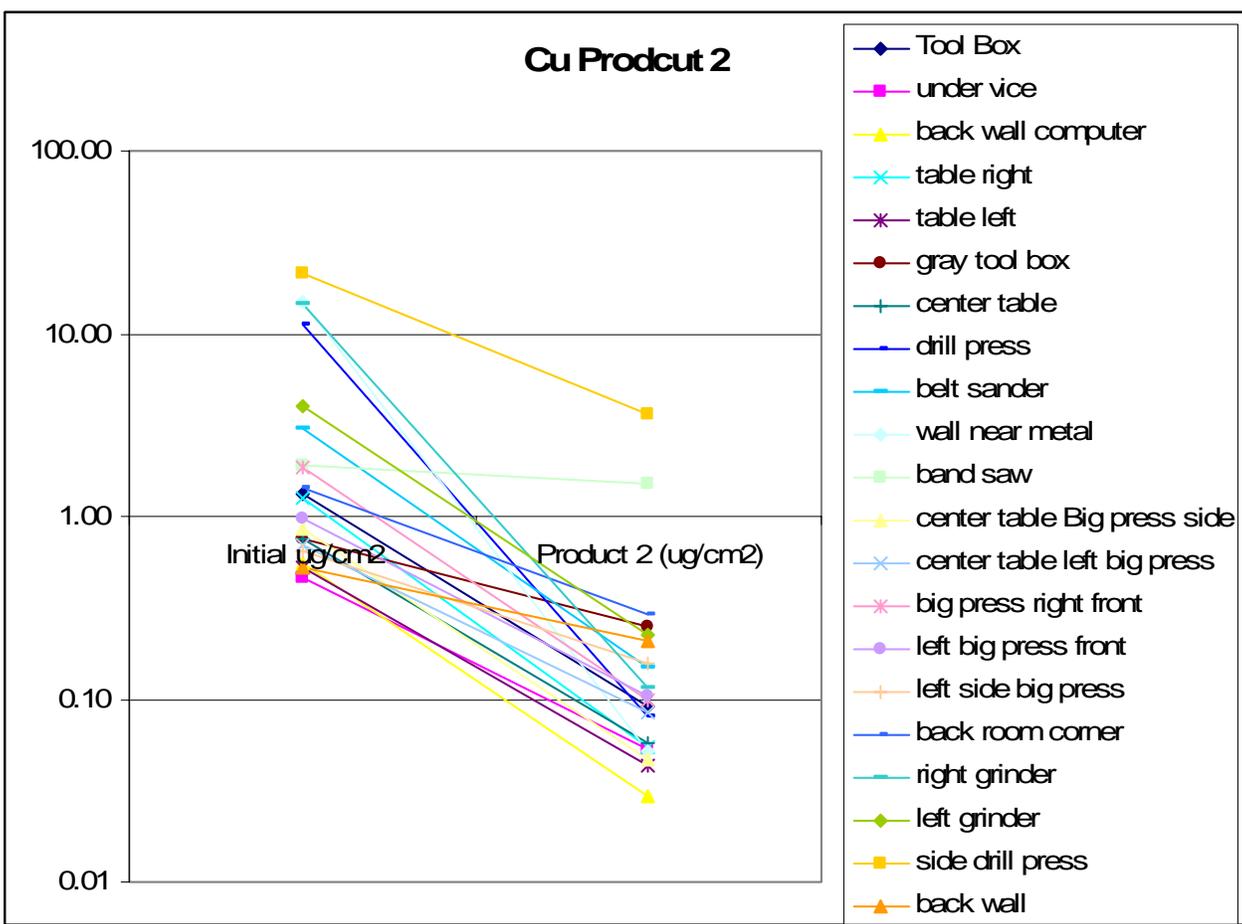


Figure 12. Before and after the use of Product 2 on copper at the 21 different locations



Appendix 1

Product 1: removable thin film coating

Product 2: removable thin film coating incorporated with the Kevlar™ fibers

Aluminum

t-Test: Paired Two Sample for Means

	<i>Initial Levels (ug/cm2)</i>	<i>Product 1 (ug/cm2)</i>
Mean	32.067143	1.639380952
Variance	2612.1629	1.463772048
Observations	21	21
df	20	
t Stat	2.7543656	
P(T<=t) one-tail	0.0061141	
t Critical one-tail	1.7247182	

t-Test: Paired Two Sample for Means

	<i>Initial Levels (ug/cm2)</i>	<i>Product 2</i>
Mean	32.067143	1.46552381
Variance	2612.1629	1.548748662
Observations	21	21
df	20	
t Stat	2.7706971	
P(T<=t) one-tail	0.0058981	
t Critical one-tail	1.7247182	

Unpaired t test between Product 1 and Product 2

P value	0.0807
Are means signif. different? (P < 0.05)	No
One- or two-tailed P value?	One-tailed
t, df	t=1.427 df=40

Iron

t-Test: Paired Two Sample for Means

	<i>Initial Levels</i> (ug/cm2)	<i>Product1</i>
Mean	24.712857	1.94890476
Variance	608.22585	2.81270649
Observations	21	21
df	20	
t Stat	4.4162423	
P(T<=t) one-tail	0.000133	
t Critical one-tail	1.7247182	

t-Test: Paired Two Sample for Means

	<i>Initial Levels</i> (ug/cm2)	<i>Product 2</i>
Mean	24.712857	1.31757143
Variance	608.22585	1.29857076
Observations	21	21
df	20	
t Stat	4.4307982	
P(T<=t) one-tail	0.0001285	
t Critical one-tail	1.7247182	

Unpaired t test between Product 1 and Product 2

P value	0.1973
Are means signif. different? (P < 0.05)	No
One- or two-tailed P value?	One-tailed
t, df	t=0.8543 df=122

Copper

t-Test: Paired Two Sample for Means

	<i>Initial Levels</i> (ug/cm2)	<i>Product 1</i> (ug/cm2)
Mean	3.979667	0.124443
Variance	36.1829	0.009054
Observations	21	21
df	20	
t Stat	2.938095	
P(T<=t) one-tail	0.004066	
t Critical one-tail	1.724718	

t-Test: Paired Two Sample for Means

	<i>Initial Levels</i> (ug/cm2)	<i>Product 2</i> (ug/cm2)
Mean	3.979667	0.347052
Variance	36.1829	0.651972
Observations	21	21
df	20	
t Stat	2.982035	
P(T<=t) one-tail	0.003684	
t Critical one-tail	1.724718	

Unpaired t test between Product 1 and Product 2

P value	0.1084
Are means signif. different? (P < 0.05)	No
One- or two-tailed P value?	One-tailed
t, df	t=1.255 df=40

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Chapter 4

Conclusions and Limitations

Conclusions

The conclusions for the null hypothesis (Ho), that the removable thin film coating technique was no more efficient than the current wet method in the abatement of hazardous particulates, are as follows:

The research in Chapter II rejects the null hypothesis that the removable thin film coating technique was no more efficient than traditional abatement methods. It was found that the removable thin film technique averaged a removal efficiency of 90% of the various metal contaminants at all three locations. Specifically, when the removable thin film coating technique was used to abate lead dust contamination from PPPL's TFTR Test Cell Basement the average removal efficiency was 82% and there was a reduction of the lead dust in the order of one magnitude. The results from the one-tail paired t-test ($p = 0.03$) demonstrated that there was a statistically significant removal of the lead surface dust. Also during this test, the removable thin film technique proved that it is very time efficient. Applying the coating to the contaminating area, letting it cure, removing the coating and bringing the lead dusts levels below the HUD guideline of 50ug/ft² only took about 128 person hours to complete, while the traditional wet method took over 3,000 person hours and did not result in bringing the lead levels below the HUD guideline.

This noteworthy decrease in person hours also decreased the exposure time to the workers reducing the exposure to lead dust.

This technique was also tested at LANL's Old Beryllium Machine Shop. The one-tail paired t-test results ($p = 0.05$) demonstrated that there was a statistically significant removal of the beryllium dust. For the removal of beryllium from the accessible occupied areas; the floors, walls, milling machines, and countertops and the limited access mechanical space; the baghouse, ventilation ductwork, and light fixtures had an average removal efficiency was 88%. The data also demonstrated that there was an average reduction of beryllium dust in the order of two magnitudes.

An active machine shop at PPPL was the final test facility where this technique was tried. Here three different metal contaminants, aluminum, copper, and iron were abated from the machine shop floor. A one-tail paired t-test was computed for each of the three metals to determine if the use of the removable thin film coating significantly reduced the metal surface dust contamination. The results for aluminum ($p = 0.006$), iron ($p=0.0001$), and copper ($p=0.004$) demonstrated that there was a statistically significant removal for each of the different metals. The average removal efficiency of the removable thin film coating for all three metals were very similar; copper 90%, iron 88%, and aluminum 89%. This test also showed that there was an average reduction of each metal dust in the order of one magnitude.

In addition to the removal efficiencies and reduction rate tests for each of the metals at all three locations, a Kendall Tau correlation coefficients was computed to investigate the

probability of removal of the contaminant with the removable thin film coating. At PPPL in both locations, TFTR Test Cell Basement and the Active Machine Shop all of the wipe samples were taken from various locations on the floor for each metal contaminant. The results from these two locations indicated that there was a significant positive correlation between the initial contamination concentration and the percentage of removal efficiency.

The LANL samples were taken from different surface locations throughout the machine shop, not just from the floor. The results of the Kendall Tau b correlation coefficient for the beryllium wipe samples at LANL did not produce a statistically significant correlation between the initial contamination concentration and the percent of removal efficiency.

The reason for these results is that the samples were taken from different surface substrates within the machine shop and the different surfaces varied in smoothness and texture. The removable thin film coating did not work as well on surfaces that contain minute scratches; the coatings could not get into these minute scratches to remove all of the contaminants that were trapped in these areas.

The research in Chapter III initially used qualitative experiments to test the null hypothesis. These experiments demonstrated that the removable thin film coating reduced the amount of visible luminescent dust from various surface substrates, therefore rejecting the null hypothesis. Additionally, these qualitative experiments showed that removable thin film coating technique worked well as a preventative method. It protected the clean surface substrates from becoming contaminated when exposed to the

contaminant. However, during these experiments it was discovered that wherever there were minute scratches, the coating did not remove all of the dust.

Further investigation to determine why the removable thin film coating was not removing the contaminant from minute scratches was conducted using a scanning electron microscope and carbon dust. First the initial carbon dust levels were measured by determining how many particulates were present on the surface area before the use of the removable thin film coating. This was compared to how many particulates were present after the use of the coating for each surface. The results of one-tail paired t-test ($p = 0.00007$) demonstrated that there was a statistically significant removal of the carbon dust. The average removal efficiency was 81%. Next the images of the surface substrates generated by SEM were examined to determine how the removable thin film coating worked at getting into micro-voids and at removing particulates less than 10 micrometers. It was determined that the coating could not get into crevasses less than $3\mu\text{m}$ and was unable to remove any particles that were wedged in there. Furthermore, in some areas where there were large clusters of carbon particles, it was observed that when the coating was removed, the weight of the cluster caused the coating to tear. The cluster, covered with a layer of the removable thin film coating, remained on the surface of the sample substrate.

Once it was discovered that there were some issues related to strength of the removable thin film coating and its ability to remove large clusters of the contaminant an attempt was made to increase the strength of the coating and therefore increase the efficiency of

removal process. The first attempt to improve the strength of the coating included the addition of Kevlar™ fibers to the removable thin film coating. Kevlar™ fibers were chosen because it was thought that the fibers would overlap each other forming a web like structure that would increase the overall strength of the coating. The efficiency of the original formula of the removable thin film coating (product 1) was compared to the efficiency of the removable thin film coating with Kevlar™ fibers (product 2). Twenty-one samples for each of the metals of interest, aluminum, iron, and copper were taken from the floors around the various machinery throughout the shop. The results for product 1 one-tail paired t-tests are as follows: for aluminum ($p = 0.006$), iron ($p=0.0001$), and copper ($p=0.004$). These results demonstrated that there was a statistically significant removal for each of the different metals. The average removal efficiency for all three metals were: copper 90%, iron 88%, and aluminum 89%. The data also showed that there was an average reduction of each metal dust in the order of one magnitude.

For product 2 the results of the one-tail paired t-tests were: aluminum ($p = 0.006$), iron ($p=0.0001$), and copper ($p=0.004$). This demonstrated that product 2 also produced a statistically significant removal for each of the different metals. The average removal efficiency for all three metals were: copper 86%, iron 92%, and aluminum 90% and there was an average reduction of each metal dust in the order of one magnitude.

To determine if at least one of the groups' means was statistically different from the others a Friedman's test was conducted. Based on the p-value ($p<0.0001$) obtained from

this test, one of the groups' mean was statistically different. Next, to determine if the two products were statistically different from each other therefore, proving that product 2 did in fact improve the efficiency of the removable thin films and eliminated the structural problems an unpaired t-test was computed. However, the results of this test ($p=.197$) revealed that there was no statistically significant difference of the removal efficiency between the two products. The addition of the Kevlar™ fiber neither increased nor decreased the efficiency or the strength of the coating.

One other attempt was made to increase the efficiency of the thin film coating. For this experiment an engineered textile was incorporated into the process. The engineered textile is absorbent and highly porous; the thickness of the textile can vary so it is able to hold various amounts of the removable thin film coating. The dimensions of the textile can be altered to cover part or all of the contaminated area. Qualitative tests were used to determine if the addition of the engineered textile increased the efficiency and the strength of the removable thin film coating. The results of these test revealed that there was an increase in the removal efficiency. There was an additional benefit to the incorporation of the engineered textile; it also improved the removal process of the coating after it cured. The engineered textile increased the strength of the coating, allowing it to be easily removed as one entity with little effort.

Study limitations

There were a few limitations with this study. In Chapter II during the sampling process it was assumed that the contaminants are evenly distributed across each sample location. The locations of the sample areas used to determine the initial concentration of the

contaminants were adjacent to locations where the removable thin film coating was applied. The reason for this assumption was that there was no way to determine how much of the contaminant was removed during the wipe sampling process and it was thought that this technique was the best way to get the most accurate data.

Qualitative tests were used for the research in Chapter III to demonstrate the removal efficiency of the removable thin film coating on various surface substrates, to determine the protective qualities of the coatings in preventing clean areas from becoming contaminated. Qualitative tests were also used to determine if the incorporation of an engineered textile improved the coating's removal efficiency and overall strength.

Quantitative tests need to be conducted for each of these areas to support the findings of the qualitative tests.

Another limitation throughout the entire study was the small number of samples and the lack of repeat samples. Despite these limitations our results demonstrated that the removable thin film coating can be used successfully as a decontamination technique for hazardous particulates. It was less time consuming and less labor intensive than the traditional decontamination methods.

Despite these limitations the results demonstrated that the removable thin film coating can be used successfully as a decontamination technique for hazardous particulates. It was less time consuming and less labor intensive than the traditional decontamination methods.

Recommendations for future research

Findings from this study support a recommendation for utilizing the removable thin film coating technique instead of traditional decontamination methods for the abatement and mitigation of hazardous particulates. However, more research is needed to improve the formula of the removable thin film coating to increase its ability to penetrate the crevasses less than 3 μ m to remove any contaminants. Also more research is needed to improve the coating's overall strength in order to eliminate the structural issues that are causing the coating to tear during the removal of large clusters of the contaminant.

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EDUCATION

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Invention disclosures: M-729: *“Development of Thin Films for the Mitigation of
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