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Tekstil Yüzeylerinde Nano ve Mikro Ölçekte Fonksiyonel İnce Film Depolanması: Gelişen Teknolojiler ve Uygulamalar

Functional Nano and Micro-Scale Thin Film Deposition on Textiles: Emerging Technologies and Applications

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Derleme Makale / Review Article

FUNCTIONAL NANO AND MICRO-SCALE THIN FILM DEPOSITION ON TEXTILES: EMERGING TECHNOLOGIES AND APPLICATIONS

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ABSTRACT: This paper discusses the emerging technologies to produce uniform films of functional materials on textiles. These include development of nano-coatings and micro-scale coatings using physical vapor deposition, atomic layer deposition, chemical vapor deposition techniques, and layer-by-layer deposition on textile substrates. Functionalities are added by these processes to textiles such as electromagnetic shielding, antibacterial efficacy, heat or UV radiation resistance, electrical conductivity, soil and oil release, self cleaning, chemical resistance and flame retardancy, with minimum effect on the strength, handle or comfort properties.

Keywords: Chemical vapor deposition, layer-by-layer deposition, physical vapor deposition, surface functionalization of textiles.

TEKSTİL YÜZEYLERİNDE NANO VE MİKRO ÖLÇEKTE FONKSİYONEL İNCE FİLM DEPOLANMASI: GELİŞEN TEKNOLOJİLER VE UYGULAMALAR

ÖZET: Bu çalışma tekstiller üzerinde fonksiyonel malzemelerin homojen filmlerini üretmek için kullanılan gelişmiş teknolojileri ele almaktadır. Bu teknolojiler, fiziksel buhar depolama, atomik tabakalı kaplama, kimyasal buhar depolama teknikleri ve çok tabakalı kaplama yöntemlerini kullanarak tekstil yüzeylerinde nano ve mikro-boyutta kaplamaların geliştirilmesini içermektedir. Elektromanyetik kalkanlama, antibakteriyel etki, ısı veya UV radyasyon dayanımı, elektriksel iletkenlik, leke ve yağ tutmazlık, kendi kendini temizleme, kimyasal dayanım ve alev geciktiricilik gibi fonksiyonellikler, bu yöntemlerle, mukavemet, tutum ve rahatlık özelliklerine minimum etki ederek tekstillere kazandırılırlar.

Anahtar Kelimeler: Kimyasal buhar biriktirme, katman-katman biriktirme, fiziksel buhar biriktirme, tekstillerin yüzey fonksiyonelleştirilmesi.

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1. INTRODUCTION

Textiles currently find applications in various industries from medical to aerospace. There is much effort being done to make textiles meet technical specifications for various needs [1]. Studies related to functionalization and modification of textile surfaces have been found in the literature to achieve properties such as heat or UV radiation resistance, antibacterial property, electrical conductivity, soil and oil release, self cleaning, chemical resistance and flame retardancy. Thin films of metals and metal oxides have long been applied to surface acoustic wave devices, solar cells, electromechanical devices and photo-electronic devices. Several techniques have been proposed for the application of nano films and coatings on the textile surfaces to impart special functionalities, such as ionized gas treatments, plasma treatment, sol–gel, polymer dispersions, chemical vapor deposition (CVD), physical vapor deposition (PVD), electroless deposition, electroplating, surface grafting, enzyme modification and atomic layer deposition (ALD). Nano-scaled coatings can be used to coat individual monofilaments with films that are as thin as 10nm in thickness, with improved functionality and durability [2, 3, 4, 5, 6, 7, 8].

Conventional finishing methods such as pad-dry-cure or coating are currently being used to impart antimicrobial, anti UV, self cleaning and flame retardant finishes. These conventional methods are often accompanied by excessive weight add on, loss of feel and drape, reduced comfort, poor durability against washing and loss of mechanical strength. Also environmental issues such as disposal of large quantity of chemicals exist. Therefore, innovative technologies, such as physical vapor deposition, chemical vapor deposition, layer-by-layer deposition technique, have been developed which are different from conventional finishes in that, special functionalities are imparted to the textile surfaces at micro or nano level, without affecting bulk properties [8].

2. THIN FILM DEPOSITION TECHNIQUES

2.1. Layer-by-Layer Deposition (LbL)

Ordered multilayer thin films can be produced by this technique. The substrate may have any size, shape, topography and topology. Stoichiometric control is not required to maintain the surface functionalities. The use of this technique will definitely be improved by the synthesis of polyelectrolytes for layer-by-layer deposition (having controlled charge density, charge location, and functionality), adsorptions from nonaqueous solutions, competitive adsorptions to prepare mixed layers, and chemical reaction of adsorbed layers. This technique is environmentally friendly, cost-effective, relatively fast and easy. Selection of the right polyelectrolytes helps to control the surface functionalities [9].

Layer-by-layer deposition is a unique method developed for the fabrication of thin composite films on solid surfaces; which involves a sequential adsorption of oppositely charged polycations and polyanions to build a series of polyelectrolyte multilayer films. With this method, nanocomposite textile fibers with special functionalities for protective clothing can be prepared. Nanoparticles such as Zn, silver, TiO_2 can also be incorporated to fibers through this technique. In this process, an appropriately charged substrate is immersed in an oppositely charged polyelectrolyte solution and then rinsed. Oppositely charged polyelectrolyte is attracted to the charged surface and bonded by the help of strong electrostatic bonds. Then the substrate coated with a monolayer is treated with the solution of oppositely charged electrolyte solution. Up to 20 ultrathin layers can be deposited by repeating this cycle [8]. Multilayer nanocomposite film deposition was obtained by utilizing Al_2O_3 nanoparticles on the surface of cationic cotton fabrics by layer-by-layer deposition technique in order to improve mechanical, UV-protection and flame retardancy properties for cotton fabrics [10]. Multilayer nanocomposite films were produced by the addition of ZnO nanoparticles on cationic woven cotton fabrics by layer-by-layer deposition technique for obtaining antibacterial efficacy [11].

2.2. Atomic Layer Deposition (ALD)

ALD deposits nano coatings using pulses of gas producing one atomic layer at a time, where the film thickness can be controlled by the number of deposition cycles. Compared to CVD, the rate of deposition is slower in ALD process [8]. ALD has an advantage of precise thickness control at the Ångstrom or monolayer level [12].

By the help of low temperature ALD, thermally sensitive materials such as organic polymers can be processed. Low temperature ALD is used for the polymers for functionalizing the polymer surface, creating unique inorganic/organic polymer composites, and depositing gas diffusion barriers on polymers. Until the last few years, ALD technique has not been used for polymers due to the temperatures needed for ALD systems, which cause the decomposition of the polymers [12].

Surface coating of textiles with Al_20_3 and ZnO thin films via atomic layer deposition technique has been performed and this method has provided high conformability with precise thickness control by atomic layer deposition reaction cycles, highly uniform coatings on complex surface geometries and low operating temperatures [13, 14]. Jur et al. [15] studied the atomic layer deposition of aluminum thin films on polypropylene meltblown nonwovens and cotton woven fabrics and investigated the process temperature dependence of the growth of aluminum oxide layer on nonreactive and reactive polymer surfaces [15]. Hyde et al. studied the atomic layer deposition of aluminum oxide on polypropylene nonwoven and cotton woven fabrics to control the fiber surface wetting properties [13].

2.3. Plasma Coating Approach

Ultra thin polymeric layers on textiles can be formed using plasma methods; either PVD (physical vapor deposition) or PECVD (plasma enhanced chemical vapor deposition) [8].

2.3.1. Physical Vapor Deposition (PVD)

PVD process or sputtering is similar to CVD, except that in PVD process, the precursors used are in solid form [8]. The PVD process which is carried out at low temperatures is performed with plasma under very low pressure. Therefore, not much chemical reaction exists at this low temperature [16].

PVD is a vaporization coating process where an atom-by-atom or molecule-by-molecule transfer of material from the solid phase to vapor phase is involved, followed by the deposition on to the textile surface. The resultant is a thin, strong and continuous film. Plasma can be used to achieve sputtering of the solid surface. PVD techniques have been used to produce protective coatings such as corrosion-resistant and wear-resistant coatings and coatings for sensor applications [7, 8]. Textile functionalities obtained include electrically conductive, optical, magnetic and biocompatible materials for a wide range of applications [7, 17, 18]. Coatings on polymeric materials by PVD methods have been found to improve the surface properties without altering the bulk properties [10-13].

Coatings by PVD offer a number of advantages over conventional textile coating methods [1, 4, 5, 7, 19, 20, 21, 22, 23, 8];

- Environmentally friendly technique for functionalization of textile materials,

  Dry process and no need to dispose of liquid waste,

  Solvent-free process,
- Uniform and compact deposition,
- Shorter process duration,
- Strong bonding between the textile substrate and coating (the interfacial adhesion between the coated layer and the fiber substrate),
- Enhanced durability of the plasma deposited metal films while maintaining their tensile strength,
- Deposition at low temperatures for polymer fibers,
- Possibility for different coating materials.

2.3.2. Plasma Enhanced Chemical Vapor Deposition (PECVD)

In PECVD, in order to enhance the coating process, the substrate is exposed to plasma to functionalize the surface before CVD. The same quality of films is achieved while subjecting the substrate to less thermal stress than that in CVD process. Carbon coatings were developed on polypropylene nonwoven fabrics for electromagnetic shielding application using glow discharge plasma and propane-butane in gaseous phase. Nano silica particles (10-100nm) were successfully deposited on polyester nonwoven surface through the use of PECVD [8].

Atmospheric Pressure Plasma CVD (APCVD) offers a number of advantages, particularly for the production-line and the continuous processes such as glass, steel or textile manufacturing. The costs and operating difficulties associated with high vacuum equipment are avoided and the coating may be a continuous, rather than a batch process, avoiding the need to interrupt the line. Schematic view of an APCVD system is shown in Figure 1 [24].

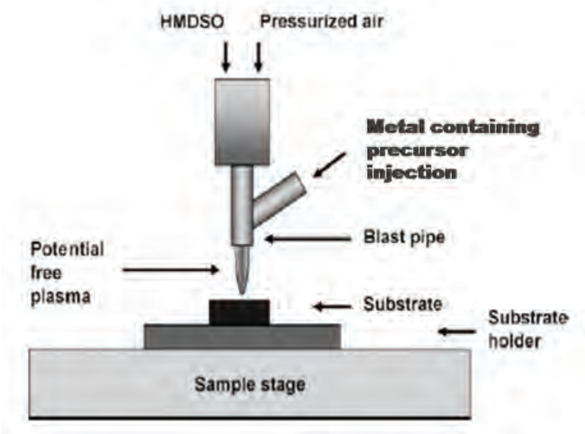

Figure 1. Schematic view of the APCVD system [24]

Conductive Coatings® company produces nickel CVD coated nonwovens in a continuous process for electrical conductivity and electromagnetic shielding [25].

Reactor geometry, gas feed, heat distribution are important for delivering coatings that are uniform across the substrate on a commercial scale [7]. Oxides such as TiO_2 may be added by low-temperature techniques to conventional textiles for imparting properties such as self-cleaning. The production of super-hydrophobic surfaces that will be self-cleaning but will remain transparent has been achieved by thermal CVD of a polymethylsiloxane coating on cellulose [7].

Oxidative chemical vapor deposition (OCVD), a novel technique to acquire higher conductive and uniform polymer layers on a variety of flexible and rigid substrates, was utilized to obtain highly conductive viscose textile fiber coated with poly (3,4-ethylenedioxythiophene) (PEDOT). The PEDOT coated fiber has a potential to be used in smart clothing for medical and military applications, heat generation, and solar cell demonstrators. OCVD is a kind of technique that does not require any solvent to be processed while depositing uniform, thin, and highly conductive polymer layers on different substrates. The polymerization parameters considered in the study were polymerization time, oxidant concentration, dipping time of viscose fiber in oxidant solution, and drying time of oxidant treated viscose fiber. PEDOT deposited viscose fibers that were manufactured by this technique displayed good electrical, mechanical and thermal properties [26].

Nano-scale silver clusters were incorporated into a $Si_xO_yC_zH_w$ (SiOCH) matrix for the treatment of textiles to be used in food industry by Plasma Enhanced CVD. It was found that silver particles as much as 10 nm in size on the surface and inside of the coating structure have provided the transparency of textiles in acceptable limits. With that much of silver particles in the structure, antibacterial efficacy was found to be sufficient by microbiological characterization [27].

3. DEPOSITION OF METALLIC FILMS ON TEXTILES

PVD techniques are commonly used for coating very thin metallic or ceramic films on to a wide range of substrates, either metallic or non-metallic. Wei et. al, studied the interfacial bonding between polypropylene (PP) fibrous substrate and sputter coated copper. Plasma treatment and heat during the sputtering process were found to have an effect on the adhesion of the coating layer to the PP fibrous substrate. It was found that the PP nonwoven itself showed the lowest abrasion resistance among the materials tested and the sputter coating of copper significantly improved its abrasion resistance [1].

Magnetron sputtering was used to obtain the surface functionalization of polyester fiber with nano-scale silver. The silver-coated polyester fabrics showed an excellent UV protection, hydrophobicity (contact angle=132.2°) and antibacterial performance (terminating 99.8% of the Staph and over 99.7% of E. coli bacteria) on the fabric surface [4].

Sputtering deposition of copper, zinc oxide (ZnO) and polytetrafluoroethylene (PTFE) on the surface of meltblown nonwoven having polypropylene (PP) fibers have been studied [5]. While deposition of Cu improved the surface conductivity of the material by reducing the surface resistance significantly, the deposition of ZnO significantly increased the UV absorption of the material displaying the UV shielding effect. On the other hand, PTFE deposition improved the surface hydrophobicity of the nonwoven material by increasing the contact angle due to the chemical nature of PTFE and the increased surface roughness formed by the sputtered PTFE nanostructures. However, Cu and ZnO sputter coatings changed the surface from hydrophobic to hydrophilic. This revealed that the sputter coatings may alter the surface wetting behavior of the nonwoven materials depending on the chemical properties of the deposited substance.

Dietzel et al. investigated the relevant correlations between the surface structures and functionalities of PVD coatings of titanium and zirconium on polyamide fabrics. According to their results, reactive metal deposition caused both chemical and textural changes on the substrate surface due to its etching effect, and a better adhesion was achieved compared to non-reactive metal deposition [28].

Wei et. al. deposited aluminum-doped zinc oxide (AZO) and doped indium oxide (ITO) films by magnetron sputtering onto the polypropylene (PP) nonwovens. The results revealed that ITO had more compact structures on the fiber surface than AZO under the same sputtering conditions and the nonwoven materials coated with ITO had lower electrical resistance than those coated with AZO for the same thickness. The nonwoven substrates deposited with nano-scale AZO showed better UV protection than the substrates coated with ITO for the same thickness [21].

Wei et al. reported a decrease in surface resistance for nonwoven materials with different fiber diameters after sputter coating of copper, where the substrate with finer fibers showed a lower resistance (100 Ω/cm) [29]. Sputter coating of copper on polyester fibers increased the surface roughness of PET fibers as the coating thickness was increased (100 nm) and the increase in coating thickness resulted in a decrease in surface resistivity [30]. Deng et al. [31] reported a decrease in electrical resistance of aluminum-sputtered nonwoven material (2.1 x 10 Ω/cm).

The sputtering of copper on polypropylene (PP) spun-bonded nonwovens was utilized and the surface morphology, pore structures and electrical properties of these materials were investigated. Change in the surface morphology with increased coating thickness was analyzed by AFM technique (Figure 2). Sputter coating by copper altered the pore structures and electrical properties of the material. As the Cu thickness increased, the surface resistivity of the material continued to decrease more due to the uniform distribution of Cu clusters on the fibers. The pore sizes of the nonwoven materials decreased to some extent; however, nonwovens did not lose their porous nature [32]. Körner et al. [33] investigated the surface topography, morphology and functionality of silver containing nanocomposites and Wei et al. [34] studied the morphological properties of the functionalized polypropylene fibers.

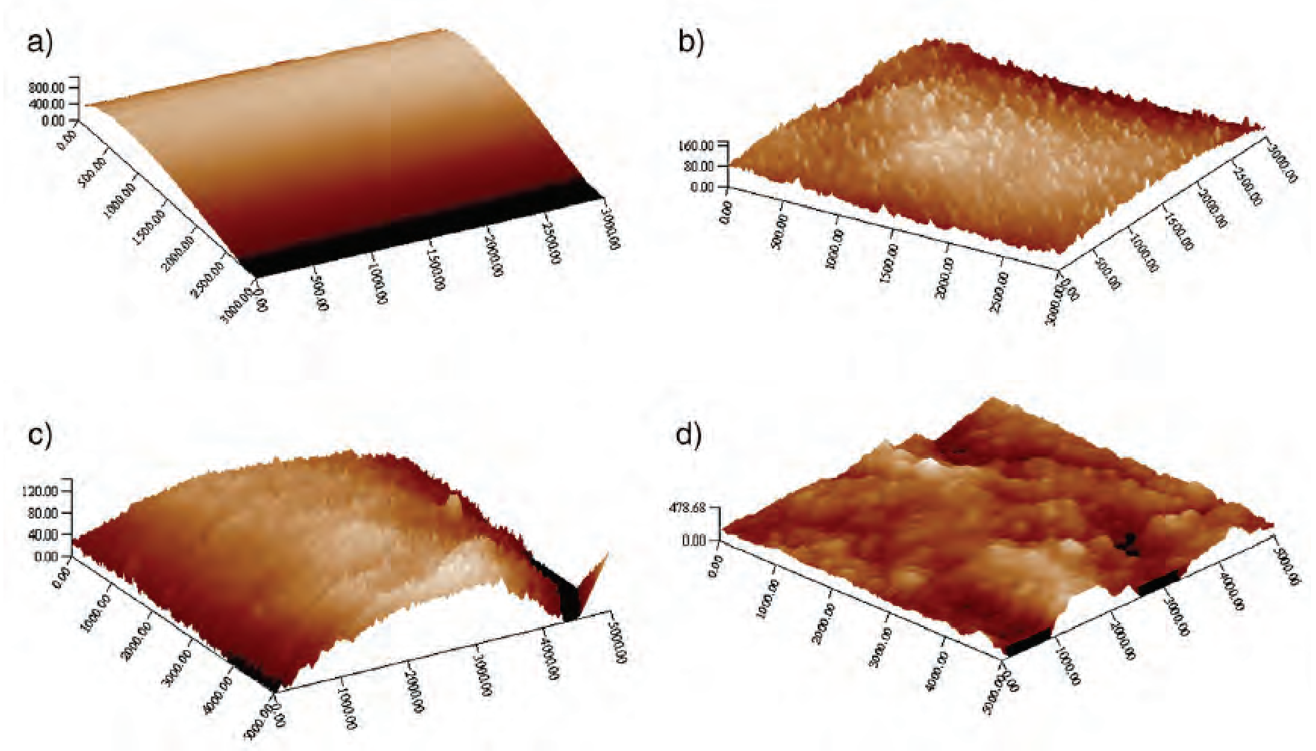

Figure 2. Surface morphology of the PP fiber obtained by AFM: (a) unprocessed fiber; (b) 10 nm coating; (c) 50 nm coating; (d) 100 nm coating [32]

Textile materials mostly made of natural fibers provide a suitable environment for microorganisms to proliferate due to their large surface area and ability to hold moisture within their structure. Textiles used in hospitals, hotels and the clothing of the personnel in these places could easily be infected by the microorganisms. Also, apparels and home textiles such as mattresses, floor coverings and shoe linings used in daily life are susceptible to problems of hygiene. To obtain antibacterial property on textile materials, different classes of chemical compounds such as organo-metallics, phenols, quaternary ammonium salts, and organo-silicones are being used. Many metallic ions and their compounds such as silver, copper, and zinc have also been utilized for this purpose. For these antibacterial agents, it is very important to have safety, non-toxicity and biodegradability besides their antibacterial property [19].

Natural fibers such as cotton are more susceptible to bacterial proliferation than synthetics due to their porous hydrophilic structure retaining water, oxygen, and nutrients creating a suitable environment for bacteria to grow. There are so many antibacterial finishes including copper and other organometallic salts that have been applied on textile materials. Shahidi et al. used DC magnetron sputtering system to deposit copper particles on the surface of cotton fabric samples to prevent proliferation of bacteria, fungi, and germs. Process duration was shorter than the duration of other conventional processes which use nonionic detergent and metallic salts. Antibacterial property of the fabrics was found to be retained after 30 cycles of washing [19].

Sputter coatings of copper and silver were performed on spun-bonded polypropylene nonwovens where the transmittance in the materials was reduced both in UV and visible light ranges. The surface resistance of nonwoven material was also reduced [35]. Wang et al., Sonehara et al. and Jianfeng et al. [36, 37, 38, 39] reported sputtering of several metals onto polypropylene, polyester nonwovens and polyacrylonitrile electrospun nonwovens, and their results showed improved antibacterial performance and electromagnetic interference (EMI) shielding efficiency.

Silver, copper, gold and platinum layers with 300 nm thickness were deposited on SiO_2 fabric by DC magnetron sputtering, and the results revealed that copper was the most effective against bacteria and fungi [40].

Innovative shielding textiles were tried to be obtained by using magnetron sputtering technique. Metallic films including Zn were deposited on polypropylene nonwoven surfaces in argon atmosphere in order to obtain shielding effect as seen in Figures 3 and 4 [41]. Electromagnetic shielding effect of metallic thin films on the woven fabrics consisting of metal/PET filaments obtained by vacuum evaporation deposition technique was investigated. According to the experimental results, it was found that different coating materials have different electromagnetic shielding effectiveness and also increasing thickness was found to increase the electromagnetic shielding effectiveness [42].

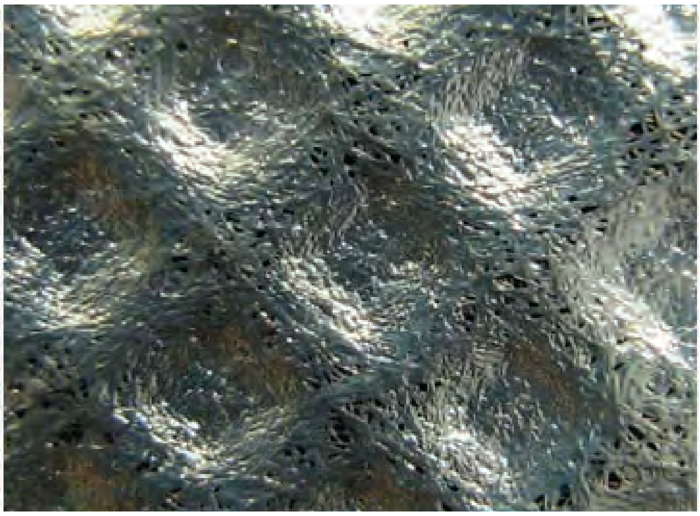

Figure 3. Surface of PP nonwoven with deposited Zn layer [41]

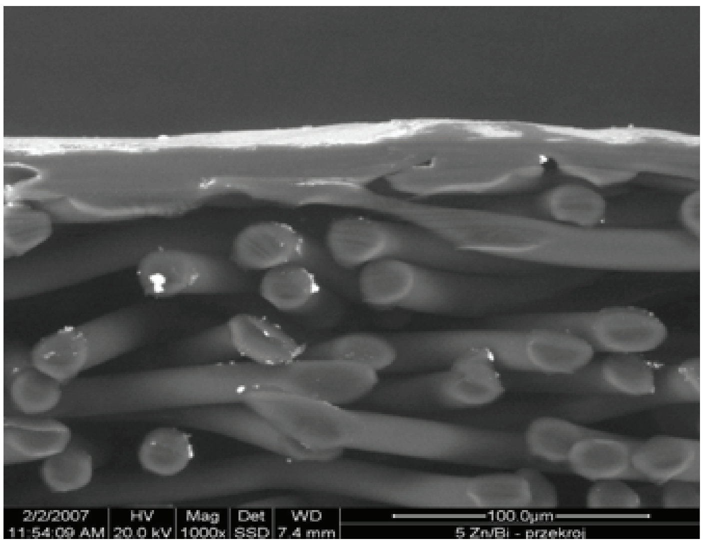

Figure 4. The cross-section of Zn-sputtered nonwoven sample (SEM 1000x) [41]

Coatings by PVD on Nonwoven surfaces at ITU MEMS Laboratory

Our ongoing study includes the deposition of metallic thin films such as silver, titanium dioxide on various nonwoven substrates. SEM images of the preliminary results for copper coatings obtained by PVD techniques on nonwoven surfaces are shown in Figures 5 and 6. The study focuses on functionalization of textile surfaces by imparting antibacterial property for biomedical applications.

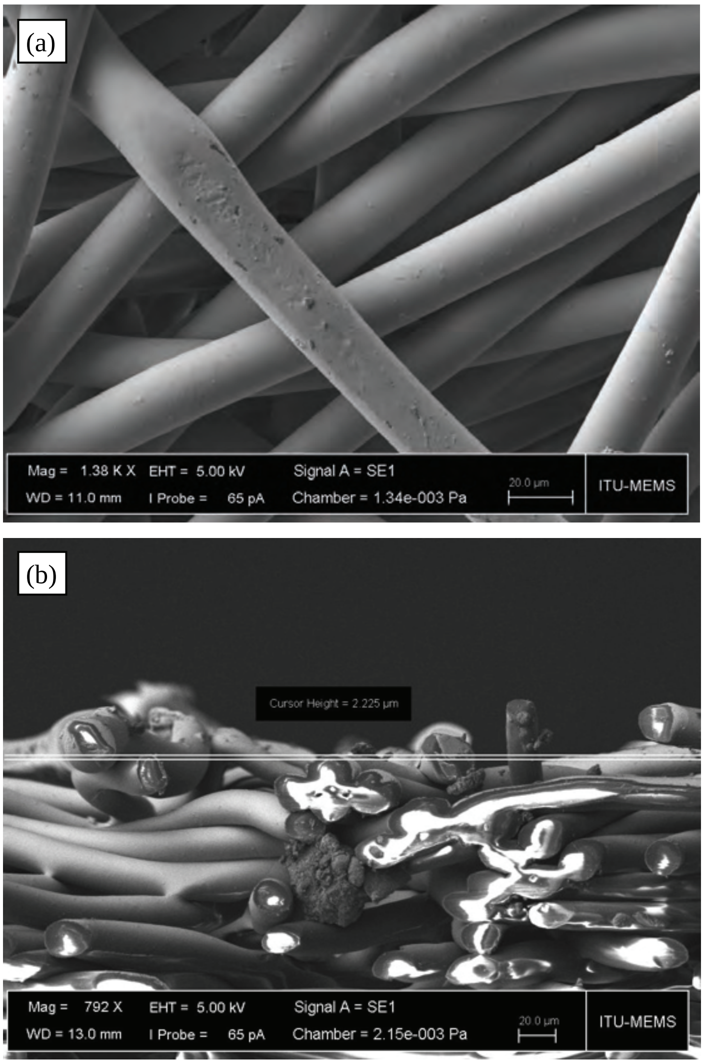

Figure 5. SEM images (a) Copper deposited on nonwoven spunbonded substrate using sputter coating (x1380) (b) cross-sectional image of copper sputter coated nonwoven substrate (x792)

Figure 6 shows the effect of deposition power on the interfacial bonding between the coated copper clusters and fibers within the nonwoven structure. The bonding between the copper clusters and the fibers is improved as the sputtering power is increased from 40 to 80 W.

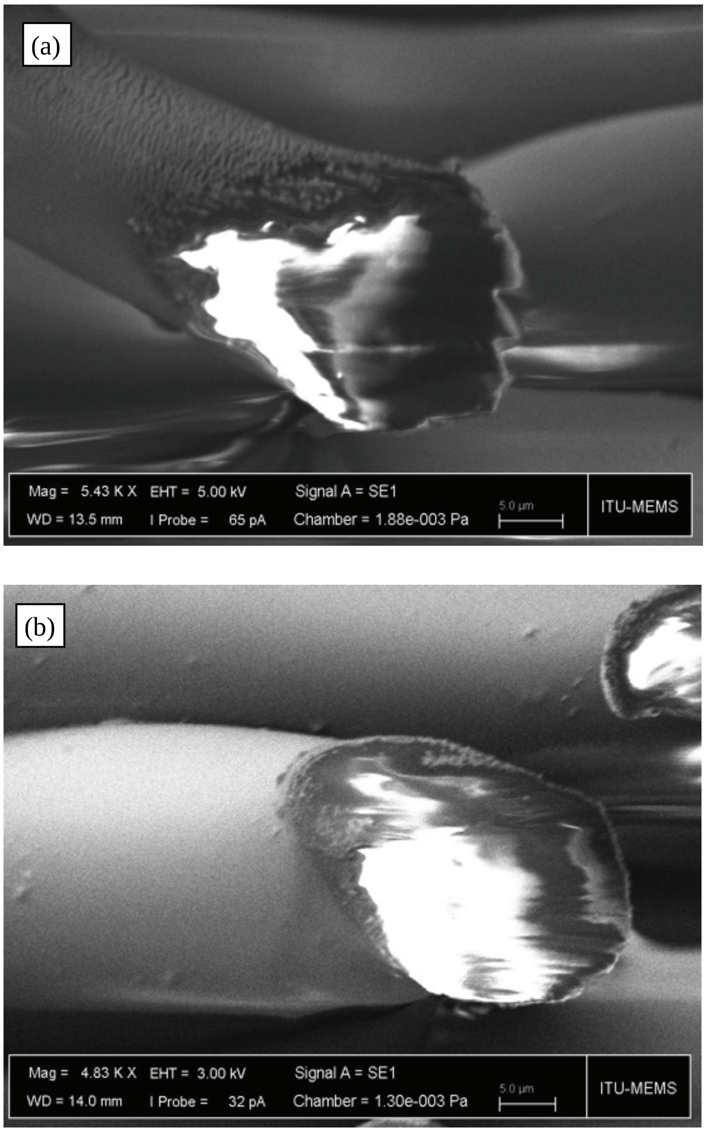

Figure 6. Interfacial bonding observed in SEM (a) 40W sputtering, (b) 80W sputtering

4. CONCLUSION

Textile surfaces with improved functionalities and finishes offer new opportunities for the future of global textile industry. It still remains as a challenge to obtain protection and performance on textile surfaces such as water-proofness, flame resistance, insect repellence, antimicrobial, self cleaning effects, UV resistance, resistance to chemical and biological agents, with minimum effect on the feel, handle, comfort characteristics and strength of textiles. Textile and apparel manufacturers may remain competitive by exploring the emerging technologies in functional finishing area. Durable, eco-friendly and cost effective solutions will be appreciated considering the mass production in the textile and apparel industry.

This review provides an insight for the researchers and industrial textile coating manufacturers about the emerging technologies in the field of producing uniform nano- and micro-scale films of functional materials on textiles. Utilization of these new technologies will help to create diverse application fields for the textiles and provide opportunities for the design of novel functional textile systems.

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