Electron Beam – Physical Vapor Deposition Coating

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ABSTRACT

The Applied Research Laboratory at Penn State is home to iMAST, the Navy ManTech Institute for Manufacturing and Sustainment Technologies. An iMAST/ARL core technology is electron beam – physical vapor deposition (EB-PVD). This industrial scale EB-PVD facility is a unique research facility and capability for developing advanced materials and coatings for a wide range of industrial applications, including turbine, auto, and aerospace. The mission of the core technology is to provide leadership in transferring the technology to private industry for Navy applications. Varieties of ceramic and metallic coatings have been produced by the EB-PVD facility since it became operational in January 1998. A summary of these accomplishments is given below.

The EB-PVD Process

Penn State's industrial pilot Sciaky EB-PVD unit has six electron beam guns, four of which are used to evaporate the coating materials and two of which are used to preheat the substrate to facilitate coating adhesion (Figure 1). Each gun has a 45 kW capacity. The chamber will accommodate up to three ingots ranging in size from 49–68 mm in diameter and 450 mm long. The overall dimension of the production unit is about 1 cubic meter. (The maximum diameter of the substrate—with vertical rotation—that can be accommodated is about 400 mm; it can be rotated at a speed of 5.5–110 rpm with a maximum load of about 100 Kg.) The unit also has a horizontal sample holder with a three-axis part manipulator: two rotary axes of 0–14 rpm and a 0–4,000 mm/min translation axis. It can carry samples weighing up to 20 kg. Since EB-PVD is primarily a line-of-sight process, uniform coatings of complex parts (such as turbine blades) can be accomplished by continuously rotating the part during the coating process.

Advantages of the EB-PVD Process

The EB-PVD process offers extensive possibilities for controlling variations in the structure and composition of condensed materials. For example, the coating compositions can be varied continuously, as in so-called functional graded coatings (FGC). Also, coatings comprised of alternating layers of different compositions can be made. These multilayered coatings can be applied on top of the FGC. The EB-PVD process offers many desirable characteristics such as relatively high deposition rates (up to 150 µm/minute with an evaporation rate of ~10–15 Kg/hour), dense coatings, controlled composition and microstructure, low contamination, and high thermal efficiency. Coatings produced by the EB-PVD process usually have a good surface finish and a uniform microstructure. The microstructure and composition of the coating can be easily altered by manipulating the process parameters and ingot compositions. Thus, multilayered ceramic/metallc coatings can be readily formed, and various metallic and ceramic coatings (oxides, carbides, and nitrides) can be deposited at relatively low temperatures. Even elements with low vapor pressure such as molybdenum, tungsten, and carbon are readily evaporated by this process.

The attachment of an ion beam source to the EB-PVD system offers additional benefits such as forming dense coatings with improved microstructure, interfaces, and adhesion. In addition, textured coatings, which are desirable in many applications, can be obtained. The state of the internal stresses can be changed (i.e., from tensile to compressive) by the forcible injection of a high-energy ion (100–1,000 eV). A high-energy ion beam (as a source of energy) is quite often used to clean the surface of the specimen inside the vacuum chamber prior to coating. The cleaning enhances the bonding strength between the coating and the substrate.

Figure 1. Schematic diagram of the ion-beam-assisted EB-PVD unit.
Success stories
Since the EB-PVD unit became operational, a variety of coatings have been produced for a wide range of applications including turbine, auto, and aerospace.

LOCALIZED REPAIR AND ALTERNATIVE TO HARD CHROMIUM ELECTROPLATING
Chromium (Cr) electroplating is known to be an environmentally hazardous process. The Cr plating process is normally used in areas where wear, corrosion, and oxidation (<600°C) are factors in equipment performance. An environmentally friendly process that provides the same or improved wear, corrosion, and oxidation protection is required. To achieve this goal, two challenges must be met: (1) identify potential candidate materials for the replacement of Cr and (2) develop/identify a process for applying the candidate coating materials. The selection of the alternative coating processes will depend upon the application and requirements including surface finish, wear-resistance properties, fatigue, and thermal properties.

A unique characteristic of EB-PVD is that it meets the above-mentioned challenges and can be used to tailor coatings for specific applications. With the EB-PVD process, corrosion-resistant materials can be applied economically on the surface where they are most needed. Applications for these coatings range from brass lighting fixtures to landing gear and other components of aircraft and helicopters. The IBAD EB-PVD process offers additional flexibility in repairing localized damage in landing gears—a task that cannot be done by any other processes without affecting the physical and mechanical properties of the base material. The significant advantage of using IBAD along with EB-PVD is the capability to enhance the metallurgical bonding of the coating with the substrate at a relatively low temperature. For instance, EB-PVD chromium coatings were flaking or debonding from the landing gear when deposited below 280°C (<550°F). Perhaps this condition was due to poor metallurgical bonding and high residual stresses in the deposit. When the same Cr coating was applied on the landing gear along with argon ion bombardment, the resulting coating had a dense microstructure with good metallurgical bonding between the base material and the electroplated chromium (region b of Figure 2b).

Figure 2a is a photograph of helicopter landing gear showing localized surface damage that needs to be repaired. The current repair process is the chemical stripping of the chromium coating followed by replating, baking, and machining of the entire component. Localized refurbishment of the landing gear (Figure 2b) was successfully demonstrated by applying Cr in the ion beam assisted, EB-PVD chamber. The SEM micrograph shows that the EB-PVD Cr deposit is relatively denser than the electroplated Cr which has spacings/voids between the grains (region a and b, respectively shown in Figure 2b). Unlike thermal spray coatings, these deposits had a dense microstructure with good metallurgical bonding with the base metal. In addition, microcrack sealers are not required. The cost saving is expected to be more than 20–30 percent with improved component life and performance.

THERMAL BARRIER COATINGS (TBCs) FOR THE TURBINE INDUSTRY
There is a continuous thrust in improving the life and performance of turbine components under severe environmental conditions.
including erosion, oxidation, and corrosion.\textsuperscript{4,5} The life of components has been increased by applying TBCs by either plasma spray or EB-PVD processes. Each process has advantages and disadvantages (Table 2). For instance, the thermal conductivity of the plasma-sprayed TBCs is about 60 percent lower than the bulk value, due to presence of porosity between the laminated grains. In contrast, the thermal conductivity of TBCs applied by EB-PVD is higher than the plasma spray coatings. The higher thermal conductivity in the EB-PVD TBCs is due to limited porosity present in the individual columnar grains. The porosity in TBCs is mainly present between the columnar grains (Figure 3). In spite of higher thermal conductivity, TBCs applied by EB-PVD are preferred over plasma-sprayed TBCs due to their smooth surface finish, better strain tolerance, better erosion–resistance properties, and improved metallurgical bonding with the substrate. In spite of significant advancement in the EB-PVD coating technology, there are still challenges in producing low thermal conductivity EB-PVD TBCs. Future thrusts are in producing a low-conductivity EB-PVD TBC by altering the microstructure and creating micro-porosity within the columnar grains, or in providing new materials with different compositions and functional graded coatings.

COATINGS FOR MACHINING TOOLS AND FORGING DIE INDUSTRIES
Metallic borides, carbides, nitrides, and oxides have long been known to be very hard, wear-resistant materials. Applying these hard coatings to tool steels and tungsten carbide–cobalt cutting inserts can increase the tool life by several hundred percent (400–600 percent), reducing costs associated with tool procurement, set-up time, and machine downtime. Wear-resistant coatings are often characterized as having high melting temperatures as well as high hardness values. However, it is important to note that the coating performance depends on several factors including coating material, the deposition process, and machining conditions. The deposition process and parameters often dictate the coating’s microstructure features (i.e., grain size, degree of texture, density, etc.) and thus its properties.

The machining of titanium and nickel-based alloys is very difficult and challenging. To improve the efficiency and life of cutting tools, titanium nitride and titanium carbide coatings have been deposited by reactive ion-beam-assisted electron beam–physical vapor deposition (RIBA EB-PVD). By evaporating titanium metal and mixing the vapor with an ionized argon and nitrogen (nitride) or acetylene (carbide) mixture near stoichiometric compounds with high Vickers’ hardness values (TiN: 2500 VH\textsubscript{0.050}, TiC: 3500 VH\textsubscript{0.050}) were produced. These high hardness values are attributed to the high degree of texturing. Tailoring the processing conditions in order to achieve a specific orientation of the grains (and crystal structure) will increase the hardness as well as the wear–resistance of the coating.

Figure 4 shows a scanning electron micrograph of the textured surface morphology of titanium nitride grown at 0.1 μm/min on 304 stainless steel at 650°C, -100 volt D.C. bias, and nitrogen ion bombardment. The high degree of surface texturing of the TiN is explained by the preferred orientation of certain planes within the TiN unit cell. Several TiC coatings were deposited by RIBA EB-PVD under identical processing conditions with the exception of the level of ion bombardment (27 μA/cm\textsuperscript{2} – 163 μA/cm\textsuperscript{2}). The hardness values ranged from 1721 to 3504 VH\textsubscript{0.050}. The general trend shows that the hardness of the TiC coating increases with increasing levels of ion bombardment, as shown in Figure 5. The generally accepted bulk hardness value of TiC is 2200–2800 VHN. The TiC coating deposited with 162 μA/cm\textsuperscript{2} (3504 VH\textsubscript{0.050}) showed over 25 percent improvement in the hardness value. This increase in hardness can be attributed to a more dense film, texturing, and/or stress.

To improve the cutting performance of WC–Co cutting tools during the machining of titanium and nickel-based alloys, TiB\textsubscript{2-x} coatings were deposited by direct evaporation under various processing conditions. Direct evaporation of TiB\textsubscript{2} yielded a TiB\textsubscript{2-x} coating with a hardness of 2940 VH\textsubscript{0.050}. The hardness of the coating was increased to 3041 VH\textsubscript{0.050} by argon ion beam assist deposition. The hardness was increased further by 15 percent by both applying a negative bias and bombarding the TiB\textsubscript{2-x} growing film with argon ions during deposition. The resulting surface morphology shows a uniform, fine-grained surface with an average grain size less than 100 nanometers.

![Figure 4](image1.png) SEM micrograph showing the surface morphology of TiN coating deposited on stainless steel substrate by IBAD EB–PVD.

![Figure 5](image2.png) Plot showing hardness increase in TiC coatings produced by IBAD EB–PVD as a function of ion beam current density.
Figure 7 shows the fractured surface of a TiB$_{2-x}$/TiC multilayer deposited on a WC-Co cutting tool. The TiC (grown by RIBA, EB-PVD) serves as a bond coat as well as to prevent chemical reactions between the TiB$_{2-x}$ and substrate material. The interface between the TiC and TiB$_{2-x}$ looks very good. The TiC has columnar structure whereas the TiB$_{2-x}$ shows a very fine-grained microstructure. Similarly, multilayered coatings composed of TiC/Cr$_2$C$_2$ have been successfully produced by the co-evaporation of the Ti, Cr, and graphite ingots in the EB-PVD chamber (Figure 1). The future thrust is in developing superhard multilayered coatings composed of Al$_2$O$_3$/TiC and of TiB$_2$/TiC/ Cr$_2$C$_2$ by the ion beam-assisted EB-PVD.

**Summary**

Electron beam-physical vapor deposition offers high flexibility in depositing ceramic and metallic coatings for a wide variety of applications ranging from microelectronic to turbine industry. Attachment of an ion source to the EB-PVD unit offers additional benefits in depositing coatings at a relatively lower temperature. Processing parameters control the microstructure, physical and mechanical properties, and residual stresses present within the coating. Chromium was successfully deposited at a relatively low temperature ($\approx 550^\circ$F) on a damaged landing gear. The hardness of TiC and TiB$_{2-x}$ was increased by 35 percent and 15 percent, respectively by controlling the process parameters and bombarding the growing film with ionized gas.