Directed Vapor Deposition: Low Vacuum Materials Processing Technology

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Abstract

Directed vapor deposition (DVD) is a recently developed electron beam-based evaporation technology designed to enhance the creation of high performance thick and thin film coatings on small area surfaces (generally 100 cm^2 or less). DVD technology development has been driven by a desire to combine four processing capabilities into one industrially appealing system. These capabilities are: 1) very high rate deposition (5 μm/min and higher over a 100 cm^2 area), 2) very high material utilization efficiencies (on 100 cm^2 areas, efficiencies should at least triple that of other coating technologies), 3) precise control of growing film atomic structure, and 4) highly flexible definition of growing film atomic composition. These criteria have led to the development of a unique plasma-enhanced electron beam evaporation tool which will be described here. Initial experimental and modeling results will also be presented to demonstrate how the selected technology solution is allowing the desired processing features to be achieved.

Keywords: directed vapor deposition, DVD, high rate evaporation, low vacuum materials processing, electron beam evaporation

Introduction

For many years vapor deposited coatings have been recognized as valuable components of numerous engineered consumer products. Such coatings are regularly incorporated into products to provide functionality unachievable through other means. Sometimes the coatings act as a barrier, e.g. environmental protection or surface wear resistance. In other instances they play roles as active components in the engineering system, e.g. converting the sun's energy to electricity or storing bits of computer information in magnetic domains. In all cases, introduction of the vapor deposited coating is motivated by some product specification which cannot be met through other means. Engineers recognize that each coating makes it possible to deliver the specified product with the required performance characteristics. At the same time, they know that use of the coating adds to the cost of the product. The competition between performance need and product cost drives the search for new vapor deposition technology which can provide desired performance at a lower market cost.

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Recently, the development of a new coating technology, directed vapor deposition (DVD), has been motivated by this drive to reduce the cost of high performance coating synthesis. For small area surface coating, DVD development has sought to impact the cost of coating by increasing the rate of coating. Specifically, DVD design has attempted to increase product throughput by raising deposition rate ($\mu m/(min \cdot cm^2)$) and material utilization efficiency (for nonreactive deposition: mass evaporated/mass deposited). For many applications, rapid, efficient coating is critical to the economic viability of the production process. As an example, Fig. 1a) illustrates the coating of engineered 140 μm diameter fibers in a standard electron beam physical vapor deposition (EB-PVD) system [1]. The economic viability of this process is highly dependent upon the ability of the EB-PVD technology to place vapor from its rapidly diverging vapor stream (inherent to the process) onto the small area of the fiber surface. This type of product manufacture appears to be a prime candidate for a faster, more efficient coating process. Development of DVD has been motivated by a desire to provide such an alternative.

Design of DVD has also been motivated by a need to create coatings that deliver the advanced product performance specified by designers in many applications. Coatings are often added to a product to boost performance to a specified level, and a coating's ability to deliver such performance is frequently dependent upon its composition and structure. Composition and structure in turn are imparted by the material processing characteristics of the particular deposition technology selected for coating synthesis. If the deposition technology used has a difficult time generating the needed composition and structure, then the company producing the coating could have trouble offering a competitive product. For example, consider Fig. 1b) which shows an EB-PVD system coating a surface with a multielement (alloy) film. Schiller and others have shown that when the vapor pressures of coating elements are significantly different, EB-PVD systems must draw the coating elements from separate sources to produce stoichiometrically-correct deposits [2]. In EB-PVD coaters, this can be a highly inefficient process in which much of the vapor from the separate pools cannot be used to coat the substrate surface with the correct, mixed composition. Even in those areas where the composition is approximately correct, sophisticated coating surface motion is probably required to produce composition mixing, and the result might be a process of marginal economic viability.

The Technology of Directed Vapor Deposition

Keeping in mind the types of technology shortfalls described above, researchers at the University of Virginia have developed a new deposition technology that builds upon some of the latest vapor deposition equipment innovations. The university's new technology, directed vapor deposition (DVD), has been designed to offer an alternate method for coating small area surfaces with high performance coatings. It has been designed to rapidly and efficiently deposit metals and ceramics of widely varying atomic composition and structure onto surfaces generally less than 100 cm^2 in area. DVD has been under study and development since the early 1990's and is now reaching the point where industrial application appears possible in the foreseeable future. Early development of the technology has been described elsewhere [1, 3, 4].

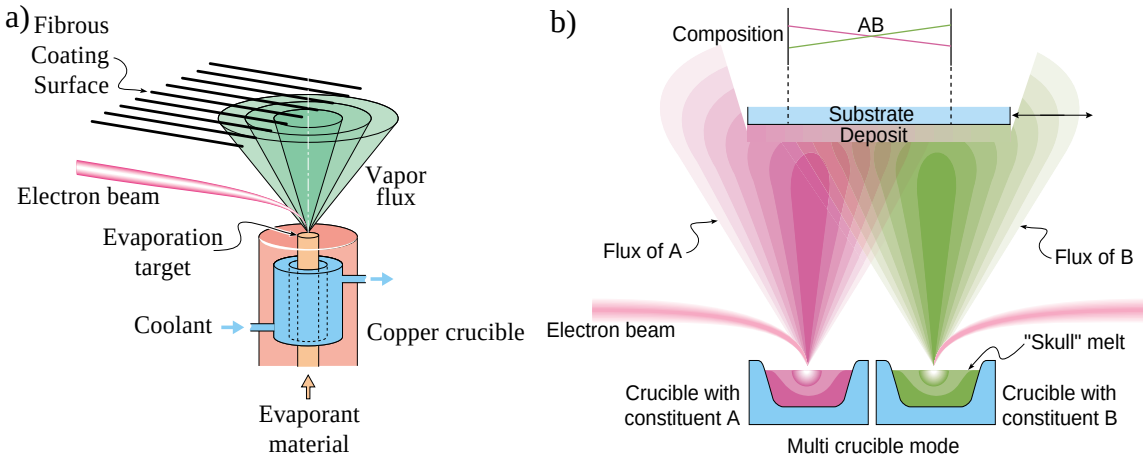

Figure 1 a) Fiber coating in conventional EB-PVD generates a large amount of vapor that does not end up on the small diameter fiber surfaces. b) Alloy creation in conventional EB-PVD is often difficult due to the limited region of composition overlap between adjacent vapor streams.

During the summer of 2000, the Fraunhofer Institute for Electron Beam and Plasma Technology (FEP: Dresden, Germany) has delivered a second generation DVD system to the University of Virginia. That system is the focus of this paper. The second generation DVD system combines the university's research-based design ideas for DVD with many FEP vapor deposition equipment innovations. The result is expected to be a tool capable of high deposition rates, high material utilization efficiencies, and precise control of depositing atomic structure and composition. To realize these challenging expectations, the system has incorporated four major technology components into its design: 1) advanced electron beam (e-beam) gun technology, 2) low vacuum, flowing gas vapor transport from source to coating surface, 3) plasma activation of the gas and vapor stream in the vicinity of the coating surface, and 4) coating surface electrical biasing (Fig. 2).

DVD begins its coating process by generating an electron beam from its e-beam gun. By focussing the beam's electrons onto a metal or ceramic surface, it is possible to vaporize those surfaces and generate a stream of atoms. This stream of atoms is then used to coat a surface. Electron beams have been recognized for many years as tools capable of rapidly producing large amounts of atomistic vapor in this manner, and an electron beam is used here (instead of sputtering or thermal evaporation methods) to help DVD achieve its first goal, the evaporation (and deposition) of material at high rate, i.e. microns per minute over areas up to 100 cm^2. The electron beam gun package delivered by FEP for this DVD system has also been configured to endow the overall technology package with an ability to tailor coating composition to the precise requirements of a given application. To facilitate composition tailoring, the FEP 10 kW/70 kV e-beam gun has incorporated high speed beam scanning (100 kHz), automatic (closed-loop) beam positioning, and

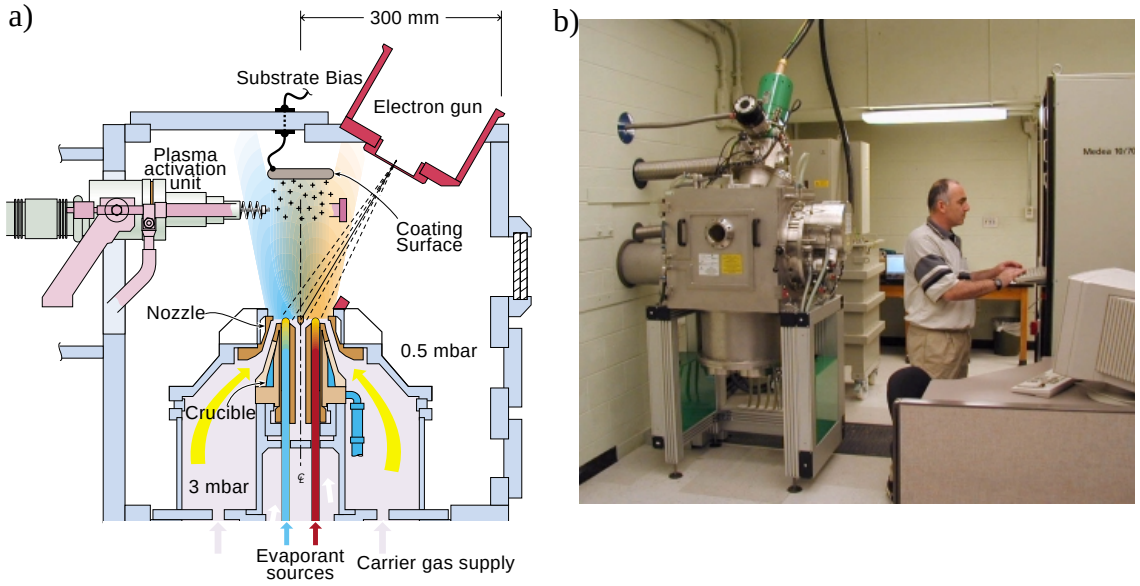

Figure 2 a) The new DVD system uses an e-beam to evaporate material from source pools. The vapor is then transported to the coating surface in a flowing gas. Near the coating surface plasma activation can occur. A coating surface bias can also be used to attract vapor. b) The second generation DVD system.

sharp (0.5 mm) beam focussing. When used with multisource crucibles, these e-beam gun technologies are designed to allow closely spaced vapor streams of distinctly different composition to be generated simultaneously (Fig. 3). When combined with the system's second major technology component (low vacuum, flowing gas vapor transport from source to coating surface) the FEP e-beam gun package is expected to creates a mixed vapor stream of controlled elemental composition. The design details and philosophy of the FEP e-beam gun used in this DVD system are described elsewhere in this volume.

Once the DVD e-beam system generates a vapor stream from one or more crucible sources, a flowing carrier gas interacts with that stream and dictates its flow towards the coating surface. Rather than allowing the vapor stream to diverge in the standard EB-PVD manner, the DVD carrier gas flow generates gas atom / vapor atom collisions that keep the vapor stream focussed and directed towards the coating surface as a tight beam [3]. As it directs the vapor stream in this manner, the carrier gas flow plays an important role in achieving all four of the desired DVD processing capabilities. First, in the coating surface region contacted by the focussed vapor stream, deposition rates are expected to be extremely high (greater than 10 μm/min) [5]. Second, in contrast to standard EB-PVD systems, DVD's use of the carrier gas flow should allow the vapor stream cross-section to be focussed down to a dimension smaller than that of many of the surfaces being coated. As a result, DVD will be able to deposit the vast majority of its vapor stream onto the coating surface for efficient material utilization. Third, through a complex set of carrier gas / vapor atom / coating surface interactions that are just now being understood [6, 7], the presence

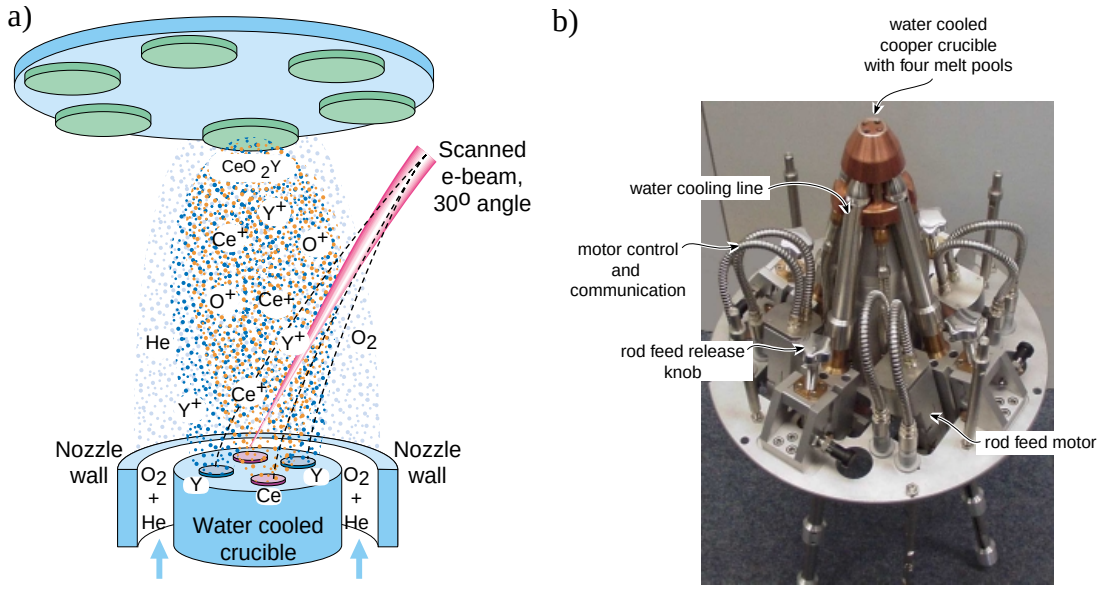

Figure 3 a) The sharply focussed e-beam (0.5 mm diameter) can be rapidly scanned across multiple vapor source pools. b) A multisource crucible.

of the carrier gas flow should influence the type of porous atomic structures that can be created using DVD. Fourth, the vapor stream focussing generated by the surrounding presence of the carrier gas flow should stimulate controlled composition definition of vapor emitted from closely-spaced crucible sources (Fig. 3). As a result efficient alloy creation will be possible from the entire vapor stream created in the system. If vapor is drawn from the source pools simultaneously, sophisticated alloys can be created. If vapor is drawn from the source pools sequentially, multilayer structures can be created. Although the experimental work has yet to be done, this second generation DVD system's high speed beam scanning capability, combined with gas phase mixing during transport to the coating surface is expected to facilitate precise alloy deposition. Focussed, efficient multilayer deposition should also be possible. Finally, in those cases where reactive deposition is desired, elements like oxygen or nitrogen can be introduced into the carrier gas flow and reacted with elements generated from the crucibles.

Although the carrier gas flow should achieve many of the stated material processing objectives for DVD, careful examination of the coatings created by the original DVD system revealed a gap in processing ability. DVD's electron beam evaporation combined with carrier gas transport did not appear to have a strong ability to generate fully-dense crystalline coatings. Instead, many of the coatings generated by the e-beam plus gas flow technology are highly defected, perhaps even porous structures [3, 7]. The explanation for this ability to produce some structures and not others seems to lie in the energy and angle of DVD vapor atom deposition [7]. The carrier gas flow appears to increase the angular distribution and lower the energy of atomic deposition [3]. Atomistic modeling has shown

that both of these characteristics can lead to the generation of highly defected, porous coatings [8].

To provide the second generation DVD system with an ability to create dense, crystalline coatings in addition to porous structures, the system delivered by FEP in the summer of 2000 introduces two additional technology components: plasma activation and electrical biasing of the coating surface. The new tool uses a hollow cathode plasma subsystem to generate a high density ionized gas and vapor flow (10^{12} ions/cm^3) just before the flow reaches the coating surface. This excitation of the atoms in the flow changes their activity level as they reach the coating surface and should generate a corresponding change in the atomic structure of the growing film. Even without application of an electrical bias on the coating surface, the self-bias generated by the presence of the plasma in the vicinity of a coating surface has been shown to modify the structure of depositing films [9]. Under certain circumstances, it is expected that atomic structure modification by the plasma can be further enhanced through the application of an electrical bias to the coating surface, and thus the new DVD incorporates both AC and DC substrate bias systems into its technology solution. The DC bias should be valuable when coating surfaces with conducting layer. The AC, pulse, bias should be valuable when coating surfaces with nonconducting layers. The second generation DVD system is shown in operation in Fig. 4, and the plasma and electrical bias subsystems are described in more detail elsewhere in this volume.

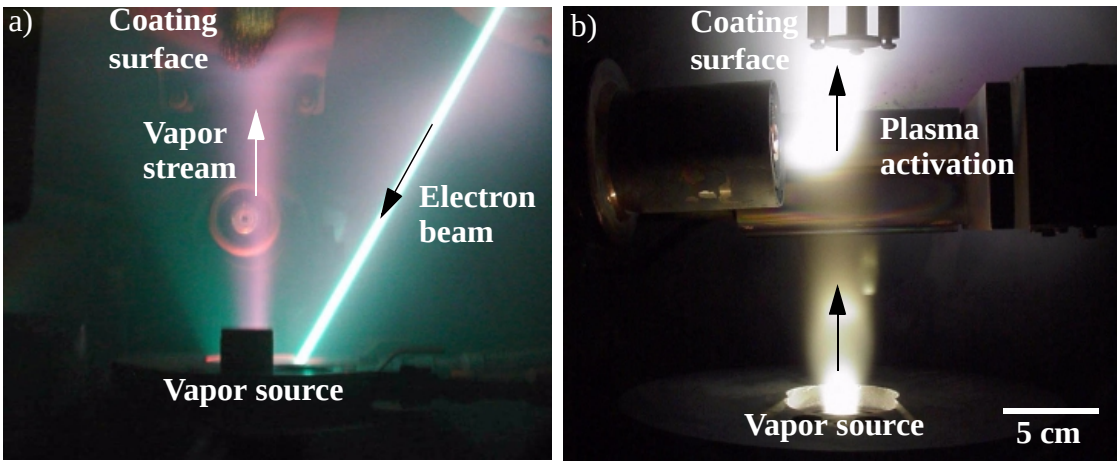

Figure 4 a) Electron beam heating, combined with a carrier gas flow generates a focussed vapor stream for high rate, efficient deposition. b) Plasma activation of the combined gas and vapor stream should enhance the ability of DVD to produce dense, crystalline structures.

Fields of Application

The ability to deposit coatings efficiently at high rate while controlling their atomic composition and structure is an attractive combination for many fields of application. While any one application requires only a few structures and compositions, production of

a wide range gives a technique like DVD broad impact potential. DVD coatings could enter such diverse fields as transportation, energy production, medicine, advanced materials discovery, and electronics. Research is underway to study DVD's production of yttria-stabilized zirconia with nanoscale porosity for thermal barrier coatings – used in aircraft engines (Fig. 5a)). Companies are inquiring about DVD's ability to produce thick and thin film components for solid-oxide fuel cell applications that can be used in clean energy power generation systems. Companies are investigating DVD's coatings on medical products. DVD's multi-crucible capability will soon be used as a materials discovery tool, searching for unique multielement metal and oxide systems. DVD could hold promise as a method of producing novel thermal conducting structures (Fig. 5b)). These are just a few of the many varied applications which could benefit if DVD demonstrates an ability to economically control atomic composition and structure in thick and thin films.

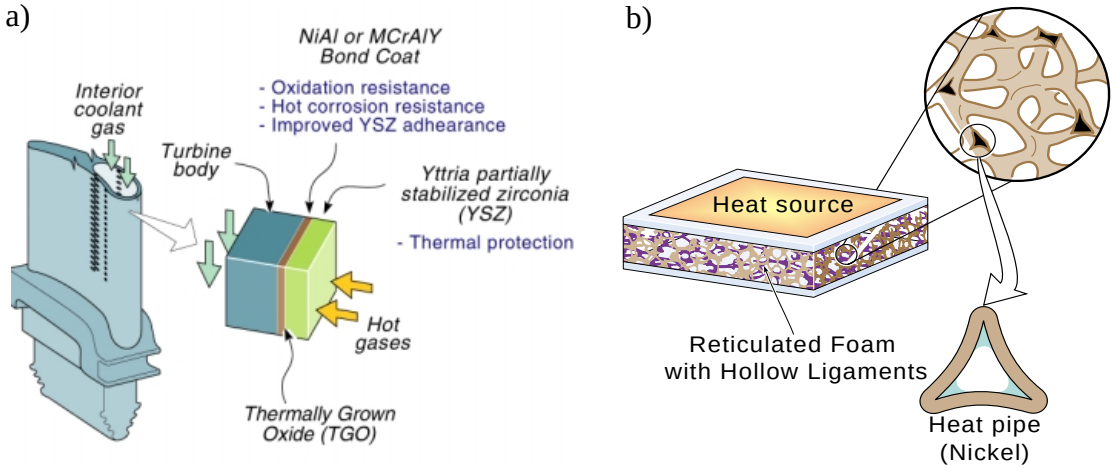

Figure 5 a) Thermal barrier coatings are multilayer coatings that protect superalloy components from the corrosive high temperature environment in turbine power systems [5, 6]. b) DVD studies suggest that the method has an ability to create thermal conducting structures from polymer foam precursors. DVD coats foams using its demonstrated non-line-of-sight coating capability [1, 3].

Initial Results

High rate deposition

The ability of the second generation DVD system to create high performance coatings is now being evaluated through experimental and modeling studies. A number of the initial results are reported here. As a first measure of DVD's ability to focus vapor onto coating surfaces for high rate deposition, a deposition profile experiment was performed. For this first experiment, a 1.27 cm diameter zirconium rod was placed in the crucible and evaporated using 50 mA (3.5 kW) of e-beam power. As soon as the zirconium vapor left the evaporation crucible, it was captured in a mixed gas flow containing helium and oxygen. The total gas flow through the system was 4.15 standard liters per minute (4.00 slm of

helium and 0.15 slm of oxygen). This gas flow rate generated a pressure of 0.26 mbar inside the DVD gas flow nozzle and 0.096 mbar in the main process chamber (See Fig. 2a).). The vapor was deposited onto a stationary flat glass coating surface located 17 cm above the zirconium source material. Following a short deposition run, the coating surface was removed from the chamber, and a surface profile was generated from the deposited coating.

Fig. 6a) shows the results of the profile measurement. The figure also overlays results of a direct simulation Monte Carlo (DSMC) modeling simulation [10]. The particular DSMC model employed, a code produced at Sandia National Labs (Albuquerque, NM) [11], utilized the same geometrical arrangement and gas flow / vapor flux conditions as the experiment. Fig. 6b) shows the model's predictions for carrier gas flow velocity in the system, and it shows the density of the zirconium atom flux leaving the crucible and nozzle. These types of data are difficult to obtain experimentally, and the ability to generate such process insight using DSMC illustrates the value of the modeling method.

To quantify the second generation DVD system's ability to enhance material deposition rates above those produced by other physical vapor deposition processes, the profile of Fig. 6a) can be fit to the standard equation used to describe PVD coating profiles [2, 3]:

$$\frac{d_s}{d_{so}} = \frac{1}{\left[1 + \left(\frac{r_s}{h_v}\right)^2\right]^{(n+3)/2}}$$

where d_s is the local film thickness on a flat coating surface, d_{so} is the film thickness directly above the vapor source, r_s is the distance from the coating surface midpoint to the point where d_s is measured, h_v is the vapor source to coating surface separation distance, and n is an exponent used to characterize the relative focus of deposited vapor. In sputtering systems, this equation generates an exponent $n = 1$. In EB-PVD systems, this equation generates an exponent $n = 3$, 4, or 5. For the DVD deposition profile shown in Fig. 6a), the exponent which fits the recorded deposition profile is $n = 53$. This significantly enhanced vapor focus corresponds to a greatly increased deposition rate in the local coating surface area contacted by the DVD vapor plume.

High material utilization efficiency

Having performed a first examination of DVD's high rate coating capability, a second set of experiments was undertaken to examine the new system's ability to enhance material utilization efficiencies over EB-PVD. To generate a comparison between EB-PVD and DVD, a first experiment was performed in which the DVD system was utilized as a standard EB-PVD coater. Because of the particular pumping systems installed on the DVD chamber, the system is capable of creating coatings like a conventional EB-PVD coater. Material evaporation can occur in high vacuum with no gas flow through the system. EB-PVD "baseline" results can thus be generated with the same e-beam gun and chamber configuration used in DVD gas flow experiments, allowing for convenient comparisons between the two deposition methods.

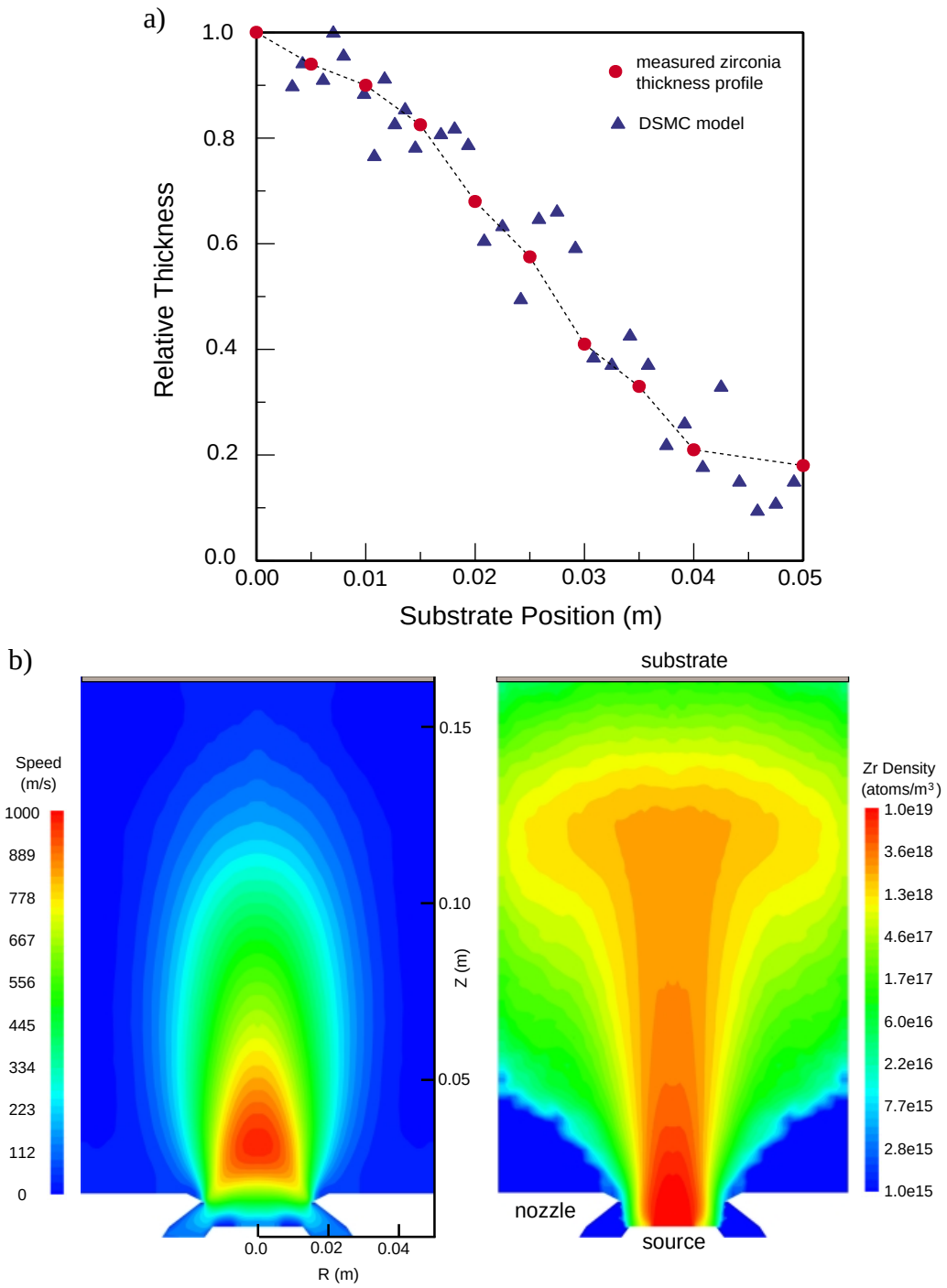

Figure 6 a) DSMC predictions of vapor distribution on the coating surface reproduce experimentally measured results. b) DSMC modeling provides further insight into the coating process, revealing the carrier gas flow velocity and the density distribution of vapor during the transport phase of the film creation process.

For this first EB-PVD experiment, a 10 cm by 10 cm flat glass substrate was placed 23 cm directly above a 1.27 cm diameter copper rod. A 1.0 cm diameter spiral electron beam scan pattern was then generated on the surface of the copper rod, and material was evaporated using 70 mA (4.9 kW) of beam power for 10 minutes. During the evaporation and deposition run, the chamber pressure was below $1x10^{-4}$ mbar, the lowest reading achievable on the vacuum chamber's capacitance manometer gauges. During the run, the copper rod was raised at a rate of 0.270 mm/min as evaporant was removed from the rod's surface. A total of 4.042 g of copper was evaporated and 0.362 g deposited. This generated a deposition efficiency of 8.96%.

To generate a comparative DVD experimental result, a second 10 cm by 10 cm flat glass substrate was placed 23 cm directly above a 1.27 cm diameter rod. Using the same e-beam scan pattern, current settings, and rod feed rates as those employed in the previous experiment, material was evaporated for 20 minutes. During the run, 8.5 slm of helium gas was introduced into the system as illustrated in Fig. 2a). This generated a chamber pressure of 0.62 mbar inside the DVD gas flow nozzle and 0.092 mbar in the main process chamber. A total of 4.481 g of copper was evaporated and 1.227 g deposited. This generated a deposition efficiency of 27.4%.

While these two material utilization efficiency experiments do not present a complete picture of the capabilities of DVD under its many process conditions, they do demonstrate that the method can at least triple the deposition efficiency generated by EB-PVD systems.

Atomic composition control

The multisource crucible capability in the new DVD system is just being brought online at the time of publication for this article. Thus no results are available at this time. Results demonstrating the vapor mixing and multilayer synthesis capabilities of the DVD method will be presented at a later date.

Atomic structure control

As noted earlier in this paper, the ability to selectively control the atomic structure of growing films was a key design criteria for this new DVD system. To provide such control, the new tool's design introduced plasma activation of the gas and vapor flow and electrical biasing of the coating surface. To evaluate the ability of plasma activation to influence atomic structure, a set of deposition experiments was undertaken.

In the first experiment, a 1.27 cm diameter zirconium rod was placed in the crucible and evaporated using 25 mA (1.75 kW) of e-beam power. As soon as the zirconium vapor left the evaporation crucible, it was captured in a mixed gas flow containing argon and oxygen. The total gas flow through the system was 4.15 slm (4.00 slm of argon and 0.15 slm of oxygen). This gas flow rate generated a pressure of 0.76 mbar inside the DVD gas flow nozzle and 0.11 mbar in the main process chamber. The vapor was deposited onto a stationary flat metal disk located 19 cm above the zirconium source material. The disk was composed of an Inconel 100 superalloy with a nickel aluminide (NiAl) "bond coat" on the coating surface. During this first run no plasma activation was attempted and evaporation

was performed for approximately 15 minutes. Following the deposition run, the coated disk was removed from the chamber, and an x-ray diffraction analysis was performed on the deposited coating. The result of this scan is shown at the top of Fig. 7 as an amorphous zirconia film. Although no active coating surface heating or temperature measurement was performed during this deposition run, it is estimated that the coating surface did not reach a temperature above 200^{o}C. This estimate is based on previous deposition experiment experience [3].

In a second experiment, a bond coated superalloy disk was again placed in the same position above a 1.27 cm diameter zirconium rod and evaporated using the same beam power. Material was again evaporated for approximately 15 minutes. During the run, 4.0 slm of argon gas and 0.15 slm of oxygen was introduced into the system through the DVD nozzle system. In this experiment plasma activation was also performed as shown in Fig. 4b). This plasma activation introduced 0.033 slm of argon from the tip of the hollow cathode plasma system and 1.00 slm of argon from the surface of the plasma anode (on the right side of Fig. 4b)). The result was a hollow cathode current of 40 A and pressures of 1.0 mbar inside the DVD gas flow nozzle and 0.14 mbar in the main process chamber. Following the deposition run, the coated disk was removed from the chamber, and an x-ray analysis was performed on the deposited coating. The result of this scan is shown in the middle of Fig. 7 as a textured, crystalline zirconia coating. Although no active coating surface heating or temperature measurement was performed during this deposition run, it is estimated that the coating surface did not reach a temperature above 400^{o}C.

The results of these two experiments demonstrate that plasma activation in the DVD system endows the technology with an ability to modify atomic structure during deposition. Although more study is clearly needed, the results of this study suggest that the new DVD system can form selected atomic structures (e.g. crystalline, textured zirconia) under conditions significantly different than those normally required for the creation of those structures. The bottom x-ray diffraction scan in Fig. 7 illustrates this point. In the original, non-plasma activated DVD system [3], production of similarly crystalline, textured zirconia requires coating surface temperatures in the range of 1000^{o}C [5].

Summary and Conclusions

The design of a new directed vapor deposition system has been presented and the philosophy behind the design has been explained. The technology designers have worked to provide industry with a new method of rapidly and efficiently creating high performance coatings on surfaces smaller than about 100 cm^2 in area. While reaching for these goals, the designers have also sought to endow the new DVD tool with an ability to tailor the atomic composition and structure of coatings. Selected results have been presented which strongly suggest that the tool's design is allowing its design specifications to be met. Additional work now needs to be performed to demonstrate the full range of DVD capabilities and to illustrate the technology's utility for the production of high performance coatings required by various market segments.

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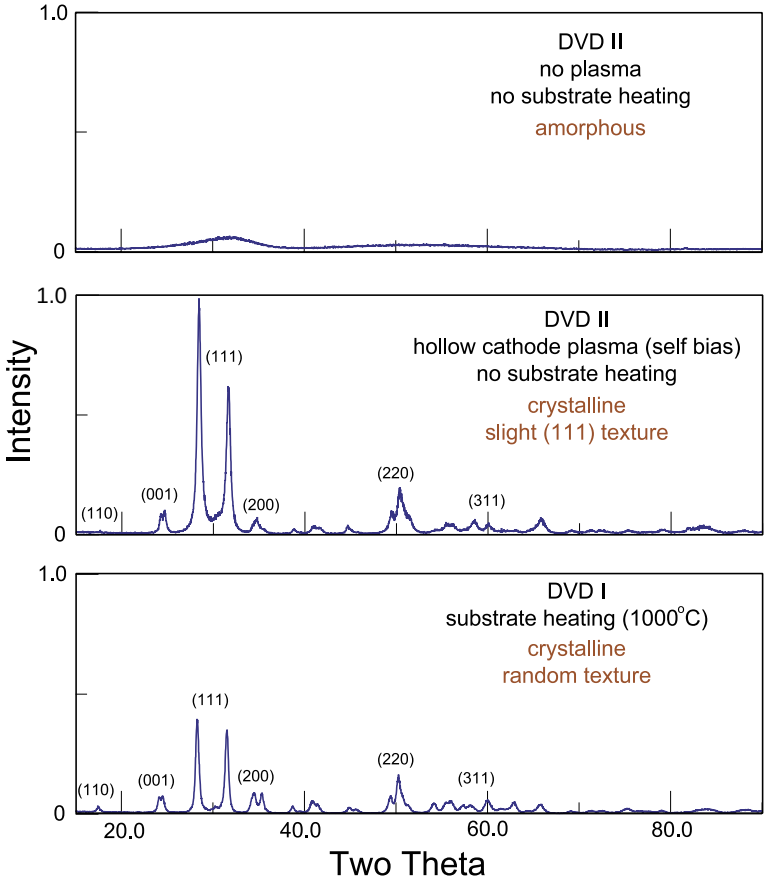

Figure 7 Different DVD process conditions generate distinctly different crystallographic structures in growing films.

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