Application Note

Gas Delivery for Low-E Glass Coating Applications

PROBLEM

Atmospheric pressure chemical vapor deposition (APCVD) processes are used to supply the low emissivity (Low-E) coating in float glass processes. APCVD Low-E coating processes employ both gas and liquid vapors as source materials and the flow of these gases/vapors must be accurately and precisely controlled. As well, flow control has to be extremely stable since these processes are expected to operate non-stop for 24 hours a day, 7 days a week with minimal maintenance. Achieving the required control and stability of gas flows in the often dusty and environmentally uncontrolled glass production environment requires extremely robust gas metering and control technology. This Application Note discusses the use of MKS Instruments' advanced mass flow control products for Low-E coating processes.

BACKGROUND

Low-E Glass Coatings

Thin film coating processes are widely used to improve the optical properties of all types of glass. Such coatings can enhance optical transmission characteristics and improve the aesthetic appearance of glass for architectural purposes. These applications require precision, accuracy and repeatability in the coating processes to produce uniform thin films in a cost effective manner.

Low-E coatings on glass minimize the amount of ultraviolet (UV) and infrared (IR) light that can pass through the glass without significantly reducing the transmission of visible light. The low IR transmittance gives Low-E glass much improved insulating properties as compared with normal, uncoated glass. Reduced UV transmission helps to avoid aesthetic problems such as sun fading in furniture fabrics and other material coverings in a room.

Two primary methodologies are employed for glass coating: on-line, pyrolytic chemical vapor deposition (CVD), which produces "hard" coatings; and off-line, batch process sputter coating, commonly referred to as physical vapor deposition (PVD), which produces "soft" coatings [1]. Hard coatings are chemically bonded to the glass, which make them a part of the glass. This characteristic makes hard coatings more robust than soft coatings which adhere to the glass surface by physical forces alone. Depending on the application, either hard or soft coatings may be preferred. Hard Low-E CVD coatings are often employed for passive, cold climate applications in which interior heat retention is important, while soft PVD coatings are used to produce Low-E solar-control films in mild to hot climate applications where interior cooling needs dominate [1].

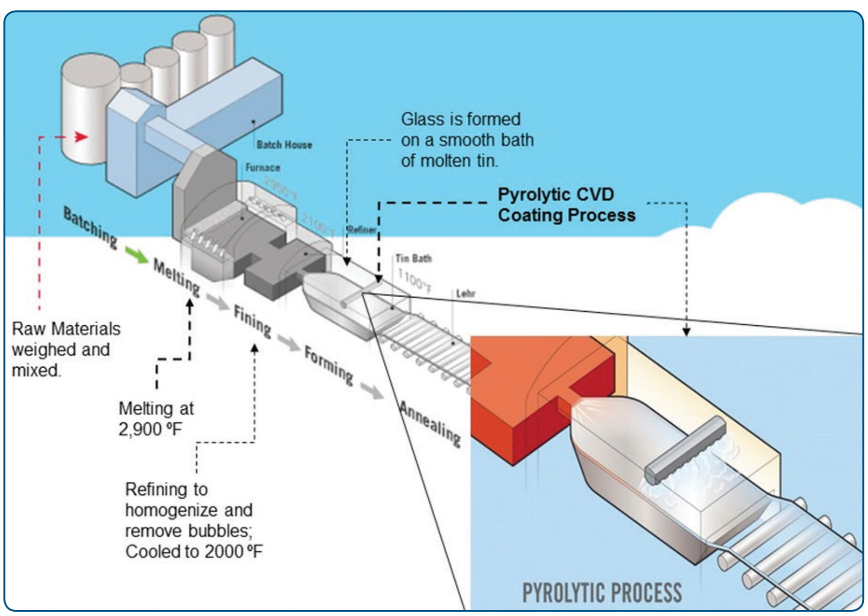

Figure 1 - Schematic of a float glass process showing Low-E film application using pyrolytic CVD [2]

Pyrolytic CVD coatings are deposited at atmospheric pressure, on-line in the float glass process (Figure 1)[2]. In the deposition zone of the pyrolytic process (Figure 2)[3], organometallic chemical precursor vapors (from liquid metal alkoxides, $M_x(OR)_y$, where M = a metal such as titanium or zinc and R = an organic group) are supplied to the high temperature deposition zone (typically 650°C). There, the metal alkoxides decompose via a mechanism such as that shown in Figure 3 to form a chemical bond between the metal oxide film and the float glass surface. This oxide bond is what makes pyrolytic coatings robust and scratch resistant. The pyrolytic process has a number of other production benefits. Since the coatings aren't easily damaged, no special handling considerations are needed in the manufacturing processes. This, coupled with the on-line nature of CVD processing, guarantees short lead times relatively high throughput and lower capital costs in the production line. Pyrolytic coated glass does not absorb moisture from the air, which gives it an essentially unlimited shelf life. Since the coating process is performed at atmospheric pressure, expensive and complex vacuum equipment such as that used in PVD processing (see below) is not required, minimizing capital costs. Finally, CVD coated glass products can be tempered similarly to standard float glass products, resulting in improvements to throughput and cost-effectiveness.

Soft sputter coating processes differ from pyrolytic processes in that they are performed off-line under vacuum conditions and at low temperatures. Figure 4 shows a typical sputter coating chamber configuration. Sputter coating has a longer history than CVD and this can make it preferred by some users owing to its well established process and application fundamentals. Sputter coating also has somewhat broader application than CVD due to the fact that it is not limited by the need for specific and volatile organometallic precursors. While the "soft" nature of these coatings makes handling an issue, effective methodologies have been established that avoid scratching in the production and inventory stages.

Sputter coated films are deposited under high vacuum through the physical adsorption of gas phase ionic species onto the glass surface. No chemical bond is formed in this adsorption process. Within the vacuum chamber, gas ions (typically Ar+) are accelerated by high voltage producing a plasma discharge. A target (cathode) is bombarded by these ions and atoms of the target material are ejected by the momentum transfer.

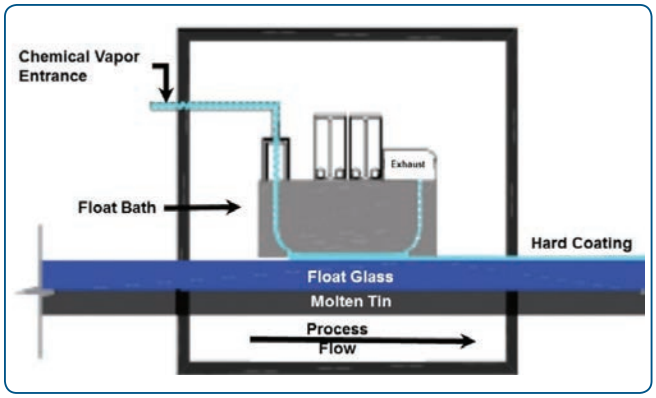

Figure 2 - Typical pyrolysis process CVD configuration

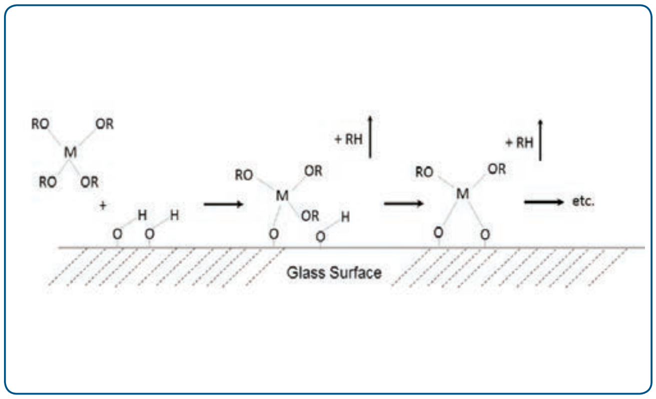

Figure 3 - Mechanism of chemical bond formation between pyrolytic CVD coatings and float glass

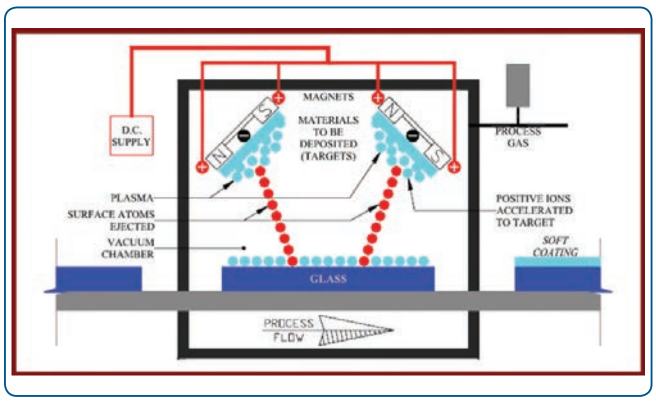

Figure 4 - Typical configuration employed for sputter coating

These gas phase ions of target material then migrate through the chamber and adsorb onto the glass surface to produce the coating layer. The chamber normally contains at least two targets, one of the desired metal (typically silver) and one of a metal oxide. The sputter glass coating is produced through the sequential adsorption of metal and oxide layers onto the glass surface with between 6 and 12 layers being normally applied. Additional targets may be employed to deposit barrier or color modification layers and sacrificial metal layers.

Depending on a user's production process and customer requirements, either hard pyrolytic or soft PVD coatings may be preferred. Figure 5 provides a comparison of the relative benefits and downsides of the two coating processes.

Gas Flow Control Requirements in Glass Coating Applications

Both CVD and PVD coating methods require precision gas flow control for precise and repeatable process operation. PVD processes typically require precision flow control for high pressure purge and inert sputter gas delivery to the vacuum chamber; this control is best achieved through the use of electronic thermal mass flow controllers (MFCs). CVD process gas flow requirements are somewhat more complex in that these processes can employ both gas and liquid vapor sources. Liquid sources are the only option for the formation of films such as aluminum dioxide, titanium dioxide or zinc dioxide. Thus, in addition to flow control for conventional purge and oxidation gases, CVD processes require specialty flow control for the delivery of the vapor from volatile liquid chemicals. The precise control of low pressure liquid vapor delivery requires the use of either a combination of pressure controllers with conventional thermal MFCs or special low vapor pressure flow control devices.

Glass coating process applications also place an additional burden on effective gas flow control. The environment in which the MFCs and other flow control devices are placed in these applications is harsh, with high levels of moisture and particulates present in the ambient atmosphere. Such environments can severely degrade the performance of conventional MFCs.

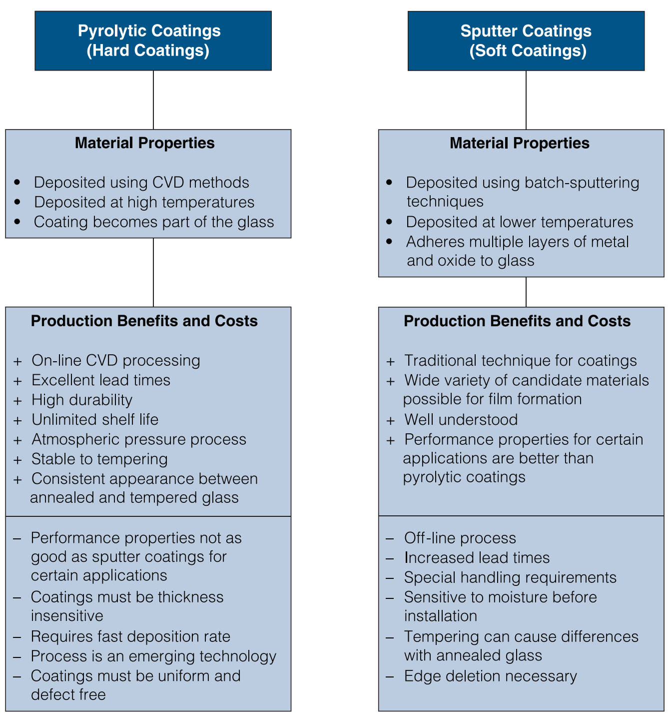

Figure 5 - Comparison of the relative benefits and costs of pyrolytic vs. sputter coated glass products and processes

SOLUTION

Process Gas Delivery

MKS Instruments' thermal MFCs (Figure 6) control the rate of molar gas flow to a glass coating process; molar flow control is critical in processes where the ratio of reactant gases can influence coating properties. MKS Instruments' I-Series MFCs are waterproof and dustproof. They are IP66 rated, designed for use in harsh environments where ingress of dust and liquids can be a problem. The MFCs are manufactured in a clean room environment and all wetted materials are 316L stainless steel, ensuring contaminant-free gas delivery.

Figure 6 - MKS I-Series General Purpose, Multi-Gas MFCs (left: IE50A, right: IE250A)

The design of the instrument hardware and firmware guarantees insensitivity to temperature and pressure variations in the gas supply, making these instruments simple to deploy with no ancillary measurements and compensations needed. Available as elastomer or metal-sealed MFCs, they incorporate advanced digital flow control electronics and a patented thermal sensor and mechanical design that ensures 1% of set point accuracy and repeatability of 0.3% of the gas flow reading. The accuracy and precision of the flow measurement, coupled with the highly repeatable control ensures that glass coating processes using I-Series MFCs are tightly controlled, and that these processes can be replicated with relative ease.

I-Series MFCs are available in a variety of communication interfaces including Profibus, Analog and 4-20 mA. In addition, MFCs can be operated via ModBus TCP/ IP via the Ethernet port located on all MFCs. They are designed as multi-gas/multi-range instruments, allowing users to change the gas in <45 seconds using an on-board Ethernet interface and an integrated, JAVA-enabled web browser that requires no special software. This latter characteristic is especially valuable for reducing inventory burdens.

CONCLUSION

Pyrolytic and sputter coating operations for the production of Low-E glass require precise and accurate flow control for a number of gases. MKS Instruments' I-Series MFCs have been shown to be ideally suited for use in this demanding application. The I-Series MFC's robust character provides critical flow control capabilities in the harsh and physically demanding environment for these applications.

REFERENCES

[1] "What is Low-E Glass", PPG Glass Education Center, http://educationcenter.ppg.com/glasstopics/how_lowe_works.aspx

[2] "Learn About Glass", PPG Glass Education Center, http://www.educationcenter.ppg.com/glasstopics/learn_about_glass.aspx

[3] Stewart Engineers, "Coating Technology – Processes", Technical Document CVD-00M-02, published in April 2004, http://stewartengineers.com/English/wp-content/uploads/2013/11/SE-Coating-Technology-Brochure.pdf

For more information on MKS Instruments I-Series Flow Control Technology, link to our web site: http://www.mksinst.com/product/Category.aspx?CategoryID=415

For more information on MKS Instruments Vapor Source Mass-Flo® Controller Technology, link to our web site: http://www.mksinst.com/product/category.aspx?CategoryID=247

For a general catalog of MKS Flow Control Technology, follow this link: http://www.mksinst.com/product/Catalog.aspx?catalogID=3

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