Typical Substrate Problems Encountered in Large Area Flat Glass Coating Operations

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ABSTRACT

Substrate problems encountered by large area d.c. sputtering systems will be discussed and examples of various defects shown. Some examples of substrate problems most commonly seen are: age of substrate, glass stain, separator and stain reduction material residues, cutting fluids and other organic residues. Other substrate defects may be process related and will also be examined.

INTRODUCTION

Substrate defects are one of the largest problems faced in the production of sputtered coatings. The effects of various defects are oftentimes cosmetic and thus usually obvious to the unaided eye. The larger the surface area coated, the more likely that there will be a defect in the aperture. In most instances the defective substrate is returned to the manufacturer for credit or destroyed since it is unacceptable.

Identification of defective substrates after coating is not desirable since the coating has high added value, and scrapping it at this point drives up the cost of production. In the present discussion, we will look at some on the substrate surface defects from a detection and prevention point of view.

Surface quality of the the tin side of the glass is variable, therefor, the coating is usually applied to the atmosphere side.

SURFACE DEFECTS

Glass Stain

Glass stain is a common problem in the architectural and residential sputter coating business where the typical substrate is float glass. Stain is a form of chemical attack of the float glass surface where sodium is leached out of the surface, leaving a microscopically pitted finish on which the coating is expected to adhere. Typically, this leached area is non-uniform over the lite. The coated lite is left with patchy hazed diffuse surface and less than ideal adhesion to the substrate. For a more in-depth discussion of the mechanics of glass stain, see reference [1].

Stain is best viewed by looking at light reflected by the surface after the glass has been washed. A dark background enhances the contrast by reducing the amount of light reaching the eye by transmission Unfortunately, sometimes even the best of viewing conditions are not sufficient to disclose the presence of stain and it is only seen after the surface is coated.

Freshly produced float glass rarely shows stain, but fresh is relative to many conditions. If the glass has been produced more than 14 days prior to use, stain is a possibility. If the glass was made in the high humidity months of summer, stain can appear in as little as 2-4 days. If the glass is stored in a cool area within the warehouse and subsequently moved to a warm area, condensation of moisture on the surface can occur, and this can lead to almost immediate staining.

Stain inhibitor quantity and uniformity is directly linked to glass staining. The inhibitor itself does not cause staining, but misapplication of the material may enhance staining conditions.

Stain inhibitors are added to the separator powder to slow the process of staining in stacked glass sheets. The inhibitor is typically an acidic powder. Because it is usually applied as a dry powder, non-uniformity of coverage can occur. This can lead to various degrees of stain on the glass surface.

Lightly stained glass can sometimes be used for certain coating types. Light stain can sometimes be removed with a polishing agent such as cerium oxide applied to the surface of the glass and washed in a standard flat glass washer. Medium to heavily stained input glass usually cannot be salvaged for subsequent coating.

Top Tin

In the production of float glass, liquid glass is floated on a pool of molten tin. The process takes place in a controlled atmosphere to minimize oxidation of the tin. In this furnace zone, some of the tin exists in vapor form. This vapor can condense on cooler spots in the tin “bath”. If enough of the tin collects, a small amount may remelt, and deposit on the top of the molten glass. The tin droplets form a small bead, and are referred to as a top tin defect. Sometimes these defects can be extremely large, but most are circular and their diameter is on the order of 1 mm and less. Such small beads are sometimes missed by the float line inspection crews or

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equipment, and are found by the customer prior to and sometimes after the coating process.

Top tin defects can be seen in either reflection or transmission. The defects look like droplets of solder. The defects may sometimes interact chemically with the glass surface to form an insoluble residue.

Top tin droplets sometimes drop off spontaneously in the packing and shipping processes or are removed by the coating machine entrance washer. Some, however, adhere so strongly they cannot be removed. The tin oxide stain is not removable, but is sometimes small enough and transparent enough so as not to be noticeable.

Cutting Fluids

Cutting fluid is necessary to produce a good "cut" edge on the glass. Cutting fluid acts as a high pressure lubricant between the glass and "cutting" wheel, extending the life of the cutting wheel. In addition, by keeping moisture out of the scribed line, it improves the cutting process by preventing the "healing" of the glass fracture which is typically enhanced by atmospheric moisture and time. The cutting fluid should NEVER be oil. A light aromatic organic fluid such as mineral spirits is preferred. Only a minimal amount of cutting fluid should be applied directly under the cutting wheel. Float glass producers typically use only a small amount, but accidents do happen, and sometimes the glass lites are heavily coated with cutting fluid. Second tier glass suppliers may provide glass that they have cut with true oils such as motor or gear oil. These fluids should be avoided at all cost since they are very difficult to remove, requiring detergents and other cleaning techniques that are not suitable to cost-effective volume production. An option called "dry cutting" can be done where no cutting fluid is used between the glass and cutting wheel. This results in premature failure of the cutting wheel, and eventually poor glass edge conditions, but may be available from the float glass supplier at extra cost.

Excess cutting fluid is easily seen in transmission or reflection. It is visible as droplets, streaks or sometimes puddles of mineral spirits. Small amounts of cutting fluid are handled well by a standard flat glass washer in good condition at recommended washing temperatures and detergents. Glass with heavy amounts cutting fluid should be avoided as excess fluid can contaminate the washer, or pass through the washer into the coater. Thus, one should have a close relationship with the substrate source to ensure that the supplied substrates are in a proper state of cleanliness.

Glass Crush

Glass crush is the result of small chips of glass falling between packed lites of float glass, being crushed under load, and abrading the glass surface when the packed glass is shipped. These chips are typically formed when the cut edge quality is poor due to insufficient cutting fluid, dull cutting wheels, or improperly annealed glass.

Glass crush abrasion is difficult to identify. Best when viewed in reflection. Tthe abrasion exhibits a linear or erratic track or path as the glass chip drops between the lites under the force of gravity as the packed lites are moved. The reflection in the abraded area is point or diamond like, and stands out from the surrounding area.

Lites with glass crush abrasion are typically rejected, and not useable in subsequent coating operations.

Scratches

Glass scratches can be caused by numerous sources. Once again, these are most easily viewed in reflectance. Although they are rarely caused by theactual float glass manufacturing process, they can occur from the float glass is packaged for shipment.

Most glass scratches are caused by improper packing techniques. Edges of lites being manually packed on a rack or case may be brought into contact with and dragged across the face of a previously packed lite. At other times the lites are manually realligned, or indexed relative to each other, resulting in sliding motion between glass faces. A rubbing type of scratch can occur with enough pressure, and/or lack of enough interleaving material.

Scratches can be caused unintentionally by metal eyelets in the safety clothing worn by packing personnel, by contact with metal belt buckles, and sometimes by safety gloves which have tiny shards of glass embedded within the glove.

Repeated handling increases the risk of scratching.

Scratched glass is usually unsuitable for use as input for the coating process.

Separator Crush

In the packing of float glass, a separator powder or interleaving material is used between each lite for several reasons. The powder is typically a mixture of tiny plastic beads and an anhydrous organic acid. The beads provide an airspace so that individual lites can be pulled apart easily. The beads also act as miniature ball bearings, keeping the lites of glass from rubbing against each other.

When the separator is crushed with improper packing, the lites can be difficult to pull apart, the beads may leave a permanent organic residue on the glass surface, or the lites of glass may physically contact each other, abrading or scratching the surfaces. Crushed separator is best viewed under a microscope in reflection.

Unless the glass is very old (definition of old depends on numerous parameters such as temperature, humidity, storage conditions, etc.), most crushed separator is readily removed in the standard washing process. Organic residues can pose a problem for coatings which are sensitive to surface cleanliness. If the residue is severe, the glass should be rejected.

Organic Residues
There are numerous other organic materials, not just cutting fluids, which come into contact with the upper and lower float glass surfaces. Organic materials may leave a permanent residue on the glass surface. Sensitive coatings may show these organic residues in reflectance or transmission.

Examples of organic residue sources include: glove prints, cutting oil soaked glove prints, suction cup marks from automated and manual glass transporters, oils from chip blowers and air compressor equipment, packaging materials like Styrofoam, cardboard separator, polyethylene wrap, rubber from float glass drive conveyor systems, greases and lubricants from overhead bearings and fittings, etc.

Once on the glass, these organic compounds are difficult to wash away with standard detergents used in typical coater washers. Putting grease and oil residues into any washer is a bad idea as it contaminates the washer, resulting in costly down time for cleaning of the washer. Minor residues, such as glove prints and float line drive roll prints, are not a problem for the washer but do not truly come off the surface of the glass either. A fog test of a washed lite will reveal the organically stained areas. Unless the lite is cleaned with an abrasive slurry such as cerium oxide, the organic stain is likely to remain. Again, for some coating types, this is not a problem. Adhesion to the glass surface may be compromised only slightly.

If the organic residue is easily noticed in transmission or reflectance on the input glass, and if the coating is a sensitive layer system, the glass should be rejected.

Metallic Residues
Metal residue is a somewhat uncommon condition. Sources include: eyelets on packing personnel protective wear, belt buckles, dragging of the tin bath curtain on the top of the glass in the float process when the glass is manufactured. Off-line cutting of the glass may have other unknown sources of metal to glass contact resulting in metal embedded scratches and rubs. Metal residues are best viewed in transmission.

Some metal residues are easily removed with steel wool. Most leave a permanent rub mark, and the glass must be rejected.

Other Defects
Still other defects can include simple warehouse dust which can slowly work its way between the lites. Dust can be quite abrasive, especially when it includes silica particulates. In the warehouse, dust can form from fork lift tires, wind-borne sand, concrete dust, etc. Most of these abrasives leave the glass surfaces marred on a microscopic level and occur during the shipping process. Most are not viewable to the naked eye.

Again, fresh float glass eliminates the time necessary for the glass to accumulate the particles. Assurance of good warehouse cleanliness and techniques from the float manufacturer to the coating machine is the best defense.

EDGE DEFECTS

Poor Cutting Quality and Edge Stress
Glass with well cut edges is necessary to assure that small chips of glass will not break off the lite in the packing, shipping or coating processes. Here, the type and condition of the cutting wheels and residual stress in the glass determine whether the edge quality will be acceptable or unacceptable.

Improper cutting wheel angle, worn cutting wheels, or improperly annealed glass will lead to high edge stresses and the formation of shell chips, hackle, discontinuous break lines and generally poor edge work. The continued stress along the edge results in flakes and chips which drop between the packed lites, scratching and abrading the glass, or may even pop off in the coater and land on the incompletely coated surface. When these relatively loosely bound particulates are removed in a subsequent washing step, coating voids will appear.

It is best to work with the glass manufacturer or glass cutter to ensure proper edge appearance. Poorly cut edges may be useable for some coatings, but not others.

INTERNAL DEFECTS

Seeds, Stones, and Other Inclusions
Within the body of the glass, a wide variety of inclusions are possible. Almost all are best viewed in transmission. Some can be quite large, but most are less than 1 mm in diameter. Float glass manufacturers typically screen out most of the defects greater than about 0.5 mm. Still, an occasional seed (air bubble), or stone(ceramic inclusion) slips by and is found by the customer. Glass with obvious seeds or stones is usually scrapped.

Other defects not easily visible with the eye include internal optical distortion, usually due to non-homogeneous mixtures of the glass ingredients across the float ribbon. These rarely cause a problem for coating use unless the distortion is severe.

SUMMARY

With proper attention to incoming substrate quality, identification of glass surface, edge and internal defects can reduce the cost of production of coated product.

REFERENCES

1. P. Duffer, “How glass reacts with water...”, Glass Digest, Sept. 1994.