# Cleaning of Parts for Precision-Optic and Glass Substrates Before Coating

J. Strobel and R.M. Hohl, UCM AG, Rheineck, Switzerland

**Key Words:** Precision-optics; Optical industry; Ultrasonic cleaning; Cleaning technology

## ABSTRACT

In order to guarantee a perfect coating, a spotless and particle-free cleaning is a must for the industrial manufacturing of glass substrates. The up-to-date cleaning process is based on watery and biodegradable cleaning media. These media are used depending upon the different types of glass used in the precision optic. The ultrasonic power/frequency as well as the chemicals, sequence of active and rinsing tanks, water quality and drying is decisive for spotless and particle-free cleaning. The most successful method of drying is either infrared or vacuum drying. The cleaning of substrates can be divided into two different steps. The first step (ready for inspection cleaning) will clean the surface of the substrates to control the individual fabrication. This separate cleaning line will clean the substrates from the filling compounds, resin, cover-up varnish and residual polish. The second step is the proper cleaning prior to the coating. With this cleaning the last contaminates are removed from the surface. The selected equipment, chemicals and the care of the bath plays a major role for successful cleaning before coating. To ensure a reproducible cleaning result, a robot handling system is a must. In addition it guarantees a high flexibility of working with different cleaning cycles. All these features will result in a perfect cleaning line for a smooth cleaning process.

## INTRODUCTION

State-of-the-art cleanliness of substrate surfaces prior to a coating process is mandatory for its success. Any contamination of the surface affects the adhesion of the coating and leads to defects. The degree of cleanliness of the surface cannot be expressed in numerical terms and can only be demonstrated by the use of suitable aids. The objective sought is “freedom from residues” and “freedom from particles,” as much as possible. Uniform, reproducible quality is indispensable, even when the products supplied from the preceding stages of manufacture vary within relatively wide tolerances in terms of shape, size, nature and degree of contamination. The solution to this type of problem requires well-tried process technologies in user-friendly equipment which operates safely and economically. One well-tried cleaning procedure prior to coating is based on cleaning with aqueous solutions plus ultrasound, followed by drying. In the course of today’s increasing awareness of environmental matters, processes which make use of solvents prior to coating have subsequently disappeared completely from factories. Cleaning in aqueous solutions is carried out in accordance with precise cleaning mechanisms. Cleaning is always a multi-stage process, in which cleaning and rinsing stages are alternately repeated. Modern multi-chamber cleaning plants are found in the optical and electronics industries, and fine mechanics as well as in the high-vacuum coating area (coated lenses and hard coatings).

## CLEANING MECHANISMS

Depending on the application and branch of industry, the substrate surfaces to be coated are soiled with a very wide variety of organic and inorganic contaminants.

The common contaminants are:

- centering oil
- grinding and polishing pastes
- Cerium-Aluminum oxide
- fingerprints, dust, oxidation

The function of a cleaning process is to wet the contaminants on the substrate surface, to loosen them, envelop them, detach them from the surface and rinse them away. Cleaning is a complex multi-stage process. The active elements in it, coming into play alternately, are chemical action, mechanical action, temperature and time.

### Chemical Action

Cleaning baths require chemical additives as cleaning agents. In addition to carbonates, silicates, phosphates, borates and complex agents, these detergent substances contain surfactants as essential components of the cleaner. Surfactants are organic molecules with two chemical groups spatially separated, one of them hydrophobic the other hydrophilic. The hydrophobic parts of the surfactants attach themselves to non-aqueous boundary surfaces and also include substances which are insoluble in water within a micelle which they form. The hydrophilic part of the surfactant molecule is always orientated to the water. This property of the surfactants makes it possible, on the one hand, to achieve complete wetting of the surfaces of solid materials, on the other to infiltrate the dirt particles and lift them off the substrate to be cleaned more



46th Annual Technical Conference Proceedings (2003) ISSN 0737-5921

easily. Depending on the nature of the contamination, alkaline, neutral and acid cleaning agents are employed.

### Mechanical Action

The process of loosening the contaminants on the surfaces of the objects to be cleaned is greatly speeded up by ultrasound. The supplementary cleaning effect of the ultrasound is primarily caused by cavitation. Cavitation consists of the formation of empty spaces in a liquid caused by a local reduction of pressure. These empty spaces (cavity bubbles) have a limited life. Incorporated piezoelectric transducers create the sub-pressure that are excited by high-frequency oscillations in the cleaning media. The collapse (bursting) of the cavitation bubbles causes a kind of "brushing effect" on the surfaces to be cleaned. In addition to the parameters of the bath liquid (viscosity, density, temperature etc.), the factors which influence creation of the desirable state of so-called "hard" cavitation are the frequency and intensity of the sound. If there are dissolved gases in the cleaning medium, for example shortly after filling a tank with water, initially "soft" cavitation occurs. In this "soft" cavitation the cavitation bubbles have a gas pressure approximately equal to the hydrostatic pressure in the surrounding liquid and their abrasive effect is low. Newly filled baths must, therefore, be degassed. This can be done by pulse operation of the generators; the actual ultrasonic frequency not only being modulated, but in fact switched on and off periodically. From a certain stage of degasification onward, the transition to "hard" cavitation then occurs. The cavitation bubbles that are now formed are largely free of the gases dissolved in the liquid. In addition to ultrasound, a further mechanical effect, which contributes to intensifying the cleaning or rinsing action, is the motion of the bath liquid, generated by a vertical motion of the objects being cleaned. The speed or cycling-rate can be set as required. In the rinsing baths additional turbulence also can be created which improves the rinsing effect at the surfaces of the objects being cleaned. Turbulent rinsing is generated by using suitable inlet nozzles to inject the rinsing water at the bottom of the tanks. By pulsing the water flow, the turbulence effect can be further intensified.

### Temperature

In the cleaning baths a temperature of 40-60°C (100-140°F) is advantageous, both for the chemical action of the cleaning agents and for cavitation effect of the ultrasound. A higher vapor pressure in the cleaning liquid leads to a slower decline of the sub-pressure in the cavitation bubbles. A raised temperature in the rinsing baths of 30-40°C (85-100°F) is advisable for good rinsing action.

### Time

The length of time for which the objects being cleaned remain in the cleaning or rinsing baths depends on the nature and degree of contamination. The average cleaning time in a single bath is about three minutes. The total time spent in the cleaning line is greatly affected by the type of drying method employed, as well as by the speed of the transport mechanism.

## CLEANING SCHEME AND PROCESS TECHNOLOGY

### Cleaning

The chemical processes in the cleaning baths are matched to the specific contaminants on the substrates. The required concentration and action of the chemicals are being determined by laboratory experiments. To support the cleaning effect, the cleaning baths usually operate at a raised temperature. Temperatures of 40-60°C (100-140°F) are usual, and reasonable in terms of economical operation (energy consumption, evaporation losses). To further support the cleaning effect, ultrasound is used. The strength of the sound field emitted should be matched to the surface area of the substrates to be cleaned. The ultrasonic power fed into the baths should be approx. 5-10 watt/liter, depending on the volume of the cleaning tank. The most commonly used operating frequencies of the ultrasonic generators and transducers are in the range of 20 to 80 kHz. The use of ultrasonic generators with output control ensures that the sound-wave power can be matched to the objects being cleaned. With glass and highly-polished workpieces, cavitation damage can occur at the surfaces if the power of the sound-wave is too great. The cleaning and rinsing action in the baths is further intensified by applying a vertical motion to the objects being cleaned. This improves the exchange of cleaning solution or rinsing water at the workpiece surfaces and also leads to a more even application of the sound waves. In order not to interfere with the ultrasonic waves the speed of the workpieces has to be selected accordingly.

### Rinsing with Tap Water

Rinsing is an essential stage in the cleaning process. Rinsing removes from the substrate any still clinging cleaning agents and dissolved contamination. Special attention must be paid to the way rinsing water is fed into the tanks, in order to achieve uniform distribution of the incoming flow. By fitting suitable nozzles in the rinsing-water feed system, further positive effects can be achieved through a turbulent air-water mixture, thus increasing the rinsing effect on the surfaces to be cleaned. It is important that the rinsing water is in constant motion, so that no loose particles can adhere to the objects being cleaned. The use of tanks with all-round overflow channels ensures that any loose residues cannot adhere to the edges of the tanks, are constantly washed away and cannot be redeposited on the substrates. The rinsing baths between the individual cleaning stages use untreated water (tap water) for rinsing. The hardness of the water should not exceed 12-15°dH (215 – 270° USA). Generally warm water with a temperature of 30-40°C (85-100°F) is used. The rinsing baths can be operated as a cascade system; i.e., the rinsing water is used in several tanks in succession, running from the last rinsing bath to the first, in the opposite direction to the advance of the objects being cleaned. The cascade arrangement makes a significant contribution to reducing the water consumption. In order to ensure the highest possible rinsing-water quality despite this, additional filters can be incorporated. Depending on the nature of the chemicals used before the cleaning bath, the rinsing water can also be operated as a closed circuit (cleaned filter systems). Plants exist which operate with two or three separate rinsing-water circuits. This type of rinsing technology makes it possible to reduce freshwater consumption to a minimum.

**Rinsing with Deionized (DI) Water**

For the last two rinsing baths in a modern cleaning plant (finest rinsing) DI water is used. The highly purified water is necessary for the cleanliness of the substrate. The DI water-rinsing system operates in a cascade. In the first DI rinsing bath the objects being cleaned are rinsed with ultrasound support. The ultrasound speeds up the water exchange at the substrate surface and in any fine gaps and drill-holes present. In the second DI rinsing bath, turbulent rinsing is used. The purified water is circulated through a special DI water station. A working life of the purified water of approx. 3-6 months is by no means uncommon.

**DI Water Circuit**

The purified water-return-flow is fed by means of a centrifugal pump into the ultra-violet sterilization unit via a return filter, and then passes into a storage tank. The water level in the storage tank is monitored, and, if necessary, fresh DI water is automatically added. From the storage tank the rinsing water passes via a pressurizing unit through an active carbon/absorption-resin cartridge, ion and cation (mixer-bed resin) cartridges, and a fine-filter combination (1 micron/0.2 micron). Before it flows back into the DI rinsing tank, a conductivity meter monitors the conductivity of the DI water. The quality of the DI water in the last rinsing baths is mandatory for perfect, particle-free drying of the objects being cleaned.

**Drying**

Three different drying methods are possible to dry the parts:

a) hot-air drying using filtered air in the circulation
b) vacuum-drying using infrared heating
c) static infrared drying in a filtered laminar-flow air stream (laminar flow box)

**Hot-Air Drying**

The duration of drying has a decisive influence on the work-cycle and the design of the cleaning plant. Due to the fact that hot-air drying requires a longer drying time, the dryer is designed as a continuous-flow tunnel which can take several baskets. Hot-air drying, therefore, requires a larger plant volume. When drying glass and plastic materials hot-air drying can generate static charges. The hot-air used for drying is circulated through a particle filter. The air circuit is provided with input flap-valves to admit fresh air and exhaust flap-valves to change the saturated air.

**Vacuum Drying**

Vacuum drying, on the other hand, avoids the disadvantages of hot-air drying. Only one basket at a time is placed in the drying chamber, which allows an individual drying cycle according to the parts. The drying cycle is controlled by pressure and temperature via predetermined programs. The temperature of the substrates after drying is 40-80°C (100-175°F). The air for ventilating the vacuum chamber is fed through fine filters, so that the surfaces are not contaminated by particles. Depending on the application, the vacuum chambers can also be ventilated with nitrogen.

**Static Infrared Drying**

In static infrared drying using a laminar flow box, the basket with the substrates is taken out from the last DI rinsing bath by a slow-pull mechanism (approx. 1 mm/s), and then will be dried under a laminar flow box (Class 10-100) with infrared radiators. This procedure is only suitable for flat substrates and is used principally in the optical and electronics industries.

**Quality Control**

Since there is no objective method for measuring the result of a cleaning process, the degree of cleanliness achieved by it can only be assessed subjectively and visually. The following methods are used to assess the substrate surfaces after cleaning:

- **Visual inspection**
  Using different sources of illumination and optical aids such as magnifying glasses, stereo microscope, etc., allows an operator to give a visual inspection to the object being cleaned.
- **Test inks**
  A drop of test ink is placed on the cleaned surface. The way in which the drop spreads, or the steepness of the angle formed at its edge, allows conclusions to be drawn about the cleanliness of the surface.

**Special Requirements in the Optical Industry**

There are three branches in the optical industry: flat glass, ophthamalic, and precision-optic. The manufacturing steps, contamination and sensitivity issues of each of these branches is given below:

1) <u>Flat Glass</u>

| | |
|---|---|
| *target:* | After the final rinsing a complete wetting |
| *sensitivity:* | Little – medium |
| *cleaner:* | Alkaline cleaners with pH 8 – 13 (to be used in 2-3 active tanks) |
| *drying:* | Slow-pull tank and static infrared drying or hot-air drying |

2) Ophthalmic

*sensitivity:* Medium

*drying:* sSlow-pull tank and static infrared drying under a laminar flow-box or hot-air drying

*parts:* Mineral glasses and plastic lenses

*manufacturing steps:*

A) After the polishing and before the intermediate control
B) Before the coating
C) Before the hard-coating (just for plastic)

To A) *target:* Surface to be free of polishing paste to meet the requirements for a partly automated surface control

Removal of polish paste is more effective with partly degassed tanks than fully degassed tanks. This knowledge is against the opinion of the experts, that is that ultrasonic tanks are only working properly when the cleaning media is fully degassed. Through a special device in the active tanks the gas content can be kept nearly constant. Through the gas bubbles the effect of the cavitation will be softened and the number of cavitation bubbles increases. This leads to a better and more efficient cleaning of the fine polishing residuals.

To B) *target:* Surface to be completely free of residuals and particles

To C) *target:* Maximum of adhesion for the varnish for plastic lenses

Standard cleaners are used with a pH of (11-) 13.5. Very high demand on the cleaning line similar to B. Mostly the cleaning process is combined with the varnish process (Dip-coating) as well as the spin coating.

3) Precision-Optic

*target:* Surface to be completely free of residuals and particles. Modern multi-stage coating systems (Multilayer) are showing no mercy for every cleaning failure.

*sensitivity:* Cleaning is characterized through the strong restriction of the possible alkaline and acid cleaning medias

*cleaner:* Depending on the resistance class of the glasses, a pH value of 6 – 10 is possible

*drying:* Slow-pull tank and static infrared drying under a laminar flow-box or vacuum drying

An important role in this cleaning process is played by the treatment time in the tanks, the ultrasonic intensity and the temperature. All of these parameters can be modified and adjusted currently so that nearly all kinds of glasses can be cleaned with watery chemicals. Typically in the precision optic are the short treatment times in the DI water rinsing tanks and the prioritizing through the control system of the substrates. The prioritizing will prevent a longer staying times in the tanks than set in the programs.

**General Conditions for all branches**

The following are required for all branches:

- All chemicals are used in a concentration of approximately 0.5% up to 5%, depending on the contamination and chemicals.
- Bath temperatures approximately 40°C up to 60°C (104 – 140°F) depending on the sensitivity of the glass substrates.
- Treatment time approximately 2-3 minutes (for precision optic approx 30 – 120 seconds).

## MINIMAL CONDITIONS

At a minimum the following conditions must be meet.

- **General Conditions for DI Water:**
  - Conductivity lower than 0.15 ms/cm (6.6 MΩ/cm)
  - Filtration 0.1μ (max. 0.2μ)
  - Stable number of particles during process
  - No cloudy DI water (no scattering under laser beam)
  - Rinsing rate for a tank up to 100 lt (26 gal) of 10 times the bath volume per hour => 1’000 lt/h (264 gal/h)

- **Typical Cleaning-Line Design: before coating**

| | |
|---|---|
| 1 US Cleaning (ph 11) | 2 US Cleaning (ph 8) |
| 3 Rinsing (tap water) | 4 US Cleaning (ph 7) |
| 5 Rinsing | 6 US Rinsing (DI water) |
| 7 Rinsing (DI water) | 8 IR drying |

Note: all active tanks filtrated (0.5 μ).

Example: Glass type BK7 and B270 complete cycle
Glass type SK15 and SK 16 starts in tank 2

## CLEANING-LINE DESIGN

Modern multi-chamber cleaning plants consist of individual tank modules which are assembled to form a sequential system in accordance with the cleaning scheme decided on. A base structure for an automatic goods-transport system is located to the rear of the plant. The substrates to be cleaned are placed in special good-baskets. Transport of the baskets can be carried out with an individual horizontal-vertical mechanism (transfer unit).

**Transport System**

An individually driven transport arm runs on a horizontal guide rail over the whole length of the cleaning plant, including the loading and unloading positions. As it does so the arm moves a basket from tank to tank on each occasion. Both horizontal and vertical drive is effected by controlled gear motors. Horizontal and vertical positioning is controlled via incremental transmitters. The automatic transport mechanism is arranged so that no drive components are located directly over the tanks, thus ensuring that any particles which may rub off do not get into the cleaning and rinsing baths. Various safety devices and interconnections (bend detector, contact detector, etc.) ensure safe operation. Controlled acceleration and deceleration phases ensure that the objects being cleaned are transported gently, with no jolting, and positioned exactly in the individual work modules. An important advantage of this type of transfer device is its great flexibility of operation. With this system it is possible by suitable programming to omit any cleaning or rinsing bath from the sequence, as desired. In plants with a large number of tanks and a high throughput performance, several transport arms are installed.

**Control System**

Modern multi-stage cleaning plants are monitored and controlled by means of a program control system (PLC). This can store a large number of different cleaning programs, which are matched to the specific requirements of the respective application. Among other factors, the programs control the duration of the ultrasound or turbulent-rinsing active periods, the ultrasound intensity, bath temperatures, flow quantities, treatment sequences (e.g., skipping individual tanks) etc. By placing simple codes on the goods baskets, the different programs can be called up and executed.

**Construction Details**

Plants for industrial use must deliver consistent cleaning results, be easy to use and service, and operate safely. Servicing and maintaining cleaning plants also means cleaning the plant itself and inspecting the baths. Only with plants that are properly serviced and maintained is it also possible to produce a clean product. Therefore in choosing plant design and construction, attention should be paid to the following details, among others.

<u>Tank design and construction:</u> Individual tanks in a modular layout, made from stainless steel. Sloping bottoms ensure complete emptying. Rounded internal corners make thorough cleaning easier. For deep tanks, cleaning flanges make access possible. Rounded, serrated overflow-edges improve the overflow and reduce contamination on the walls. Uniform tank construction makes any later conversion work easier.

<u>Pipe work:</u> All pipe work should be executed in poly-propylene, with welded, not glued, joints. Fittings (rotary valves, slide valves) in poly-propylene and stainless steel.

<u>Ultrasonic:</u> Ultrasonic transducers built into hermetically sealed housings, arranged over the bottom of the tank, are preferable to transducers bonded to the bottom or walls of the tank from the outside (tank durability/replace ability). Piezo-ceramic transducers, with their high-efficiency and long working life, are currently state-of-the-art. The new system of tubular transducers ensures all-around emission and, thus, better sound distribution in the tank. Simple replacement of tubular transducers is provided by flange design.

**Economic Considerations**

Apart from capital investment costs and the associated financial provision, the running costs of a cleaning process are essentially the cost of water and energy. In comparison with this, costs for cleaning chemicals and labor are generally negligible. Nevertheless it must be said here that cheap cleaning agents are not always also economical or produce the desired results, especially in respect of quality assurance. Labor costs relate essentially to handling the objects to be cleaned. The operation of the cleaning line is due to the high degree of automation neglectable as well as the maintenance. Maintaining a cleaning plant consists principally of periodic cleaning of the tanks, especially when cleaning or rinsing baths are renewed. The time required for cleaning is however, small, provided that the plant has been properly designed and built in respect of servicing and maintenance.

Measures to reduce water consumption consist of the followng:

a) Extending the working life of the cleaning baths by re-circulating the contents using built-in filter systems. Here a separate circulation system is provided for each cleaning bath.
b) By operating the rinsing baths by cascading, using built-in filter systems, the water consumption can be reduced by 50-70%.
c) By incorporating adjustable flow meters as well, the freshwater feed can be varied to suit individual requirements. In addition, the active period of the rinsing process (rinsing-water feed) can be controlled via the PLC system. Rinsing then switches on only when there is a basket in the tank.

d) In normal continuous-flow operation a 60-70 l (16-18 gal.) rinsing tank consumes rinsing water at the rate of c. 10-12 l/min (2.5-3.0 gal./min). With single-shift operation and 50% switch-on time, consumption is 3000-3600 liter/rinsing tank (750-900 gal.). If three rinsing tanks are operated in the continuous-flow arrangement, this consumption is tripled. With a cascade connection the rinsing-water consumption can be reduced to one third. If the rinsing baths are operated with special filter systems, the rinsing-water consumption can be reduced to evaporation and carry-over losses. It must, however, be taken into consideration that, depending on the degree of contamination and throughput performance, possible renewal of the circulating water should be checked at intervals of one to two months.

## CONCLUSION

In an operational production system “cleaning” as a work-process has hitherto not been regarded as creating added value. But, meanwhile, in all these places experience has shown that only general and expert cleaning prior to coating offers an assurance of reliable production and a guarantee of consistent product quality. Regarded in this light, “cleaning” certainly constitutes the creation of added value, because it contributes to avoiding faults, lowering reject rates and reducing costs.