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Jet Vapor Deposition of Single and Multicomponent Thin Films

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The pivotal, distinctive feature of jet vapor deposition (JVD) is the collimated sonic jet in a low vacuum that serves as the deposition source. It is a feature that invites comparison of JVD with physical vapor deposition (PVD) and chemical vapor deposition (CVD) methods. In general, PVD techniques, such as evaporation and sputtering, involve low pressures (<1 mtorr), remote generation of film species and collision-free, line-of-sight deposition; CVD, conversely, is carried out at high pressures (>1 torr), with complex film precursors convected relatively slowly toward decomposition on a hot substrate. Some of the features of PVD and CVD are also found in JVD, but the physical behavior of the sonic jet source creates an entirely new set of possibilities.

The JVD strategy of using jets in low-vacuum, fast flows for film deposition was originated by Halpern[1] and Schmitt.[2] Jet vapor deposition jet sources operate over a range of several torrs to tens of torrs, accommodate diverse vapor generation schemes and transport film species efficiently to substrates at near-sonic speeds. In common with PVD, the JVD film components are generated remotely by diverse physical processes and are delivered at high speed. In common with CVD, JVD can also operate at higher pressures as well as use similar chemical strategies; however, JVD is distinguished by the influence of jet gas dynamics, which confers operational simplifications and fruitful synthetic possibilities. Jet collimation and high speed lead to fast, efficient and economic deposition. Single or multiple jets in combination with mobile substrates enable production of simple, multilayer and complex multicomponent film structures over large or small areas; the constituents may be selected almost at will from metals, semiconductors, oxides, nitrides, dielectrics and polymers. Jet vapor deposition is thus a general, high-rate gas dynamic approach to film deposition that provides distinctive options not accessible with PVD and CVD. Here we review the principles and implementation of JVD and describe several ongoing research projects and production runs at Jet Process Corp.

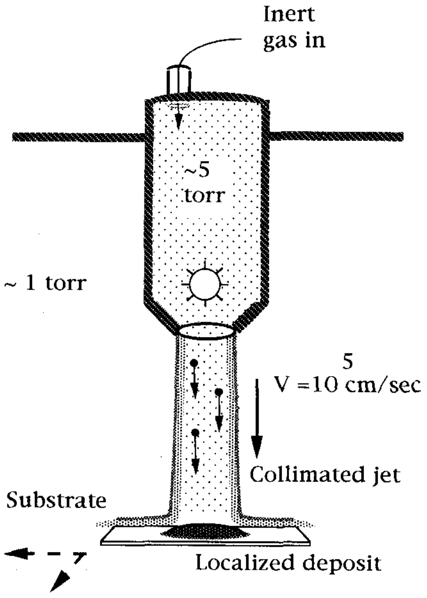

Fig. 1. Generalized jet vapor source. The vapor generation source, positioned near the nozzle exit, can be based on thermal evaporation, glow discharge sputtering and microwave or DC discharges. Jet diameters can be several millimeters to several centimeters.

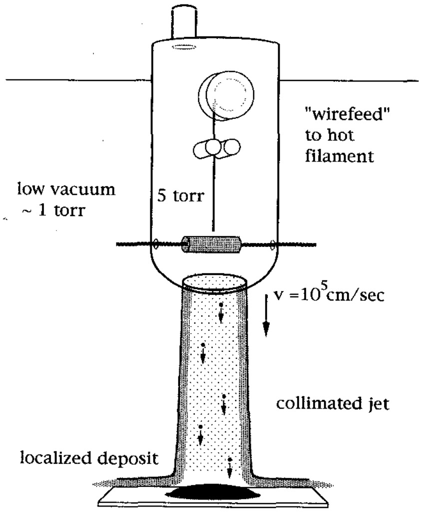

Fig. 2. "Wire-feed" jet vapor source. Metals such as gold, copper, silver and aluminum can be vaporized by feeding wire against a bare or sheathed hot tungsten filament. The rate of feed determines the deposition rate; throughputs can be as high as 0.1 cm³ of solid metal, giving growth rates of microns per minute over 1000 cm².

PRINCIPLES OF JET VAPOR DEPOSITION

Deposition from a generalized jet in low vacuum is illustrated in Fig. 1. In brief, a jet of inert carrier gas propels condensible atoms, molecules or clusters to a substrate downstream.[1–4] The jet is formed by a nozzle of large exit diameter, several millimeters to 2 cm, supplied with helium or hydrogen carrier gas and incorporated in a fast-flow, low-pressure system. Steady-state flow and pressure are sustained by mechanical pumps with speeds ranging from 400 to 20,000 L/min; depending on nozzle size, this results in a nozzle pressure (P_n) of 1–10 torr and a downstream pressure (P_d) of 0.1–1 torr. For $P_n/P_d > 2$, critical flow obtains; a well-collimated jet emerges from the nozzle at the speed of sound, 105 cm/s for helium.

A vapor source is placed just upstream of the nozzle exit. This source can be based on thermal vaporization, glow discharge sputtering, microwave discharge chemistry, laser vaporization, etc. Figure 2 shows a computer-controlled wire-feed jet source of metals, such as gold, copper, silver, and aluminum.[3] For example, gold atoms are generated by feeding gold wire against a tungsten filament held at 2000°C. Each evaporated gold atom is swept out of the nozzle by the sonic carrier flow and intercepted by the substrate downstream; the linear wire feed rate (in centimeters per minute) determines the deposition rate. On a stationary substrate, one observes rapid formation of a bright disk of gold comparable in area to that of the jet. The boundary of the deposit is sharp; however, the deposit itself is nonuniform, being thickest at the center.

To obtain uniform deposits over larger areas, either the substrate or the jet must be moved. One choice is to mount the substrate on a carousel that can both spin and oscillate along its own axis, as shown in Fig. 3. The carousel motion is computer controlled, so that a variety of motions can be programmed. If the carousel is stationary, atoms deposit in a circular zone, as described above. If the carousel spins, the deposit forms a band around it; if the carousel oscillates along its axis as well, its entire surface (and that of every substrate on it) is efficiently coated to uniform depth.

The versatility of JVD can be explained in several ways. First, in low vacuum the jet is collimated, and deposition is localized; film growth is fast and efficient. Second, several different jets can be arrayed around the fast-moving carousel and substrates; this multiple-jet, mobile-substrate strategy creates a powerful route to reactive synthesis of a host of complex films[3,4] at the submonolayer level but at a high rate; the products can be alloys, multicomponents, multilayers and host-guest films whose constituents are metals, semiconductors, oxides, nitrides, dielectrics, organics and polymers.

The synthetic capability of JVD can be traced to the pressure range of jet operation (1–10 torr in the nozzle; 0.1–1 torr downstream). At these pressures, the jet is collimated and nearly cylindrical; indeed, the collimation has been verified by chemiluminescent techniques that make the jet contours visible. Fast radial diffusion, which tends to flare the jet, is outweighed by faster, near-sonic transport down the jet axis, so that atom deposition is localized and efficient. At much lower pressures, radial diffusion would result in a divergent jet; entrained vapor would bypass the substrate and reduce deposition efficiency. High-vacuum PVD processes such as thermal evaporation do not have the JVD economy of material usage; they result in line-of-sight deposition, but vapor is wasted in the wrong directions. Much higher pressure, however, risks gas-phase cluster formation and particle growth at high-vapor inputs; film quality can then be compromised. In JVD, clustering will not occur at prevailing pressures and transit times; only single atoms or molecules reach and condense on the substrate, even at high deposition rates. The JVD jet sources accordingly operate in an optimal regime—the pressure is high enough to collimate the jet and realize high-deposition rates but low enough to ensure arrival of single atoms and molecules at the substrate.[1] Sonic flow means that the vaporization mechanism within a nozzle is unaffected by downstream reactive species; for example, refractory metal sputtering targets inside a nozzle are protected even from atomic oxygen downstream. Jet collimation is particularly advantageous for synthesis; because the jets do not interfere in the gas phase, a moving substrate can reactively sum the deposition fluxes from several jets to give complex multicomponents.

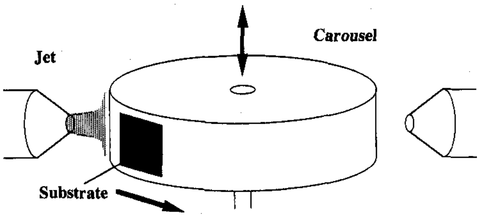

Fig. 3. Movable substrate carousel. The substrates move in two dimensions with respect to the jet to give uniform coverage over large areas (1000 cm²). Several jets can be aimed at the carousel for synthesis of multilayers or alloys.

The JVD process thus combines high-speed gas dynamics with inexpensive, fast-flow, low-vacuum technology to give synthetic versatility, convenient operation and economic throughputs. The advantages of JVD strengths can be summarized as follows[1–4]:

1. *High-rate, efficient deposition.* The jet in a low vacuum is efficient because virtually all atoms injected into the flow travel to the substrate in a straight line at near-sonic speed. For example, more than 95% of the gold wire vaporized in the wire-feed source in Fig. 2 is captured by the substrate, with virtually no loss inside the nozzle.

2. *Versatile, inexpensive and convenient apparatus.* Only high-speed mechanical pumps drive the flow; the deposition chambers need not be large or complex. Compact vaporization mechanisms can be positioned in the nozzle throat. These utilize thermal evaporation, sputtering, laser vaporization and microwave and DC discharges, and several different mechanisms can operate at once in different nozzles. Low-vacuum operation and small-footprint chambers translate into fast turnaround times and easy access to substrates and jets.

3. *Uniform, large-area coverage.* This can be achieved by means of the scanning motion of the rotating/translating carousel. Even though the deposition profile from a single jet is narrow and nonuniform, the carousel motion exposes every point on the substrate to the same flux for equal times; the resulting film is uniform over the 1000-cm² carousel area.

4. *Synthetic versatility.* Coupling of multiple jets and mobile substrates produces the most powerful consequence of jet collimation—deposition from several independent jets can be summed. As each substrate rotates with the carousel, it integrates material contributions from individual jets. This strategy allows fabrication of complex alloys, multicomponents, multilayer structures and host/guest assemblies, all at high rate over large areas, with accurate uniformity. It qualifies JVD as both a versatile synthetic tool and a reliable, economic manufacturing technology.

5. *Hot or cold substrates.* Even if a jet is hot, carousel rotation ensures a short (<0.1 second) residence time for a substrate passing through it, and substrate heating is negligible. Optional lightweight heaters mounted on the carousel permit substrate temperatures higher than 700°C, even for a moving substrate.

6. *Negligible transport limitations.* These are absent in both gas phase and growing film. The speed of the jet overwhelms diffusive constraints in the gas. At the substrate, carousel rotation can be increased to ensure submonolayer deposition per substrate pass; micromixing from different jets results in diffusion-free growth, even at overall high growth rate.

7. *Gas dynamic impact energy deposition.* Energy delivery to the growing film can have a singular effect on film quality.[5] Heavy passenger species seeded in a light carrier gas can attain kinetic energies of several electron volts during a free jet expansion.[6] If the expansion takes place into high vac-

uum, that kinetic energy remains available on impact with a surface. In the low vacuum of JVD, collisions with carrier gas atoms tend to dissipate the passenger kinetic energy; nonetheless, proper choice of jet substrate spacing and upstream/downstream pressures potentially allow a large fraction of that kinetic energy to be liberated on impact.[7] Energy can be delivered either by the depositing species or by an inert copassenger, such as xenon or argon seeded in a hot hydrogen jet.

8. *Masking is possible for jet deposition of patterns.* The jet flux penetrates cuts in a mask despite low vacuum, to give fine lines and sharp detail.

9. *Clean deposition.* Despite low-vacuum operation, the immunity to contamination of JVD is equivalent to very high vacuum. Contaminant levels are determined mainly by input carrier purity; this is easily in the parts per billion range, so that chamber pressures of 1 torr correspond to contaminant pressures of 10^{-9} torr. Under fast-flow, high-speed pump conditions, downstream leaks cannot give rise to large steady-state chamber concentrations; impurity molecules are pumped away too fast, and backstreaming from the pump is eliminated. Moreover, the higher deposition rates made possible by JVD suffice to overwhelm background impurity deposition of whatever origin; consequently, film purity is high even in low vacuum of JVD.

10. *Environmentally friendly processing.* None of the JVD sources require toxic chemicals, and no toxic effluents are generated. The JVD process has already economically replaced electrochemical plating in one commercial application because of environmental concerns.

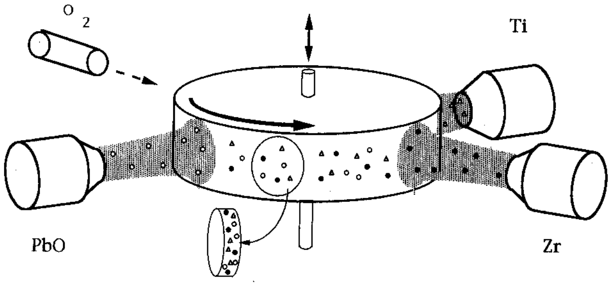

Fig. 4. Multiple-jet, mobile substrate JVD. In this example, four jets operate together to produce the multicomponent oxide, lead zirconate titanate (PZT).

EXAMPLES OF JET VAPOR DEPOSITION FILMS AND APPLICATIONS

COPPER-GOLD MULTILAYER ELECTRODES; ALUMINUM-ALUMINUM OXIDE MICROLAMINATES

Copper and gold can be thermally vaporized and deposited at high rates by means of JVD wire-feed sources such as shown in Fig. 2. At present, we can uniformly coat an area of approximately 1000 cm^2 to a depth of 1 µm in one minute by means of copper and gold jet sources that dissipate only 60 W. Structures containing alternating layers of copper and gold are made with two jet sources, operated alternately for appropriate times.

Control of copper-gold layer thickness via the wire-feed approach is precise. We verified this by depositing 100 alternating layers of copper and gold, each intended to be 50 Å thick. Total thickness and uniformity were confirmed by stylus profilometry; the accumulated thickness was measured at 15 points over an area of 6 cm^2 to be 4996 Å ± 10 Å. Interface sharpness was verified and layer thickness confirmed by low-angle X-ray scattering.[8]

This high-rate, large-area metal multilayering capability is currently being put to a successful test in production runs on a service basis for a defense contractor. Their substrates are 50-mm-diameter wafers less than 1 mm thick; these require a 1.5 µm copper electrode, flashed with 50 nm of gold, on both sides of the wafer, with a 0.25-mm border, free of metal and cleanly defined, at the wafer perimeter. Wafers are mounted on the carousel in accurately machined receptacles that serve to support the wafer, orient it toward the jets and define the perimeter border.

Wafers are processed at the rate of 500 per week by means of a single JVD deposition chamber operated by one person. In this application, JVD has displaced an electrochemical technique on grounds of both quality and environmental concerns. Given that gold comprises a considerable part of the operating expense, the localization and efficiency of JVD are a decisive advantages.

We used a variation of this technique to deposit 100-µm thick microlaminates consisting of 50-nm layers of aluminum alternating with 5 nm of aluminum oxide. This was done in 10 minutes by means of a steady, high-rate jet of aluminum (0.1 cm^3/min of metal) into which oxygen was pulsed (slightly downstream of the nozzle) at appropriate intervals. Microlaminates having nanoscale component layers are expected to show enhanced strength and mechanical properties; JVD nanocomposites exhibited a hardness of 2.5 GPa (D. McIntyre, personal communication), equal to that of microlaminates made by sputtering,[8] a far slower ultrahigh vacuum process.

LEAD ZIRCONATE TITANATE: FERROELECTRIC FRAM NONVOLATILE MEMORIES

Lead zirconate titanate (PZT) is a candidate for thin-film memory because it can store charge at high density and retain it in zero applied field; films of several thousand angstroms can switch states within the 5-V range of computer power supplies. The JVD process is one of several (sol-gel, vacuum sputtering) now competing for this future market.

The JVD process uses four jet sources to supply lead, titanium, zirconium and oxygen to platinum-coated, heated silicon wafers mounted on the spinning carousel, as shown in Fig. 4. These jet sources build up a 1-µm film of PZT in less than one hour.[9]

This procedure yielded high-quality PZT films at high rate. The ferroelectric perovskite phase appeared on deposition; after annealing, the film was entirely 110-oriented perovskite. Key parameters for effective PZT memory cells and the values measured for JVD PZT films are the following: remanent polarization, 6–20 µC/cm^2; coercive field, 60 kV/cm^2; switching endurance more than 10^{11} cycles; dielectric constant greater than 1300. These values are equal or superior to those obtained with more conventional methods. We are currently developing JVD PZT for ferroelectric memory elements under a

Defense Advanced Research Projects Agency (DARPA)-sponsored Phase II SBIR program.

In addition, PZT thin films are potentially useful for pyroelectric infrared detectors; we are pursuing this application in a Phase I SBIR program sponsored by the U.S. Army EDTL.

ELECTRONIC GRADE SILICON NITRIDE

Silicon nitride films of remarkable electronic quality were generated by using a microwave-discharge-equipped jet source that produces silicon atoms, silicon-bearing molecular fragments and nitrogen atoms (T. P. Wang et al., unpublished observations). A hydrogen atom jet, run for several minutes, was used to remove thermal oxide from a silicon wafer. Slow deposition on that substrate gave a nitride whose electrical behavior in a metal-nitride-semiconductor capacitor was superior to that of any previously reported (T. P. Wang et al., unpublished observations). In many respects, such as breakdown strength, radiation hardness and interface trap density, these nitride films were equal or superior to the best thermal silicon dioxide grown at high temperatures (>1000°C). The etch rate in buffered oxide etch was a low 10 Å/min; the index of refraction was 2.03 at 632.8 nm, close to that for stoichiometric silicon nitride; however, the outstanding fact is that this silicon nitride was produced on a room temperature substrate; this should enable coating of thermally sensitive substrates, such as mercury-cadmium-telluride, in a number of electronic applications.

The JVD process silicon nitride shows great promise as a gate as well as for passivation. The reason for this high quality is not fully understood; we suspect that high-speed transit from discharge to substrate conveys small numbers of excited species that liberate energy at the growing film and annihilate imperfections in it, provided that the deposition rate is low.

FIBER COATING FOR COMPOSITE MATERIALS

Coating of ultrafine fibers and multifilament tows is important for fiber-reinforced composite materials; JVD exploits jet collimation in several unique approaches to the problem. Jet conditions can be controlled to obtain uniform coating despite jet directionality; jet momentum is sufficient to disrupt and agitate fiber bundles and overcome shadowing of one fiber by another. In this way, we have achieved uniform coating of 12.5 μm alumina fibers, singly and in bundles of several hundred, with copper and aluminum metal several thousand angstroms thick, as verified by scanning electron microscopy. We are currently developing, under DARPA sponsorship, reel-to-reel techniques for JVD fiber coating.

COATING OF THERMALLY SENSITIVE MEMBRANES

The low substrate temperature capability of JVD is being exploited in the coating of gold and platinum as fine-line electrodes on 9-μm PVDF piezoelectric membrane. PVDF is wrapped around the carousel and covered with a 0.001-in. foil mask that defines the electrode pattern (0.25-mm wide line, 10 cm long). The jet deposits a 2000-Å film through the pattern as the carousel spins. A similar line is deposited on the other side of the PVDF, perpendicular to the first electrode; the 1 mm × 1 mm intersection zone defines a capacitor whose output can be used to detect impinging sound waves (C. Everbach, personal communication). PVDF is thermally sensitive and can be depoled at a temperature of 350°C; the low-temperature capability of JVD is therefore critical. The JVD process also deposits the noble metals only on the mask/substrate area, where they are needed.

CERAMIC HOST-ORGANIC GUEST FILMS

We have trapped complex organic guest molecules, such as rhodamine B and methyl red, in a range of ceramic host films; such as silicone dioxide, silicone nitride, aluminum oxide and magnesium oxide. Codeposition takes place at room temperature, with no degradation of the organic guest; for example, trapped rhodamine still fluoresces under ultraviolet light. The guest concentrations are high; a 1-μm film of methyl red in silicon dioxide appears deep crimson, implying doping levels approaching 1%.

Such host-guest films can have optical electronic and thin-film sensor applications. With R. Zanoni and colleagues at Oklahoma State University, we are currently testing the deposition, patterning and photobleaching of ceramic host-guest methyl red films to make thin-film waveguides. We have also observed that guest methyl red in microporous silicon dioxide changes color from red to yellow when exposed to vapors of hydrochloric acid or ammonia; methyl red is a well-known acid-base indicator. Ceramic host-organic guest films can be made by mechanical or sol-gel methods,[10] but these are multistep processes, limited to soluble species, often including time-consuming thermal treatment. In JVD, the host-guest combination is generated in minutes at room temperature by a vapor deposition technique compatible with existing semiconductor microelectronic processing.

POLYMER DEPOSITION: PARYLENE

Parylene [poly(*para*-xylylene)] has properties, such as high electric breakdown strength, impermeability to water and biocompatibility, that make it attractive for protective functions. Parylene can be vapor deposited by a unique mechanism in which di-*para* xylylene is cracked at high temperature and convected slowly to a cold surface, where polymerization then takes place; this is the conventional Gorham process.[11] The main weakness of parylene is that it adheres poorly to surfaces when deposited in this way; however, we have observed that adherence and hardness were greatly improved by deposition from a sonic JVD source. In addition, a 2- μm JVD parylene film on a platinum wire survived 93 days in a soak test at 5 V of applied potential (D. Edell, personal communication). These results suggest that JVD parylene has an enhanced capacity to withstand rigorous electrical and mechanical conditions.

SUMMARY

The use of single or multiple jets in a low vacuum coupled with mobile substrates makes JVD a flexible technology for a wide range of film applications. The JVD process links high-speed gas dynamics with low-cost, low-vacuum equipment to give synthetic versatility at economic throughputs. Jets operating in the JVD pressure regime are collimated, intense sources of localized deposition. Much of the versatility of JVD can be explained by the coordination of several spatially separated, independent jets, resulting in multicomponent, multilayer alloy structures, synthesized from component metals, semiconductors, dielectrics and organics. The possible material combinations are numerous and unconstrained by the identity of the components; the synthesis of known and potentially useful film materials is being systematically explored. MF

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Quality and Filtering

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It is an undeniable fact that over the past 20 years, the development of highly efficient acid baths and the increasingly stringent quality requirements in all areas have both resulted in surface treatment technicians improving and increasing the filtering of baths in which parts are treated. Filtering is in point of fact a major quality-control factor. You have only to ask yourself how much a batch of parts costs that has been rejected for faults due to insufficient bath filtering to be convinced that filtering is indispensable and can be indirectly considered a source of savings.

The process of filtering removes all solid forms of contamination from all origins (atmospheric dusts; anode slime; precipitates; particles or various slurries from the parts to be treated, especially in tubes and castings; incipient metallizing in chemical baths, etc.). Filtering helps circulation of the electrolyte and is often the only circulation source. Experience has shown us that although know-how varies according to particular industries, companies and countries, the majority of electroplating baths are filtered.

FILTERING EQUIPMENT

Industrial filters include filters used for surface treatments and more particularly for electrolytic and chemical baths. There are various filter types, and each is suitable for and designed to solve a specific problem. The nine main types of filtering equipment encountered include the following:

- Horizontal chamber filters
- Wound or extruded cartridge filters
- Sack and bag filters
- Panel filters
- Pleated cartridge filters
- Fabric cartridge filters
- Band filters
- Press filters
- Lens filters

Alternative versions based on certain of these basic models are available: automatic and semiautomatic filters, reversible-flow filters and coating tank filters.

FILTERING AGENTS

Filter manufacturers make technical choices for their devices with regard to the filtering agents used. A large variety of agents of various origins exist from which to choose, the most common being paper or board and fabric, nonwoven material or wound wire. In certain cases, but far less often, cartridges made of plastic materials are used. The most commonly used filters are chamber filters and wound-cartridge filters.

FILTER PAPER

The filter paper used must be of excellent quality, with a solidity sufficient to withstand pressure and prevent tearing. It must also be resistant to acids and bases. Filter paper comes in the form of thick or thin disks and in plates or bands.

Various porosities are available, but 10 µm is the one most often used because it permits the filter cake to be recovered with the least possible liquid.

FABRIC

Fabric(s) or nonwoven material(s) are used in a variety of forms, porosities and textures, owing to the many types of equipment used, as well as to their subsequent application (disks, bags, bands, panels, etc.). These materials must exhibit excellent resistance to pressure, tensile strength and the various corrosive liquids, yet also have a calibrated porosity.

A filter aid (diatomaceous earth or cellulose) must frequently be used with the fabric to protect it from encrustations, to facilitate its washing and to refine filtering while considerably increasing the filtering surface.

FILTER CARTRIDGES

A large variety of cartridges are available. Some are very "technical" and meet precise quality requirements when filtering to less than 1 µm is necessary. Others have specific characteristics or can contain activated carbon; however, in general, the most commonly used cartridges are made of polypropylene and have filtering thresholds of approximately 1, 5, 10, 25 and 50 µm. Pleated cartridges also exist with a very large filtering surface made of disposable paper or washable and reusable fabric.

GOALS OF FILTERING

The main purpose of filtering is optimum clarification of the liquid and total elimination of solid particles. The three major factors that affect these results are the filtering surface, the filtering fineness and the filter flow rate.

The filtering surface must be large enough to allow liquid to pass through without excessive pressure drop and to recover a volume of sediment in relation to that generated by the bath. Possible use of filter aids or of activated carbon must also be taken into account. The recommended surface is 1 m^2 or a 3- m^3/h flow rate with chamber and panel filters. In the case of cartridge filters, a flow rate of 0.6–0.8 m^3/h is recommended per 10-in. cartridge.

The filtering fineness is frequently determined by chemists or bath manufacturers according to predetermined requirements. (*Warning*: The clogging rate increases with flow rate but much quicker.) Saving on filter size can actually lead to considerable operating cost overruns (owing to high cleaning frequency).

Polypropylene cartridges enable a cartridge model to be chosen with a precise filtering threshold (e.g., 5 µm). According to the efficiency of this cartridge, most particles larger than 5 µm are stopped.

Precision is less good with commonly used papers, and a range of 10–25 µm of initial porosity should preferably be chosen. With respect to fabrics, porosities vary considerably according to the manufacturer and the