ULTRAPURE GASES

HOW PRODUCTION TECHNOLOGIES INFLUENCE THE SURFACE QUALITY OF ULTRACLEAN GAS-SUPPLY EQUIPMENT: MATERIAL AND CONSTRUCTION

(Fourth of four parts)

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The increasing memory capacity and decreasing structural sizes associated with VLSI/ULSI technology place ever higher demands on process media and equipment. The suitability of components for systems with high-purity requirements depends essentially on these four characteristics:

1. Leakage rate.
2. Corpuscular purity.
3. Chemical cleanliness.
4. Purging characteristics/retention capacity.

For ultraclean gas components, these requirements are related to surface, material, and construction.

The first three articles in this series demonstrated how the topography, structure, purity, and energy state of a surface can be altered, and how the required characteristics can be created during equipment manufacture. Thus far, it has been shown that optimization of a surface with regard to particle release and chemical purity is not in itself sufficient. The component's material and construction must also be taken into consideration, since these, too, can greatly influence quality. Among the issues relating to these two concerns are material purity, dead zones, sizes of medium-wetted surfaces, internal and external leakage rates, purgeability, and the like.

This article will explain how both the material and structural design can be instrumental in eliminating sources of contamination, as well as how geometric surfaces as a measure of absorption and desorption rates can be reduced.

MATERIAL

In addition to specific physical characteristics (spring material, diaphragm, valve bodies, and the like), the basic requirements for metallic components in ultraclean gas systems are as follows:

- Corrosion resistance (resistance to process media and moisture penetration).
- Electropolishing suitability (the material must be polishable by means of chemical or electrochemical surface-treatment procedures).
- Material purity (minimization of tendency to form nonmetallic material inclusions and segregations).
- Cutting properties.
- Weldability.

The materials that have been used in ultraclean gas components to date are AISI 304 (1.4301), AISI 304L (1.4306), and (most frequently used) AISI 316L (1.4404 or 1.4435). AISI 304, which has the highest carbon content, is hardly ever used because of its carbide-forming tendency.

The characteristics of an austenitic chromium-nickel steel depend on many factors, including the following:

- Manufacturing procedures (melting, casting, forging, drawing, and the like).
- Manufacturing parameters (cool-down speed, heat treatment, type and amount of deoxidizing/denitrating agents, and the like).

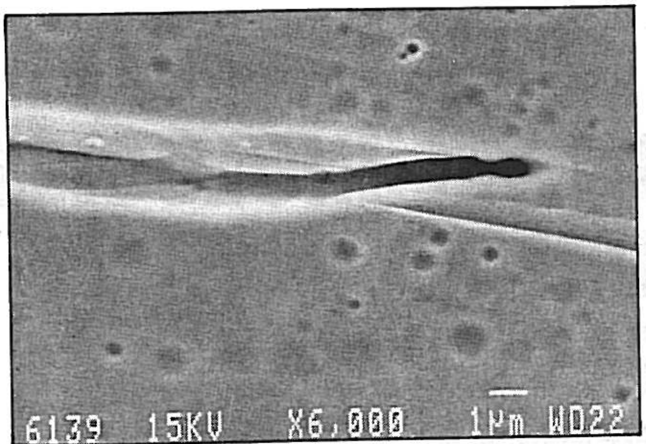

Figure 1: Inclusion lines exposed by electropolishing. These lines are usually filled with a multitude of particles. An ultrasonic treatment has removed all nonmetallic inclusions from the segregation line (6000 ×).

- Composition/analysis (e.g., chromium, nickel, iron content).
- Auxiliary elements (added as, e.g., manganese, molybdenum, copper, titanium, niobium, aluminum, sulfur, nitrogen, carbon, phosphorus).
- Surface treatment (surface structure, pollutants, tensile stress, and compressive strain).

An alteration in even one of these variables can influence the material quality. In order to maintain material characteristics that can be extensively reproduced, it has been necessary to severely restrict the essential parameters. For example, if defined solely according to DIN 1.7440, none of the above materials would fulfill the requirements of ultraclean gas process technology, particularly with regard to particle generation in the electropolished condition. The most frequently used materials, 1.4404 and 1.4435, tend to exhibit an increased formation of segregations due to their molybdenum content.

Figures 1 and 2 show nonmetallic inclusions exposed by electropolishing. Here, not only are the frequency and importance of segregation development dependent on the manufacturing process, but there is also great variation within the batch. Only a partial improvement can be achieved through material qualification and selective testing; a more precise material specification is needed to minimize such inclusions and segregations. Electroslag refining (ESR) offers the most effective remedy to this problem.

The media used in ultraclean gas process technology do not require the use of materials containing molybdenum. The process gases are so narrowly defined in purity and, especially, in low residual moisture content, and they are so closely monitored during service, that from the viewpoint of corrosion chemistry almost any austenitic chromium-nickel steel could be used. If moisture were to appear in the process gas, reaction products—some of which are extremely aggressive—would form, according to the type of gas being used. Molybdenum-containing materials are only conditionally resistant to this type of corrosion.

Because of the extended physical load on selected component parts (e.g., bellows, diaphragm, and spring), other materials must also be used—for example, 1.4310 AISI 301. This grade is sufficiently resistant to conditions

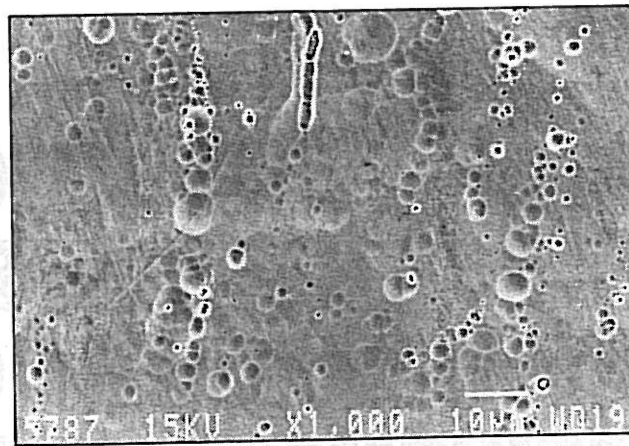

Figure 2: Surface with especially high inclusion rate after electropolishing. Each depression that can be distinguished in the surface represents a nonmetallic inclusion (1000 ×).

Figure 3: Corrosion caused by moisture and the simultaneous presence of chlorides. It can be seen that such a corrosion site has a strong tendency to generate particles (350 ×).

of normal use (i.e., dry process gas conforming to specifications). However, sudden moisture penetration can trigger corrosion, which may lead to increased particle expulsion or even cause component failure (Figure 3).

Note that preconditioning of the material by means of electropolishing does not necessarily lead to an improvement in the surface. With AISI 303 (1.4305), AISI 301 (1.4310), and Hastelloy C276, for instance, the particle-releasing tendency of a surface can actually be increased by electropolishing (Figures 4–7).

Since all of the materials used tend to form segregations and inclusions, some rethinking needs to be done. If one considers the most important requirements for gas-wetted surfaces—i.e., freedom from particles, low retention capacity, chemical purity, and corrosion resistance—then 1.4306 (AISI 304L) in ESR quality with restricted analysis (e.g., $<$0.02 carbon, $>$19 chromium, and $>$11 nickel) is the best alternative. Even with this grade, however, the problem of nonmetallic inclusions is not entirely eliminated. The release of nonmetallic particles that are inherent in the material must therefore be taken into account in the surface treatment (e.g., an ultrasonic treatment can be added following electropolishing to remove the exposed nonmetallic particles).

Packed Valve. Packed cylinder valves are sometimes used with corrosive process media that do not have special high-purity requirements (Figure 8). These valves have certain disadvantageous features—for example, a coarse, single-piece valve stem with a particle-dispensing thread in the gas chamber, and a voluminous, gas-wetted interior.

To be suitable for use with pure, corrosive gases, packed valves could be redesigned to minimize the gas-wetted interior space and to separate the upper and lower spindles (Figure 9), with the lower spindle transferring the shutting force to the metallic seat through a threadless, rotation-free movement. (Note that these valves are characterized by high decontaminating speeds during purging.) A further improvement can be made by fitting the valve with a pneumatic actuator (Figure 10). In addition to the safety advantages this would provide, particle release would also be diminished through the controlled and damped lift during spindle travel.

Bellows Valve. High-purity gases require the use of components that do not permit atmospheric contamination through the diffusion of air back into the system. External leaks must be either totally prevented or at least kept within a range of 10^{-10} mbar L/sec. Many believe such a low leakage rate can be achieved only through the use of metal seals.

When selecting valves for use in systems that will convey particle-free media, bellows valves should be considered on the following characteristics, not just their good leak-tight features:

- The bellows is contained in a dead-end flow path, and its folds create additional dead space.
- The bellows disturbs the gas-displacement characteristics of the overall system.
- The rough surface of the bellows increases the effective surface area and the outgassing rates.
- Most bellows materials do not stand up to all corrosive gases. In addition, residual moisture trapped in dead spaces can affect operation.

Diaphragm Valve. Diaphragm valves have the following characteristic features (Figure 11):

- Small, medium-wetted surfaces.
- Negligible dead volumes.
- Interior designed for easy electropolishing.
- Good gas-displacement characteristics.
- High leak-tightness integrity.

Valves at this level of technology can be even further improved by varying the configuration of the lower spindle assembly with regard to the evacuating aptitude. Figure 12 shows the influence of differently shaped spindle heads, together with variously designed spring-

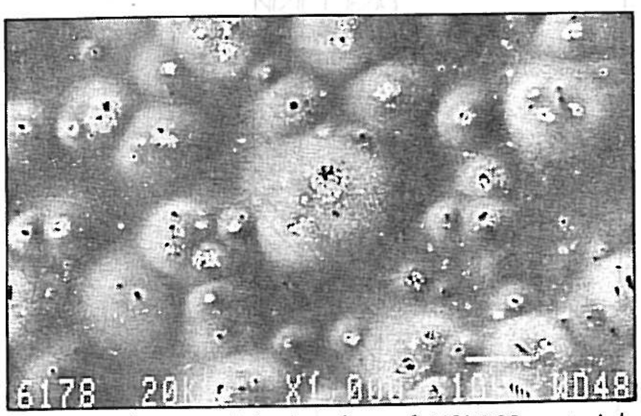

Figure 4: Electropolished surface of AISI 303 material. The dark areas are the openings of segregation lines, while the light areas are the nonmetallic particles generated from the segregation lines (1000×).

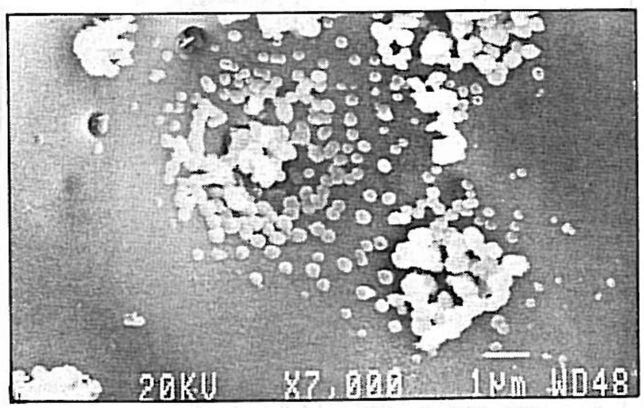

Figure 5: Detail of electropolished AISI 303 surface shown in Figure 4. Particles are mainly manganese and copper compounds; particle concentration on this type of surface can be as high as 10^6 particles/mm² (7000×).

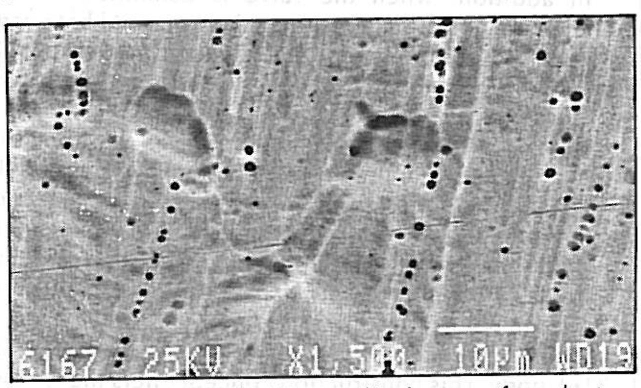

Figure 6: Surface of AISI 301 diaphragm after electropolishing. The dark dots are cavities from released carbide inclusions (1500×).

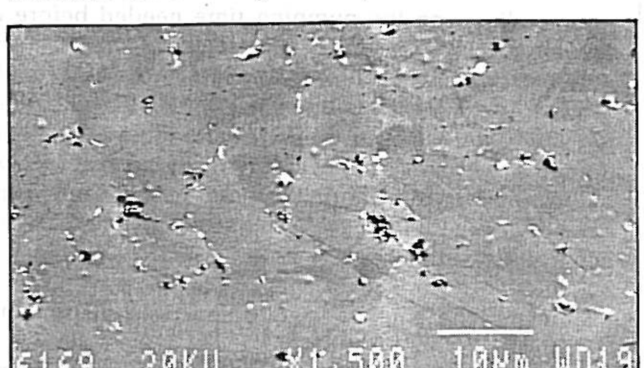

Figure 7: Surface of a Hastelloy 276 diaphragm after electropolishing. The light areas are molybdenum dispersions appearing at the grain boundaries of the material. Particles are between 0.3 and 1.0 μm, at concentrations between 10^4 and 10^5/mm² (1500×).

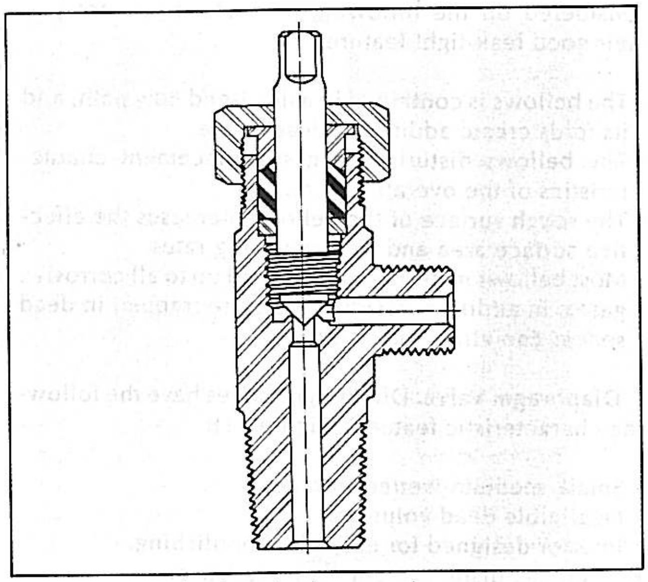

Figure 8: Standard packed-type valve.

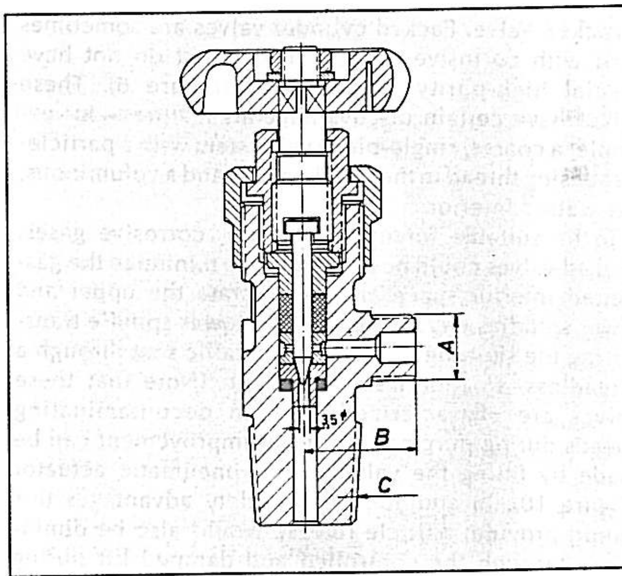

Figure 9: Packed valve with divided upper and lower spindle for pure process gases.

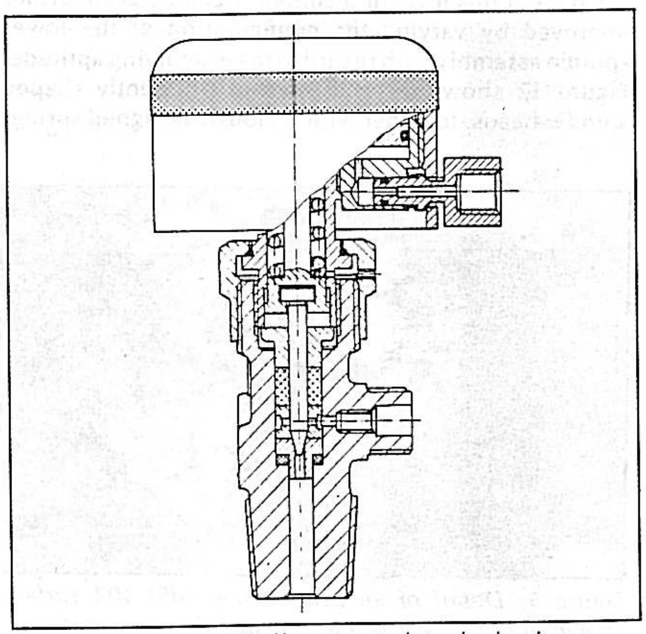

Figure 10: Pneumatically operated packed valve.

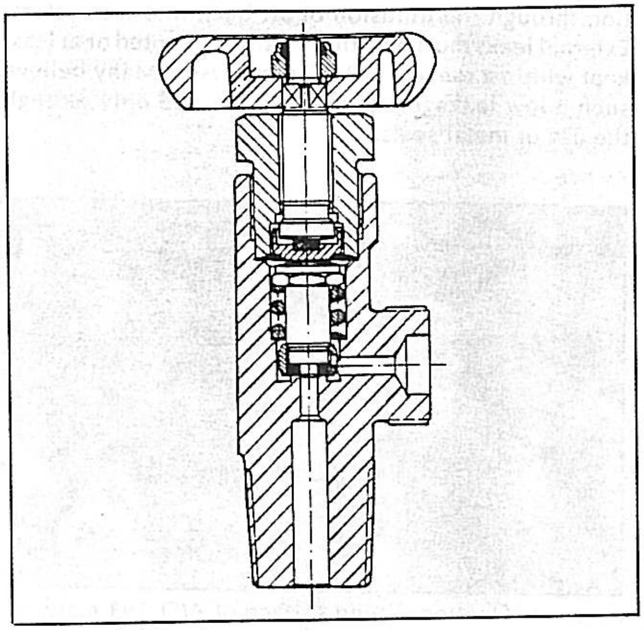

Figure 11: Diaphragm valve.

support discs, on the pumping time needed before a vacuum of 1×10^{-2} mbar is reached.

Further attempts to eliminate particle sources through design led to the development of the ultrapure diaphragm valve with tied lower spindle (Figure 13). (See the third part of this series in the April issue of *Microcontamination* for information on particle-releasing behavior.) The following additional improvements were achieved with this design:

- Deleting the torsion-loaded, particle-dispensing spring.
- Reducing gas-wetted inner surfaces by one-third compared to a conventional, spring-supported diaphragm valve.
- Eliminating dead volume.
- Placing the flat valve seat in a hemispherically shaped valve interior, resulting in greater ease in electropolishing and a more favorable setup for the displacement of process media and purging gases.

In addition, when the valve is combined with a pneumatic actuator, damaging vibrations and between-positioning can be avoided, and dynamic particle expulsion can be eliminated to the greatest possible extent (Figure 14).

Bloc Manifold. The bloc manifold was developed in the attempt to optimize total ultraclean gas-supply systems. The adoption of an extremely compact construction form encompassing several functional units reduced both the medium-wetted surfaces and the volume of a whole supply panel. Shutoff valves, pressure regulators, check valves, safety valves, and a vacuum generator were integrated in a massive, AISI 316L body. This construction concept offers the following advantages:

- Minimal dead volume.
- Minimal gas-wetted surfaces.
- No threaded or welded connections.

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- No tubing interconnecting elements internally.
- Optimum conditions for application of suitable processing, polishing, and cleaning techniques.
- Final assembly, inspection, and packing done in a cleanroom.

Figure 15 illustrates the particle-releasing activity of the bloc manifold (note the speedy reduction in particle concentration as the purging time increases). These extremely low values can be guaranteed directly at the factory, conditional on the duration of the standard purging procedure (using pure nitrogen) that follows final assembly in a Class 10 cleanroom.

CONCLUSION

The quality of ultraclean gas equipment can be markedly improved through proper material choice and specifications and through the adoption of a construction concept that adheres strictly to the requirements of ultraclean gas-supply systems. The most important factors to be considered are material purity (inclusion rate), reduction of the medium-wetted surface, elimination of dead volume, interior layout favorable to medium displacement and polishing, and leak-tightness.

As described throughout this series of articles, it has been shown that it is possible to manufacture high-quality equipment with narrow tolerance limits for ultrapure media when the following conditions are met:

- Prior analysis of the whole process (parameter studies).

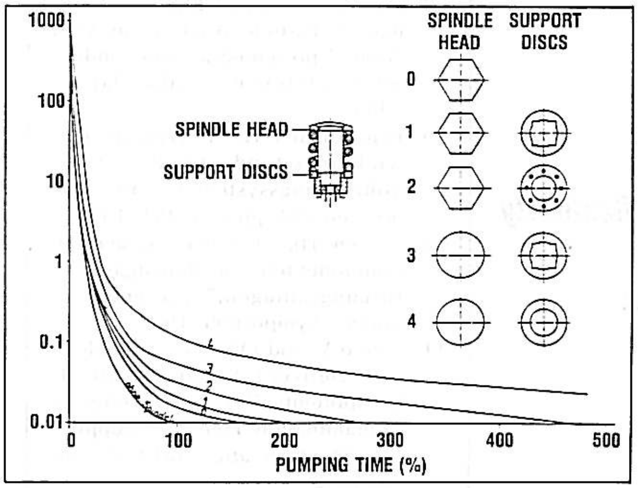

Figure 12: Evacuation time of diaphragm valve interior, depending on the configuration of the lower spindle complex.

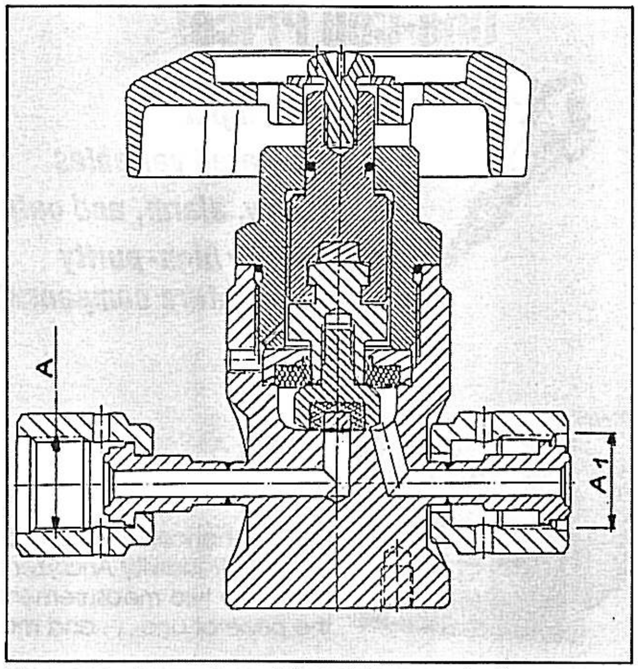

Figure 13: Line valve with tied lower spindle.

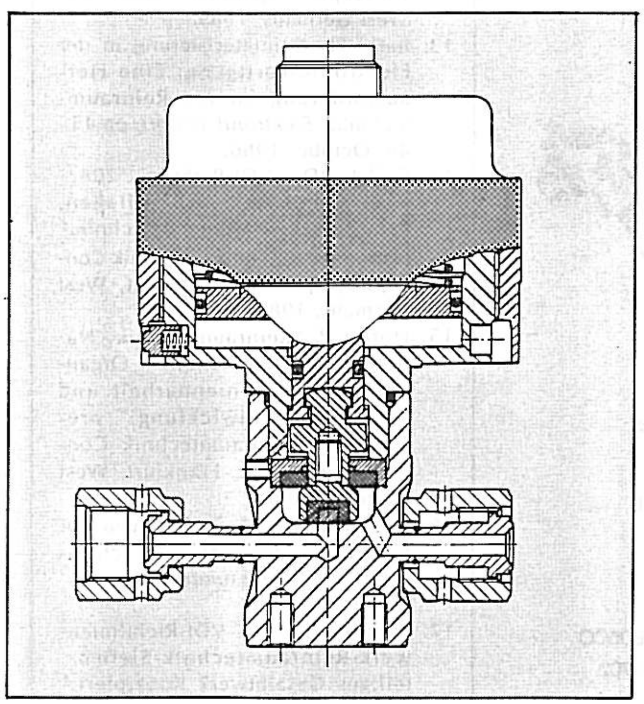

Figure 14: Pneumatically operated diaphragm valve with tied diaphragm/spindle.

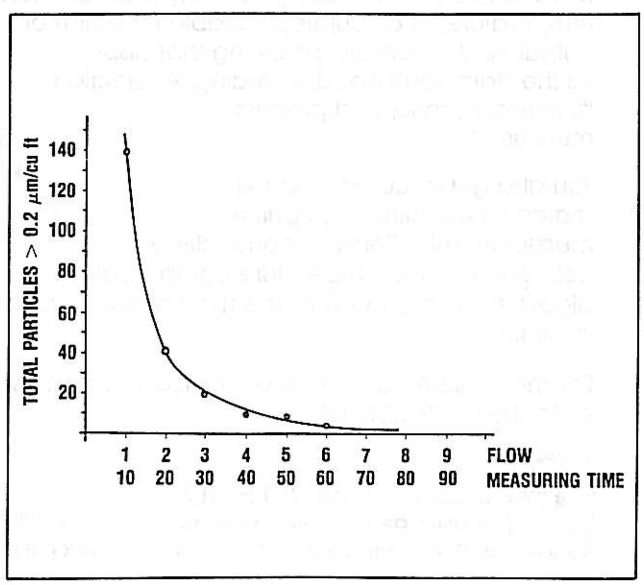

Figure 15: Particle-releasing activity of bloc manifold.

- Consideration of all factors influencing component quality (design, material, surface topography/cleanliness, and the like).
- Adaptation of inspection technology to the processing.

The following supplementary acceptance-testing criteria would be useful:

- Long-term particle-release evaluation (e.g., material purity, surface topography/cleanliness).
- Particle release during alternation of load (valve design).
- Purging and outgassing conduct (medium-wetted absolute surfaces, dead volumes).

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(Editor's note: The above references pertain to the complete set of articles in this series, which ran in the February, March, and April issues as well as this month's edition. Due to the delay in printing the references, please note that the final number of entries is 49, not 51, as originally called out in text.)

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