Gas Cleanliness

Staff of Kaufman & Robinson, Inc.

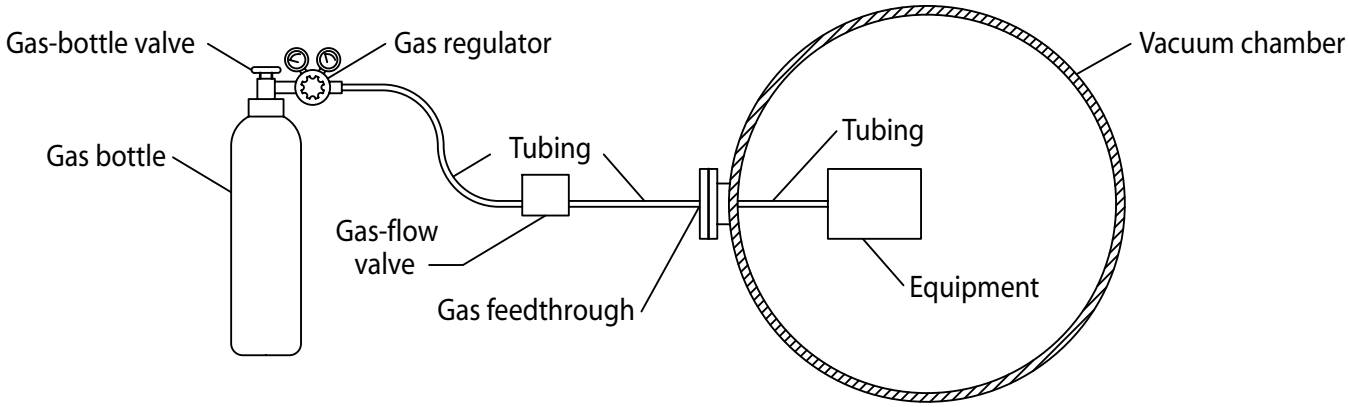

Fig. 1. Typical gas-bottle installation

INTRODUCTION

Gas cleanliness is important to some vacuum-process equipment and processes. For example, contamination can decrease the lifetime of hollow cathodes and plasma-bridge neutralizers by a factor of ten or more. The techniques required to assure gas cleanliness are reviewed herein.

A typical gas-bottle installation is shown in Fig. 1. The gas is supplied from a gas bottle to a two-stage gas regulator, which decreases the pressure from the high value inside the gas bottle to about one to two atmospheres after the regulator. The gas then flows through tubing to the gas-flow valve, which may also be called a mass-flow controller. There is additional tubing from this valve to a gas feedthrough at the vacuum-chamber wall. Inside the chamber, there is more tubing from the gas feedthrough to the device that uses the gas. Gas fittings connect sections of tubing together and to other components.

GAS BOTTLE

The gas bottle should contain gas of 99.999% or greater purity. Sometimes welding-grade argon is substituted for high-purity argon. Make sure this hasn't happened.

GAS-BOTTLE REPLACEMENT

The routine replacement of an empty gas bottle is a common cause of gas contamination. When a new gas bottle is attached to the gas regulator, a volume of atmosphere is trapped between the bottle and the regulator. Immediately opening the valve on the gas bottle will result in this trapped volume contaminating the pure gas inside the bottle.

The proper procedure is to leave the gas-bottle valve closed, pump down the vacuum system, and open all valves from the vacuum system up to the gas bottle - including fully opening the gas regulator. After the gas line is pumped out, as indicated by the vacuum chamber reaching a normal base pressure (≤1×10 6 Torr), close the gas regulator and the gas-flow valve.

Next, open the gas-bottle valve enough to show the bottle pressure on the pressure gauge before the regulator. Then open the gas regulator to give standard pressure after the regulator. If you do not have a standard pressure for use after the regulator, use 138 kPaG (20 psig). Open the gas-flow valve to give a flow of at least 10 sccm and continue for 15 minutes. This should remove essentially all of the contaminants adsorbed on the surfaces in the trapped volume between the gas bottle and the regulator and between the regulator and the gas-flow valve. Fully open the valve on the gas bottle. The gas-flow valve can then be closed. If desired, the regulator can also be closed. Proper replacement of gas bottles is necessary for gas cleanliness.

GAS REGULATOR

A high-purity, two-stage gas regulator should be used. A high-purity regulator has been cleaned ultrasonically and uses metal seals. A two-stage regulator maintains a more constant pressure, hence more stable process control.

TUBING

The total length of tubing should be minimized to reduce contamination.

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Polymer

Polymer is used here as a generic name for a variety of plastics and elastomers. All polymers that we have tested collect impurities upon exposure to atmosphere, then gradually release these impurities at vacuum. Some polymer tubings even permit contamination to diffuse through the tube. If stainless-steel loom covers a section of tubing, you may need to check with the manufacturer to find out the tubing material. In summary, don't use polymer tubing to transport clean gas!

Stainless Steel

Seamless stainless-steel tubing is the standard material for vacuum systems. Lubricants are used in the manufacture of this tubing. These lubricants are removed from the outside for cosmetic reasons. Unless the inside of the tubing has been cleaned, these lubricants are still there to contaminate gas for months or even years.

There are standard processes, such as electropolishing, that will clean the inside of stainless-steel tubes. Unless you know that an acceptable cleaning process has been used and the tubing was capped to keep out contamination after cleaning, assume that the inside of stainless-steel tubing requires cleaning. For long sections of tubing, there is no practical alternative to one of these standard processes. Simply rinsing the inside of a tube with a solvent is not an adequate substitute.

It is possible to clean short sections of stainless-steel tubing by hand. Use pieces of stiff wire to push through wads of lint-free tissue that have been saturated with alcohol. Repeat this process until the emerging wads show no dark residue. Rinse the inside of the tube with distilled water, then blow-dry with dry nitrogen or another clean, dry gas. Debris left inside a clean tube after cutting it should also be removed.

TUBE FITTINGS

The number of fittings should be minimized to reduce the likelihood of leaks. The fittings should be cleaned ultrasonically before being used. If ultrasonic cleaning is not available, use lint-free tissue and alcohol, followed by a rinse with distilled water and drying.

Fittings are used to connect sections of tube together or to connect tubes to other components. Different fittings have different tolerances for being opened, then reconnected. In general, any fitting connection to a gas tube should not be opened any more than necessary.

Teflon® tape should be used for threaded seals at the gas regulator. There may be a very small amount of leakage at such a joint, but the clean gas is not contaminated if the joint is kept under positive internal pressure with clean gas.

Swage-type fittings, in which the gas-tight seal is obtained by swaging a ferrule around the tube, are particularly sensitive to being opened, then reconnected. Each time a swage-type fitting is opened on the atmosphere side of the vacuum chamber, the end of the tube should be cut off, a new ferrule installed, and the fitting retightened following the manufacturer's instructions.

Swage-type fittings inside the vacuum chamber should be considered on an individual basis. It is likely that a small leakage out of the tube to the lower pressure vacuum environment will be acceptable. In practice, few problems have been encountered due to the reuse of ferrules inside a vacuum chamber, including normal retightening without reference to the manufacturer's instructions.

INITIAL SYSTEM PURGE

An initial purge is recommended after a gas-bottle system has been assembled using clean components. Connect a gas bottle as described above in "Gas Bottle Replacement," except that the flow of 10 sccm or more should continued for at least an hour.

PURGE AFTER BREAKING VACUUM

Opening a vacuum chamber will expose the gas line back to the gas-flow valve to atmospheric contamination. After the subsequent pumpdown, a flow of at least 10 sccm is recommended for 15 minutes before starting operation.

A common alternate procedure is to maintain the gas flow at 10 sccm or more during the time that the vacuum chamber is open. Operation can then be started as soon as the vacuum chamber reaches a satisfactory pressure. If it is necessary to check the base pressure of the vacuum chamber, the flow can be stopped after the subsequent pumpdown reaches a pressure of less than one milliTorr.

A purge after breaking vacuum is probably the least important of the procedures described herein to assure cleanliness. It should therefore be the first procedure omitted for processes and equipment that are only moderately sensitive to gas contamination.

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