Check your gas-handling system to maximize laser performance

A well-designed gas-distribution system assures purity at point of use. Here's how to set up and maintain your system to optimize gas-laser performance.

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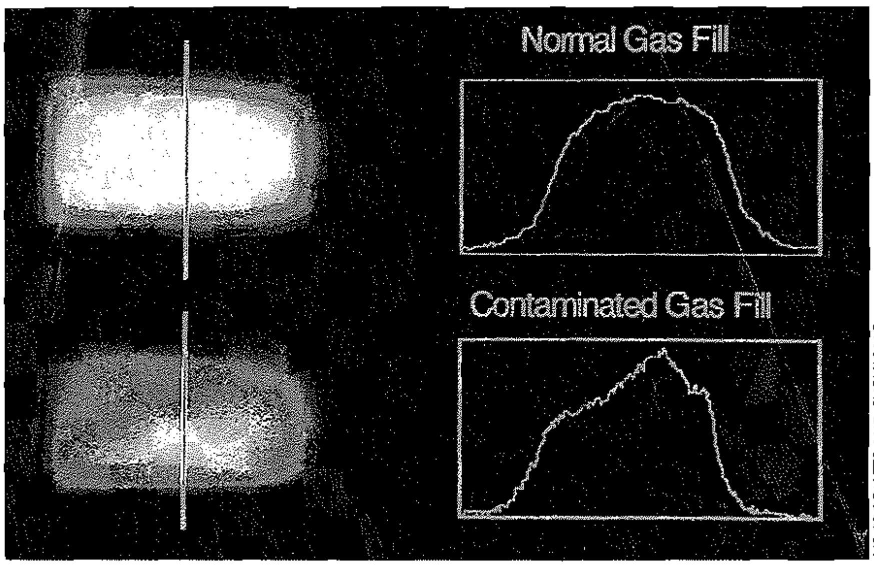
Gas purity is a key factor in optimizing excimer and carbon dioxide laser performance. Gas impurities in these laser systems degrade beam profiles, reduce output powers, and shorten laser lifetimes. Much has already been done to reduce impurities—cleaner supply gases are now available, and laser systems are being constructed of materials that produce significantly lower amounts of gas impurities.

But a source of gas impurities that is often overlooked is the gas-distribution system a user constructs to connect a laser to its gas supply. This source of impurities is often out of the gas supplier's and laser manufacturer's control. Many gas-laser users have little or no experience with setting up "tight" gas-distribution systems and may compromise laser performance by inadvertently introducing gas impurities into the delivery system.

Site preparation guides that most laser-system manufacturers supply cover the basic requirements for an acceptable gas-distribution system. Users should also consult their gas suppliers to ensure that the gas-distribution system is properly designed for optimal laser performance.



Energy profiles indicate purity of KrF gas blend. Top scan and intensity plot show a "clean" gas fill. Bottom scan and plot reflect contamination with 200 ppm of air.

Gas storage and pipelines

This article discusses halogen-gas storage, pipelines, seal connections, leak checking, and halogen passivation of pipelines—all key aspects of gas delivery systems. It also reviews gas-distribution-system maintenance.

Gas storage, especially of such toxic halogens as fluorine and hydrogen chloride gas blends, is the first step in gas management (see *Laser Focus World,* Feb. 1991, p. 75, and Mar. 1992, p. 81). Halogen-gas blends should be stored in well-ventilated and explosion-proof cabinets. There should be a purge line from an inert gas supply (helium) on the halogen-gas line to remove residual gases from the line and to minimize air infiltration during cylinder changeover. Most excimer-laser gas suppliers can supply approved gas cabinets to safely store and deliver halogen-gas blends [**Editor's note:** *Excimer lasers are now available with on-board halogen generators;* see p. 124].

The pipelines connecting a gas supply to a laser system can compromise gas quality and laser performance in several ways. First, contaminants previously absorbed to the inner surface of the pipeline can be introduced to the gas flowing through the pipe. Second, atmospheric impurities can diffuse through the pipe wall itself or at connection points such as valves. Third, in the case of excimer lasers, metal corrosion can occur unless prevented by halogen passivation of the inner surface of the pipeline.

Piping materials routinely used on carbon dioxide lasers are flexible plastic (PVC, Teflon, or polyethylene) and rubber hosing. These materials are relatively inexpensive and considered chemically inert, but they can pass atmospheric impurities—that is, oxygen, nitrogen, and water vapor—into the gas-supply system. This is especially a problem when plastics or rubbers are used with carbon dioxide laser systems that operate at subambient pressures. A test of Teflon piping flushed with helium for six hours revealed increased levels of oxygen and nitrogen in the helium gas stream of approximately 15 ppm for each impurity.[1]

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Plastic piping systems are also sources of hydrocarbon and organic impurities containing low-molecular-weight components that are adsorbed on the inner surface of the pipe. These impurities can be swept off or can react with gases introduced into the pipe. Organic impurities can then be resorbed on laser windows and mirrors, reducing power output by damaging the optics. Hydrocarbons at the 100-ppm level can reduce the output of carbon dioxide lasers by 10% or more.[2]

Gas contaminants such as oxygen,

Integrated halogen generator makes excimer lasers easier to use

Anyone who has worked with excimer lasers is familiar with the problems of handling poisonous, corrosive halogens such as fluorine (F_2) and hydrogen chloride (HCl). The problems include gas leaks, passivation of gas lines, regulator corrosion and failure, and so on. Whether F_2 and HCl gases are obtained as 5% in helium or as fully diluted premix, they are not only dangerous, but their purity slowly degrades during storage in the bottle. And while gas-fill lifetimes now exceed 10^7 shots for most excimer gas mixtures, laser gas refilling, or at least injection of fresh halogen, is still necessary on a regular basis.

Users often list handling halogen gases as the biggest drawback of using excimer lasers. Until recently, is was difficult to avoid gas problems. An internal halogen generator for excimer lasers from Lambda Physik GmbH (Göttingen, Germany), which was introduced at LASER 93 in Munich, Germany, eliminates the need for handling noxious gases. This system generates a nearly continuous supply of freshly prepared, ultra-pure halogens, eliminating the need for an external halogen bottle.

Microprocessor control

The integrated halogen generator is a completely sealed unit that contains all necessary stable chemicals and is controlled by the laser's microprocessor. The generator is housed entirely within the laser, and for added protection, is enclosed in a safety vessel that includes a halogen absorber. During a fill cycle, the microprocessor signals the need for halogen generation, and the chemicals are activated in a reactor chamber to generate sufficient gas (HCl or F_2) for one fill of the laser. This gas immediately fills the laser chamber to the correct partial pressure.

Other than the obvious advantages of increased safety, lower operating costs, and convenience of operation, integrated halogen generation ensures gas purity. Laser output power and gas-fill lifetime are highly dependent on gas purity. Because the halogen is freshly produced for each fill, Lambda Physik can guarantee the purity of the halogen, ensuring that the laser performs to specification and reducing the need for routine maintenance such as optics cleaning.

Both the F_2 and HCl halogen generators are charged at the factory with sufficient chemicals for at least 100 laser fills, which can last for several years of XeCl operation. The generator can either be fully recharged at Lambda Physik and returned, or exchanged by air freight, avoiding any laser downtime.

More convenient excimers

The excimer-laser market, which increasingly includes users less expert in laser technology, continues to grow as excimer lasers become more reliable and simpler to operate. Integrated halogen generators make these lasers more convenient for existing applications, as well as more accessible to potential users.

Currently, integrated halogen generators are available with the entire scientific/medical LEXtra series as well as the Lambda 3000 and Lambda 4000 series industrial lasers. Halogen generators can also be supplied as a retrofit for late models of all these lasers. In addition, the halogen generator for the Lambda industrial lasers is available with an optional pressurizer that enables the source to supply periodic halogen injections to extend gas-fill lifetimes.

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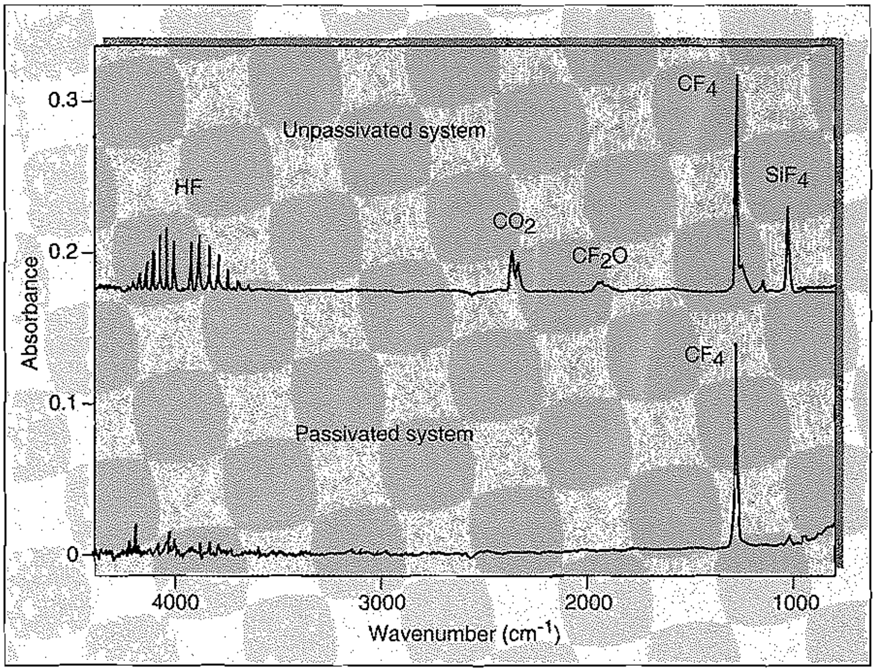

FIGURE 1. Fourier-transform infrared (FTIR) spectrum reveals several impurities in 5% fluorine and helium gas blends in unpassivated gas-distribution system (top). Fully passivated system contains only CF_4 (bottom).

nitrogen, water, and carbon dioxide affect excimer-laser performance and can reduce power output by as much as 17% per 100 ppm of impurity.[3] The beam profile is also compromised by air impurities in the gas supply. Air impurities can adversely affect the beam profile for an excimer laser operating when approximately 100 ppm of air is added to the KrF gas fill (see photo on p. 123).

Stainless steel or copper piping is preferred for gas lasers; even metal pipes can introduce hydrocarbon impurities into the gas streams via desorption from the inner surface walls. Metal piping put into gas service must be precleaned with a solvent to reduce impurities.

Only stainless steel or copper piping should be used to deliver fluorine, hydrogen chloride, and other halogen gases. Soft copper tubing is not recommended because it is susceptible to pinhole leaks and leaks at seals. These metal pipes must be scrupulously cleaned before they are passivated and placed into halogen service. If an excimer laser is to be used with both fluorine and hydrogen chlorine, then each gas should have a dedicated gas line to minimize introduction of impurities during cylinder changeover or pipe passivation.

Seal connections and leak checking

Regulators and valves must be viewed as potential sources of impurities. For example, polyhalogenated hydrocarbons are commonly used as lubricants in regulators.[4] These lubricants may be sufficiently volatile to introduce organic impurities into the gas stream; thus, regulators should be dismantled and scrupulously precleaned by the vendor. Shutoff valves, such as ball valves, should also be dismantled and precleaned with a solvent to remove lubricating greases.

A gas distribution system must be thoroughly leak-tested before commissioning. The gas lines should be evacuated and pressurized with helium to at least the working pressure of the system. While under pressure, the system should be examined for gas leaks with a leak detector. If there are no leaks, then the gas-distribution system is considered "tight." It should then be evacuated and purged a few times with the operating gas feed before use.

Halogen passivation

Halogen pipelines must be passivated before use to impart a protective layer onto the inner surface and render it inert. The surface of stainless steel has a passive layer of chromium oxide that enables it to resist rusting. Pipelines in halogen service develop a metal halide surface layer, such as iron fluoride or copper fluoride, during passivation.[5] This surface layer "seals" the surface

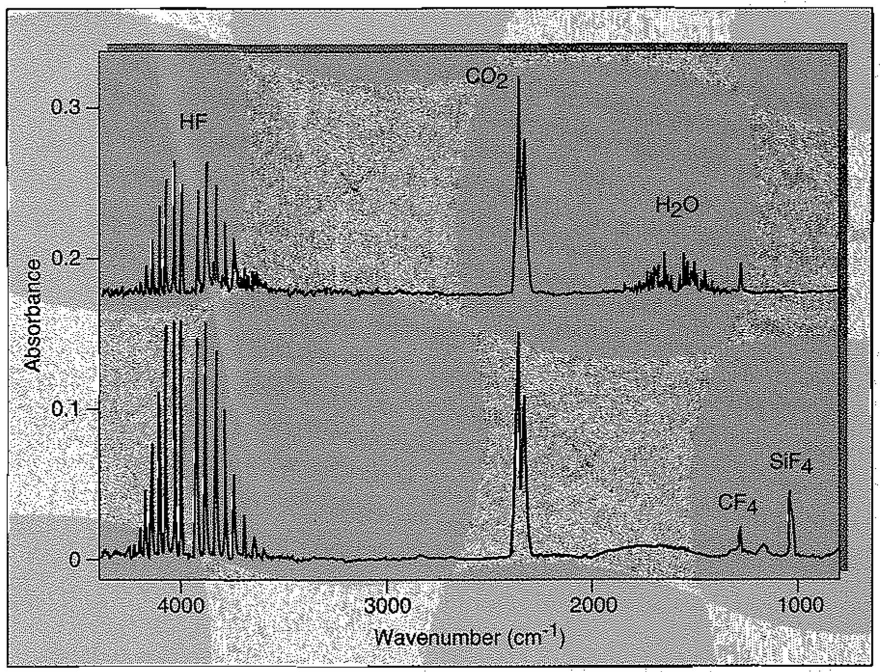

FIGURE 2. FTIR spectrum of gas fill within a minute of filling shows evidence of an air leak (top); after two minutes, spectrum no longer shows water peaks (bottom).

and impedes halogen penetration along the microscopic boundaries of the metal grains. Halogen penetration can promote metal fatigue, leading to leaks or catastrophic failures.

When passivating a gas system, the material must be treated under expected operating conditions. For example, if a 5% fluorine in neon blend will be transported to an excimer laser at 60 psig of pressure, then the gas-delivery system should be passivated step by step until these conditions are met. The system should first be exposed to the 5% fluorine blend at 15 psig. After approximately 30 minutes, the system should be flushed with helium and evacuated, then refilled with 30 psig of 5% fluorine, and so on. This flush-and-evacuation process should be repeated until the gas system is pressurized with 60 psig of 5% fluorine.

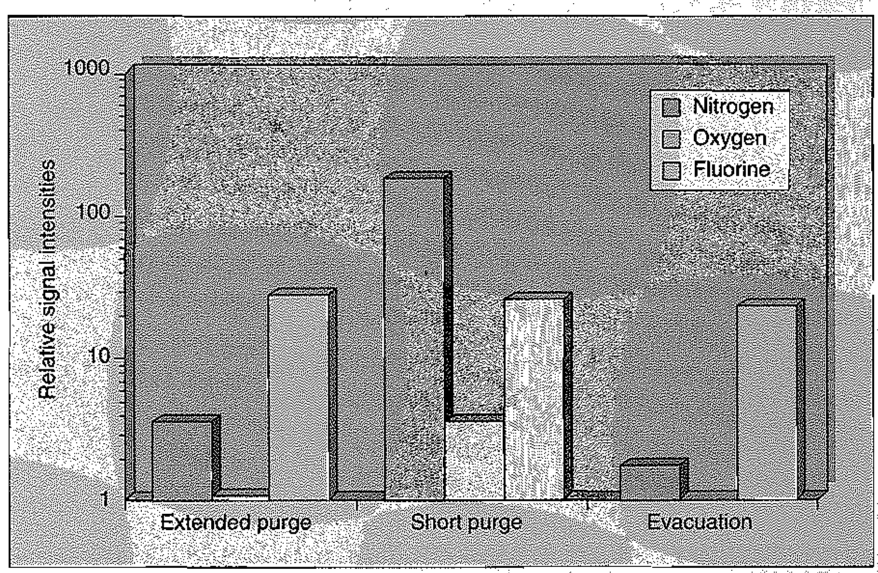

FIGURE 3. Evacuation with helium, which removes most of the nitrogen and oxygen from the gas fill, is the most efficient method of cleaning a fully passivated gas-distribution system that has been exposed to dry air before refilling.

In some cases it may be necessary to passivate three or four times at operating pressure to assure the treatment is complete. For example, during fluorine passivation of stainless steel, gas impurities are released by the reaction of the halogen with the metal surface. The chromium oxide layer on the steel, as volatile as chromium fluorides, is removed during passivation, and an iron fluoride layer is formed in its place. Additional gas impurities, such as hydrogen fluoride, carbon dioxide, carbonyl fluoride, and silicon tetrafluoride, are released during fluorine passivation of stainless steel (see Fig. 1).

If the system is improperly passivated, excimer-laser users run the risk of introducing these residual gas impurities into the halogen feed gas. Because the impurities are exactly like those often detected in lasers operating on a cylinder of "bad gas," the simple, but false, conclusion is drawn that the gas supply is contaminated and new gases must be acquired. When a leak-tight and properly passivated gas supply system is constructed, the user, the laser manufacturer, and the gas supplier are all saved considerable downtime cost in locating and correcting contamination in the distribution system.

System maintenance

Maintaining a "tight" system after passivation is also imperative. Exposure of the passivated surface to air, especially humid air, can severely compromise the passivated surface and be another impurity contributor (see Fig. 2). As an experiment, a pipeline for a fluorine blend was purposely contaminated by introducing a small air leak. The air had a relative humidity of 65%, and the moisture present in the air rapidly converted to hydrogen fluoride. Carbon dioxide and silicon tetrafluoride were also observed in the gas fill. The presence of silicon tetrafluoride suggests that the passive film was altered, requiring repassivation.

Materials in contact with hydrogen chloride must be kept dry and the gas system must be kept leak-free because "wet" hydrogen chloride is especially corrosive. And while carbon dioxide gas systems do not require the passivation procedures needed for excimer laser systems, any gas-delivery system opened to air should be purged and evacuated before it is refilled.

Evacuation is recommended for gas-delivery systems because purging does not always remove all residual air and moisture. In a study of a fluorine gas-delivery system, the relative amounts of nitrogen and oxygen that contaminated a fluorine/helium gas blend were measured by a mass spectrometer and compared for three different methods of system preparation (see Fig. 3). The first method was a "long" purge of the pipeline with helium at 45 psig for 20 minutes. The second was a "short" purge that consisted of a static helium fill at 45 psig, venting the pipeline to atmospheric pressure, refilling the line with another static gas fill, and then venting the system to atmospheric pressure before introducing the fluorine gas blend. The third method was an evacuation of the system to 10 mbar of pressure before filling with the fluorine gas blend.

The data indicated that the relative amount of fluorine in the system after filling was the same, but the amount of air contaminating the gas fill varied significantly according to method of system preparation. Helium purging by itself is a slow process. Evacuation proved to be a quicker means to a clean gas-distribution system.

The changeover of a gas cylinder can also introduce gas impurities when the delivery system is opened, because some air will enter. After a gas-cylinder changeover, the gas line should be evacuated, filled with gas from the new cylinder, and evacuated again before the line is refilled for use. When poor laser performance is noted after changing the cylinder, it can often be traced to a failure to remove the air from the gas feed.

Inexperienced gas-laser users may compromise laser performance by inadvertently introducing gas impurities into the delivery system.

As carbon dioxide and excimer-laser applications continue to expand, their success will depend on laser reliability. The availability of clean supply gases, the use of "tight" gas-delivery systems, and the construction of more-efficient lasers will increase reliability, thereby enhancing laser performance. □

Acknowledgments

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REFERENCES

1. W. Schmidt, *Ind. Laser Rev.* 7(6), 5 (1992).
2. J. Larue et al., US Pat. #5,073,897 (December 17, 1991).
3. G. Jursich et al., *Appl. Opt. 31*(12), 1975 (1992).
4. D. Vassilaros and A. Varn, personal communication.
5. A. Cabrera, E. Karwacki, and J. Kirner, *J. Vac. Sci. Technol. A.* 8(6), 3988 (1990).

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