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PERFORMANCE OF ALUMINA-SUPPORTED Pt CATALYSTS IN AN ELECTRON-BEAM-SUSTAINED CO, LASER AMPLIFIER

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SUMMARY

The performance of an alumina-supported Pt catalyst system used to maintain the gas purity in an electron-beam-sustained (636) isotope CO_2 laser amplifier has been tested. The system characteristics using the two-zone, parallel flow reactor were determined for both continuous- and end-of-day reactor operation using on-line mass spectrometric sampling. The laser amplifier was run with an energy loading of typically 110 J- ℓ/atm and an electron-beam current of 4 mA/cm². With these conditions and a pulse repetition frequency of 10 Hz for up to 10,000 shots, increases on the order of 100 ppm O_2 were observed with the purifier on and 150 ppm with it off. The 1/e time recovery time was found to be approximately 75 minutes.

INTRODUCTION

Purification of gas mixes used in isotopic or long-life carbon dioxide lasers has become an increasingly active area of investigation. For ground-based applications where power and size requirements are not limiting factors, traditional catalyst technology may be employed, such as the well-developed alumina-supported platinum catalysts. In the present work, we have used an alumina-supported Pt catalyst to provide either on-line continuous or "end-of-day" purification of 13:2:1 He:N₂:¹³CO₂ laser gas mixtures used in a large-scale laser amplifier.

The laser amplifier system (CORA) is an atmospheric pressure, electron-beamsustained, pulsed discharge device with the parameters of Table I. The design emphasizes the use of clean and stable materials for maintaining the purity of the isotopic CO₂ used in the 6200- ℓ device. The catalyst system is incorporated into the amplifier by attaching it to an ancillary system that provides purging of the anode triple point and the electron-beam drift regions of the gain module assembly. Approximately 9 ℓ /sec are removed from the gain module, of which 3 ℓ /sec are sent through the catalyst bed housed in a separate unit.

The catalyst system was built to our specifications by Applied Photonics, Inc. It consists of a two 30" Iconel retort tubes each containing 4 to 5 mesh-size platinized

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pellets layered between stainless steel strainer elements. The tubes each have a surface area of approximately 7500 cm², are separately heated, and are arranged in a parallel flow configuration. Based upon vessel size and flow rates the time required to pass a module volume through the purifier is approximately 34 minutes. Typically the catalyst zones are held at 600 ± 5 °C during operation with the gas entering and leaving the purifier at room temperature.

Initial treatment of the catalyst after receipt from Applied Photonics was to heat the catalyst to 800 °C in steps which maintained a 100 mTorr or better vacuum above the catalyst. The catalyst was then held at 800 °C for one hour after outgassing was complete and allowed to cool. The system was flushed with a laser mix using ¹²CO₂, evacuated, and the heating process repeated to 300 °C. At this point the purifier was again flushed with laser gas, evacuated again, and the catalyst heated to 800 °C under a continuous flow of laser gas. The laser gas was discarded, the complete system pumped to 5×10^{-5} Torr and refilled with a clean gas mix. Initial experiments were performed with carbon-12 carbon dioxide. The quality of the laser gas in the module was monitored using a quadrupole mass spectrometer and the discharge behavior of the gas. Since the discharge diagnostics included color video as well as current and voltage monitors, it was very easy to spot changes in the gas composition either by changes in the discharge color or the presence of bright spots in enhanced field regions of the discharge, as well as by changes in the discharge impedence.

The quadrupole mass spectrometer was a Dycor 200M, 0-200 amu gas analyzer with computer control and data acquisition. The position available for sampling from the gain module was not directly on the flow loop, so the mixing time of the flow loop with other regions needed to be considered in measurements involving real-time monitoring of the gas composition. The mixing time was observed to be the order of 30 minutes. The pressure at the QMS was maintained at a tolerable level by providing a differentially pumped sample volume. The first aperture was a smalldiameter, thin-walled hole in a copper disk while the second was provided by a sampling valve provided by Dycor. A mechanical forepump was used as the differential pump. Calibration of the spectral intensities was made using the known composition of the commercial laser gas mixes and assuming constant ionization efficiencies. Examples of the data are shown in Figure 1.

RESULTS AND DISCUSSION

The first round of experiments involved running the system with the catalyst cold. The gas was sampled before and after the accumulation of 14,000 shots at 3-kJ energy loading per shot. The O₂ concentration rose 208 ppm from 135 ppm to 343 ppm; an oxygen generation rate of 30 ppm - L/kJ for the device. This was lower than expected based upon DVT studies in a single-shot device using similar materials. The reason is due the the ability to effectively electron-beam scrub the insulator surfaces of the discharge volume in the CORA device. We found that electron-stimulated desorption from the insulator surfaces produced large quantities of oxygen and water and that 15,000 shots of the electron beam (order 2 mA/cm² and 150 keV

at the insulator) and simultaneous flushing of the gas were required to produce a clean discharge and stable gas composition.

The catalytic purifier was then heated and the laser gas cycled for several hours. The plotted data in Figure 2 show that the oxygen concentration decayed with a 1/e time of 75 minutes.

For our application, use of the purifier at all times is desired. A typical run of the system is several short bursts of a few thousand shots within an hour of a 10,000 shot run at 10 pps and full energy loading and a run time of just over 17 minutes. The amplifier then requires a two-hour cool-down period. Under these conditions continuous on-line purification maintains a stable initial gas composition. For example, running the purifier at 600 °C while operating through the accumulation of 32,000 shots in one day resulted in a change in O₂ concentration, as measured within a half hour of the final firing, of 20 ppm over that at the beginning of the day. The gas composition recovered completely by leaving the purifier on a longer time. An estimated "dynamic" production rate, which ignores the issues of gas mixing times, is 20 ppm - L/kJ. This is two thirds of the rate of oxygen production during a run without the purifier operating.

Table I

CORA WIDEBAND AMPLIFER - GENERAL INFORMATION

- Electron-Beam-Sustained Pulsed Discharge
- Gas Mix: 13:2:1 He:N₂:CO₂
- Clean System, Designed for 13 CO $_2$ Isotope
- Parameters
 - 10-pps burst-mode operation of 10,000 shots
 - ~110-J/2-atm energy loading
 - ~5-mA/cm² electron beam
 - 25-kV, 3.5-kA discharge
 - ~2.5-m active length
 - 2.5 ms⁻¹ transverse flow through cavity
 - 2400 l flow loop volume 6200 l total volume



Figure 1. Mass Spectra for 13 C Isotope Showing Reduction in O $_2$ by Catalyst



Figure 2. Purifier Performance: Gas Cleanup After 14,000 Shots