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Demonstration of a vapor density monitoring system using UV radiation generated from quasi-phasematched SHG waveguide devices

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ABSTRACT

Many industrial applications require non-intrusive diagnostics for process monitoring and control. One example is the physical vapor deposition of titanium alloys. In this paper we present a system based on laser absorption spectroscopy for monitoring titanium vapor. Appropriate transitions for monitoring high rate vaporization of titanium require extension of available IR diode technology to the UV. The heart of this vapor density monitoring system is the 390nm radiation generated from quasi-phase matched interactions within periodically poled waveguides. In this paper, key system components of a UV laser absorption spectroscopy based system specific for titanium density monitoring are described. Analysis is presented showing the minimum power levels necessary from the ultraviolet laser source. Performance data for prototype systems using second harmonic generation (SHG) waveguide technology is presented. Application of this technology to other alloy density monitoring systems is discussed.

KEYWORDS

Second harmonic generation waveguides, laser absorption spectroscopy, periodically poled waveguides, density monitoring, UV radiation, process monitoring, process control, physical vapor deposition.

1. INTRODUCTION

Laser absorption spectroscopy (LAS) continues to be a useful tool for monitoring a variety of physical vapor deposition processes. LLNL has demonstrated such a system for the vaporization of a uranium-iron alloy[1,2], and is now developing similar systems for process monitoring and control for the evaporation of titanium alloys. These alloys are of particular interest to the aircraft manufacturing industry. In particular, the use of titanium alloys in metal matrix composites offer the potential of dramatically increased performance and economy in aircraft engines[3]. This performance increase is due to their superior high temperature material properties and a lighter weight in comparison with nickel-based superalloys. One particular intermetallic composite, Ti_2AlNb, has the advantages of high specific strength, low temperature ductility and low temperature fracture toughness as compared to other alloys.

Electron beam physical vapor deposition (EB-PVD) is the method of choice for high rate deposition processes[4]. Composition control of Ti_2AlNb evaporation is difficult due to the vast differences in vapor pressures of the various constituents. Laser

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absorption spectroscopy is an ideal technique for accurately measuring the absolute vapor composition and providing a corresponding feedback signal to correct for composition fluctuations during the physical vapor deposition process.

A key component in an absorption spectroscopy based density monitoring system is the light source, and presently, the majority of laser systems used for spectroscopy are based on argon-ion ring dye lasers. Ring dye laser systems are very flexible in terms of tunability and output power, but their cost, size, and maintainability makes them impractical for most industrial settings.

The feasible transfer of LAS technology from a laboratory setting to an industrial environment requires the replacement of the argon-ion pumped ring dye laser by a laser diode. The diode laser must be compact, single longitudinal mode, and scannable. Distributed feedback (DFB) and distributed Bragg reflection (DBR) laser diodes meet the aforementioned requirements, but the number of transitions concurrent with commercially available DFB/DBR lasers is very small. Single mode tunable external cavity diode lasers (ECDLs) are now commercially available at various wavelengths throughout the IR. Although more expensive than DFB/DBR lasers, their narrow linewidth, tunability, compact size, and low power requirements, make the ECDL an appropriate choice for laser absorption spectroscopy applications in industrial settings. However, monitoring of many metal vapors (i.e. Ti, Al, Nb) requires extension of the presently available IR diode technology into the blue to ultraviolet (UV) wavelength regime. Although extension of this wavelength range is being developed through several approaches, a compact, scannable, single mode, low cost system is not currently available as a commercial product.

The objective behind the work described in this paper is the development of a compact, low cost, diode-laser based system for accurate monitoring and control of titanium metal vapor deposition processes, and the extensions of that technology to other metal alloy deposition applications.

2. LASER ABSORPTION SPECTROSCOPY

Laser absorption spectroscopy has been used to monitor electron beam generated vapor plumes at Lawrence Livermore National Laboratory for more than fifteen years. LAS has proven itself to be an accurate and reliable means to monitor both density and composition. During this time, the diagnostic has moved from a research tool to being a robust component in a process control system.

In LAS, the light absorbed by the vapor is a measure of the vapor density. The light is scanned in wavelength through an atomic transition. By scanning the laser wavelength slightly wider than the actual transition, a zero baseline is established, thus providing an absolute calibration for each scan. LAS is also very attractive for composition monitoring and control as it is highly selective. Interference from other elements is very unlikely due to the narrow laser linewidth, as well as to the large separation of atomic transitions as compared to the transition linewidth.

Figure 1 illustrates the key components in an industrial LAS density monitoring system. As mentioned previously, the laser diode makes the system compact, reliable and economical. The acousto-optic (AO) modulator and lock-in-amplifier (LIA) combination allow for synchronous detection for increased signal to noise, as well as the ability to monitor multiple species simultaneously. Synchronous detection is especially critical for monitoring electron beam vaporizer systems as the background light from an electron beam vaporizer is more than an order of magnitude higher power than typical laser powers in the vapor. A reference detector located just prior to the vessel entrance provides two key functions. First, although the light incident on the reference detector never passes through the actual vapor, it provides a means to ratio out any laser amplitude or light delivery fluctuations which would have been interpreted as density fluctuations. Second, and more importantly, the ratio of signal to the reference provides a measure of the transmission as a function of frequency. The computer takes the outputs of the signal and reference LIAs and calculates state density using an algorithm based on Beer's Law, $T=e^{-n\sigma l}$, where T is the transmission, n is the state density, σ is the cross section for the atomic transition being monitored, and l is the path length. The total density is then calculated based on an assumed (or measured) internal temperature. A voltage proportional to the total density (or vapor rate) is then provided in analog form to a vaporizer control system where parameters such as feed rate and electron beam current are adjusted to maintain the vapor rate setpoint of interest[2].

3. SYSTEM GOALS

In order to design a titanium density monitoring system, the appropriate atomic state transition or transitions must be chosen. In general, this depends on the anticipated vapor density, the vapor distribution, the anticipated internal temperature range, and the available diode technology. The details of the methodology used to choose the appropriate transitions for monitoring metal vapors have been discussed by Berzins et al[5]. Based on this analysis, the following system goals for a titanium density monitoring system are defined below:

center wavelength (air) = 391.588nm
titanium state monitored = 170cm^{-1}
bandwidth = 10GHz
laser linewidth < 1MHz
signal to noise ratio (SNR) = 100
detection bandwidth = 1kHz

Once the system goals have been defined, the next step is to determine the minimum laser power for an operable system. In order to define the minimum laser power, the anticipated conversion efficiency of the second harmonic device, the power currently available from single mode IR diodes, and the transmission and performance of all components of the density monitoring system at 390nm must be evaluated. In the next section, we describe the key components of the density monitoring system. The performance of each component is discussed in terms of efficiency and transmission in order to define a minimum power level for the laser system.

4. SYSTEM DESCRIPTION

A UV laser system was developed using an external cavity diode laser (ECDL) and quasi-phasematched second harmonic generation waveguide, (see Fig. 2). The ECDL is based on a commercially available New Focus 6200 laser, but was modified for increased output power. Incorporation of a high power 785nm diode laser into the external cavity, combined with careful selection of the intracavity diffraction grating, resulted in a continuously tunable single longitudinal mode output at power levels exceeding 30mW. A miniature optical isolator based on a terbium gallium garnet (TGG) crystal was developed and provides the laser diode with 30dB of optical isolation. The SHG waveguide is mounted in a temperature controlled housing. This housing is inserted into a micropositioning stage with piezoelectrically actuated X and Y translation for accessing different waveguides, as well as for optimizing optical alignment. Mechanically integrated input and output coupling lenses were incorporated as part of the SHG assembly. We found that using separate stages for the input and output coupling lenses, as is commonly done in laboratory environments, results in unacceptable amplitude noise due to acoustic coupling. The UV and infrared radiation are separated using a dichroic beamsplitter, and the UV radiation is directed through an AO for amplitude modulation at frequencies of up to 100kHz. The achievable modulation index is between 0.5 and 1.0 depending on optical alignment, applied RF power, and drive waveform. The amplitude modulated UV light is then coupled into either a single mode or multimode fiber for delivery to the vacuum vessel. Note that just prior to the fiber coupler, a half-wave plate is used compensate for any rotation of the polarization through the fiber optic delivery system. The interface to the vacuum vessel consists of a sending unit located on the input vessel window. The sending unit, fabricated from 1/4" copper and mounted onto a standard 2 3/4" conflate vacuum window, houses all of the optics necessary to spatially format the beam and deliver it through the vessel. The 1/4" copper enclosure is critical for protection from the X-ray radiation exiting the vessel windows. Within the sending unit is a fiber collimating lens, a polarizer, a beamsplitter, and a gas scattering cell, where a flow of argon prevents coating of the vacuum window. The receiving unit, located on far end of the vessel directly across from the sending unit, consists of an identical gas scattering cell, a lens, and a signal detector. Both the signal and reference detector outputs are delivered to the LIAs, whose reference frequency is set to that of the UV AO modulator (approx. 10KHz-25KHz). The outputs of the LIAs are sent to the A/D board of the computer system.

Several SHG waveguide devices have been tested for use within the density monitoring system. The $LiNbO_3$ periodically poled device was developed specifically for generating radiation at 391.5nm to monitor the Ti 170cm^{-1} transition. This device was fabricated in a two step process[6]. The first step uses a Ti indiffused mask to create the ferro-electric domain grating of period 2.2μm on the surface of a $LiNbO_3$ wafer. The grating was diffused at 1100°C for 10 minutes into the $LiNbO_3$

substrate. The next step utilizes an annealed proton exchange (APE) process to create the channel waveguides, which are oriented parallel to the grating wavevector. Specifically, the APE waveguides were fabricated by a 10 minute proton exchange process in pure benzoic acid at 200°C through a 4μm channel in an SiO_2 mask, followed by an anneal time of 2 hours at 330°C. The waveguide structure which phase-matched closest to room temperature is approximately 4.0μm wide, 2μm deep, and 1 cm in length with a QPM period of 2.2μm. The overall dimensions of the SHG chip are 5.0mm wide, 1cm in length, and 1mm thick. Approximately 100 waveguides transverse the chip.

Another device tested within our system was fabricated in KTP for generation of 385nm radiation. The KTP device tested has a segmented waveguide structure. This structure is formed by applying a metal mask with the appropriate segmented waveguide pattern to the KTP substrate. The waveguides are then formed by immersing the masked KTP in a 320°C Rb and Ba ion-exchange bath for approximately 45 min[7]. The resulting KTP waveguides are 4μm wide by 4μm deep with a QPM length and period of 10mm and 2.825μm, respectively. The overall dimensions of the KTP chip are approximately 2mm wide, 10mm in length, and 1mm thick. Approximately 30 waveguides tranverse this chip.

Both KTP and $LiNbO_3$ have advantages for use as SHG waveguide devices. Lithium niobate is less expensive, easier to grow in large uniform wafers, and an extensive experience base has been built around this material. KTP, a relatively new material by comparison, has a larger second harmonic figure of merit, superior damage threshold properties, and the periodic domain regions in KTP can be fabricated more uniformly, resulting in higher conversion efficiencies[7]. Results regarding the performance of these devices are discussed in the next section.

5. SYSTEM PERFORMANCE

Specifying the appropriate laser power levels required for a density monitoring system starts with a complete system loss and noise analysis. Many noise sources can exist with an e-beam vaporization system, and the noise sources specific to a particular vaporizer must be quantified and mitigated. We will start by assuming the noise in the system is dominated by detector noise. The first step is to determine the minimum power requirements incident on the detector. Our initial specifications are based on the use of a UDT455UV photodiode packaged in a custom LLNL trans-impedance amplifier configuration. The detector is operating in the photoconductive mode with a reverse bias of -15V in order to achieve a 3dB bandwidth of 25kHz. The trans-impedance amplifier stage provides a gain of 10^6. An analysis of the noise equivalent power (NEP) of the detector configuration at 390nm reveals an estimated NEP of $1x10^{-12}$ W/root Hz. At a detection bandwidth of 1kHz, this results in an NEP of 32pW. To obtain a system SNR of 100 requires 3.2nW incident on the detector. The amount of absorption through the vapor must also be taken into account. At 10% transmission, the theoretical minimum required power must be increased to 3.2nW/0.10=32nW (-45dBm) in order to compensate for the absorption of the signal through the vapor.

Now consider the losses of the optical transport system from the output of the SHG device to the signal detector see Figure 2. Initially, there were four major contributors to the system loss; the UV AO modulator, the optical fiber transport system, the loss through the input and output windows within the vessel, and the loss through the sending and receiving units. The UV AO modulator should diffract 80% of the beam into the first order at a carrier drive frequency of 75MHz. Assuming 100% modulation in the 1st order with a modulation frequency of 20kHz, the average power is reduced by a factor of 2. The device itself also has an insertion loss of 0.2dB, resulting in an overall loss through the modulator of 4.2dB. In regards to the fiber transport system losses, experimental results show that with a TM_{00} mode exiting a waveguide, we can routinely couple greater than 40% of the beam into the fiber. This is very dependent, however, upon the spatial output of the SHG device. With the higher order spatial mode output of the $LiNbO_3$ device, only several percent coupling into a single mode fiber was achieved. Ideally, transporting the UV radiation to the vaporization chamber through single mode fiber is desirable, as a much smaller diameter, collimated beam can be sent through the vapor. Multi-mode (MM) fiber has the advantage of increased coupling efficiency of higher spatial order beams, but the larger beam size and divergence angle at the collimated output can become an issue in propagating the beam through the longer path lengths of larger vaporization chambers. Due to the higher order spatial output of the $LiNbO_3$ waveguide, we chose to utilize a 50μm MM fiber for our prototype demonstration in order to increase the coupling into the fiber to approximately 70%. Further tests will be done to determine if the collimated output of a multi-mode (as opposed to a single mode) fiber, incident on the signal and reference detectors will add any substantial noise to the system. In general though, a single mode delivery system is preferable, and often times required for vaporization chambers with long path lengths. Although enhanced for the blue-UV wavelength range, the attenuation of the fiber (Dyeguide 50mm core) at 390nm is also to our disadvantage at 80dB/km. A survey of various fiber vendors showed that this was typical. Even over short lengths of fiber (approx. 30m), this adds a significant contribution to system losses, (2.4dB). The total loss anticipated for a density monitoring system where the single mode fiber length is approximately 30m is 6.4dB. The third and most significant loss was due to the poor transmission (3%/window) of the UV radiation through the leaded glass viewports on the vessel. These viewports were an essential part of previous density monitoring systems at LLNL to insure sufficient protection against x-ray exposure. Most of the monitoring systems previously deployed at LLNL were ring-dye laser based systems, where power was generally not an issue, and/or were at wavelengths >420nm where the transmission through the leaded glass increases significantly higher with increasing wavelength. This 30dB loss, however, was eliminated by the replacement of the leaded glass windows with AR coated fused silica windows, and x-ray protection was then incorporated into the walls of the sending and receiving units. Lastly, the optics within sending and receiving units add another 2.5dB of loss. With a well engineered system, the loss from the remaining optics, if optimized for transmission at 390nm should add less than 0.5dB. Thus the total loss through an optimized system is 13.4dB.

Using this result, we can now work backward to define a lower bound to the required second harmonic power for a system limited by detector noise.

$$P_{SHG} = -45.0\text{dBm} + 13.6\text{dB} = -31.4\text{dBm} = 0.72\mu\text{W}$$

As mentioned previously, various noise sources can exist within an e-beam vaporizer system. The configuration of the viewports with respect to the vapor source, as well as the electronic configuration can add significant noise. In practice, with the current system design, it is difficult, but possible to make density measurements with approximately 5μW of UV power. Clearly, an industrial system will require many times this power level for reliable performance.

Now that the minimum second harmonic power is defined, the normalized conversion efficiency of guided wave QPM SHG devices can be investigated. In the literature, devices with normalized conversion efficiencies of up to 600%/W-cm^2 have been reported for SHG at 480nm and at 425nm[6,7]. Unfortunately, no literature was found reporting conversion efficiencies for devices fabricated for second harmonic wavelengths less than 400nm. As will be seen below, the efficiency and stability of the SHG device critically drives the achievable system performance.

Several devices have been tested as part of this density monitoring system. The LiNbO$_3$ device as described above and fabricated specifically for this project was reported to have an initial maximum internal conversion efficiency[5] of 80%/W-cm^2 at New Focus. This level of conversion efficiency, assuming >7.5mW of IR coupled into the waveguide, would produce >50μW of UV. Although this UV power level should be sufficient for our density monitoring system, both the IR throughput and UV power drop precipitously within seconds after exposure to the IR light. Figure 3 shows the normalized conversion efficiency versus time for a waveguide first being exposed to IR radiation. Although the maximum conversion efficiency of this particular waveguide was much lower in comparison with other waveguides on the same chip, the decreasing IR and UV power trend is typical of this sample. A photo-chromatic effect is believed to be the culprit and new processing techniques are being investigated to better understand and correct the problem. The highest normalized conversion efficiency of this sample measured at LLNL, 36%/W-cm^2, was obtained with 25mW of IR incident on the chip, 6.5mW of IR coupled through the chip and 15μW of UV radiation exiting the waveguide. This conversion efficiency, as well as the IR throughput, would then decrease within several minutes to a typical efficiency of 20%/W-cm^2. Although this device outputs >5μW for periods of days, a commercial system where the radiation is fiber optically delivered to the vaporization chamber is desirable. Further optimization of the LiNbO$_3$ SHG waveguide fabrication process is currently being pursued to acquire and maintain normalized conversion efficiencies of >80%/W-cm^2.

A KTP SHG device not fabricated specifically for this project was also tested in this identical system, but at a slightly shorter wavelength. The nominal operating SHG wavelength of this device at room temperature was 387.5nm. In order to reach another titanium 170cm^{-1} transition, the device was heated to 48°C in order to temperature tune the second harmonic to 388.1nm. In a test performed at Dupont Experimental Station, this device, while being pumped by a Ti:Sapphire laser, measured normalized conversion efficiencies of over 80%/W-cm^2 for over 5.5 hours with no observable signs of degradation, see Figure 4. This device was measured at LLNL to produce > 40μW of UV light in a TM$_{00}$ mode with 7.5mW of IR coupled through the waveguide. The slightly lower conversion efficiency (>70%/W-cm^2) is most likely due to the particular spatial characteristics of the IR beam and how it couples into the SHG mode of the waveguide. This device was also tested in the above described diode-based system at LLNL with conversion efficiencies of >50%/W-cm^2. Here again, the spatial mode of the IR beam is very different than that of a Ti:Sapphire laser. The original diode laser system was designed for optimal coupling into the LiNbO$_3$ waveguide. The conversion efficiency of the KTP second harmonic device would be improved by optimizing the IR/SHG mode-matching into the 4μm x 4μm KTP waveguide. Further fabrication and testing of segmented KTP SHG devices for use in density monitoring systems is being conducted.

Based on a nominal 80%/W-cm^2 normalized conversion efficiency, 90% transmission through the delivery optics and optical isolator, and 25% IR coupling into the waveguide, an output power of 20mW from an ECLD at 785nm is sufficient to meet our minimum second harmonic power requirements, provided the UV output is in a single spatial mode. More power may be required to handle higher spatial modes. Our goal for a reliable density monitoring system is to have devices fabricated specifically for 391.5nm that exhibit normalized conversions efficiencies of greater than 80%/W-cm^2 for 1000's of hours, as well as to optimize the optical and detection system.

In order to test system performance of the integrated components, several short duration experiments were conducted. These experiments utilized in a small, non production vaporizer that operates at much lower densities. For this reason, alternate transitions were chosen and the SHG devices were temperature tuned to these wavelengths. This allowed us to single pass the laser through the small chamber and still obtain a reasonable absorption signal with the greatly reduced vaporization rate. Although the beam was not transported to the chamber via optical fiber, all other aspects of the system were the same as described above. With approximately 5μW UV radiation exiting the waveguide, absorption waveforms of 25% were obtained and densities on the order of 10^{11} were measured throughout the duration of the 20 minute experiment. Figures 5 and 6 show a typical absorption waveform and the measured density in the chamber versus time.

6. ALTERNATE APPLICATIONS

A diode based ultraviolet laser source provides many new opportunities for laser sensing. The 390nm radiation was chosen as an excellent starting point because of the availability of high power fundamental diode lasers at 780nm, and the large number of other atomic transitions within that same UV wavelength range. For the metal sensing application alone, transitions exist for aluminum, niobium, cobalt, hafnium, tantalum, vanadium and zirconium that are potentially accessible with the existing New Focus doubled diode system. Several of these are important constituents in high performance aircraft alloys. The waveguide douber technology is also easily adapted to wavelengths between 380nm and 500nm. In addition to the aforementioned metals, there are also a variety of other metals, such as barium, indium, yttrium, potassium, etc., whose densities could be monitored with doubled diode radiation within the 380nm-500nm wavelength range. Development of this technology, together with commercially available fundamental diodes, provides nearly continuous wavelength coverage from 380nm to 1000nm, and potentially beyond. A specific example of interest is the monitoring of niobium during the production of certain titanium alloys. In these applications, the niobium ground state population can be monitored with radiation at 425nm. DuPont has conducted extensive research in devices fabricated at this wavelength, and SHG devices which produce stable output powers of several milliwatts have been demonstrated. DuPont Central Research and Development provided us one of these waveguide chips so that we could demonstrate it's use for the monitoring of niobium density in a small vaporization chamber. Figure 7 shows the absorption waveform for this particular niobium ground state transition utilizing the segmented KTP waveguide chip to generate the required UV radiation.

7. SUMMARY

LLNL has been utilizing laser absorption spectroscopy for process monitoring and control for over 15 years. We have adapted and characterized a prototype system for the monitoring of titanium alloy physical vapor deposition processes using UV radiation generated from waveguide quasi-phasematched SHG devices. A theoretical system noise and loss analysis demonstrates the relatively low level of UV (<1μW) radiation required for an optimized, detector noise limited system. In a prototype system which utilized a multi-node fiber for light delivery to the chamber, measurements were made with UV power levels as low as 5μW. Realistically, the minimum power level should be increased to insure a robust and reliable system in an industrial setting. Both KTP and LiNbO$_3$ were evaluated for short term performance in a prototypic laser absorption diagnostic. Each offers advantages, as described in Section 4. Since at this point, it is not clear which option is optimal, evaluation of both will be pursued for potential future density monitoring systems. This titanium monitoring system is planned for deployment at a prototypic production facility in early 1996. Future work relating to this system will include analysis and mitigation of system specific noise sources, as well as measurement and evaluation of long term overall system performance.

8. ACKNOWLEDGMENTS

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Figure 1 - Basic LAS System
Figure 2 - Ti Density Monitoring System
Figure 3 - Normalized conversion efficiency of $LiNbO_3$ at 391.5nm
Figure 4 - Normalized conversion efficiency of KTP at 388.1nm
Figure 5 - Ti 392.1nm absorption waveform using radiation from $LiNbO_3$ device
Figure 6 - Density measurements using Ti 392.1nm using radiation from $LiNbO_3$ device
Figure 7 - Nb ground state absorption waveform using radiation from KTP device

Vapor density is calculated from the absorption of laser light by the vapor

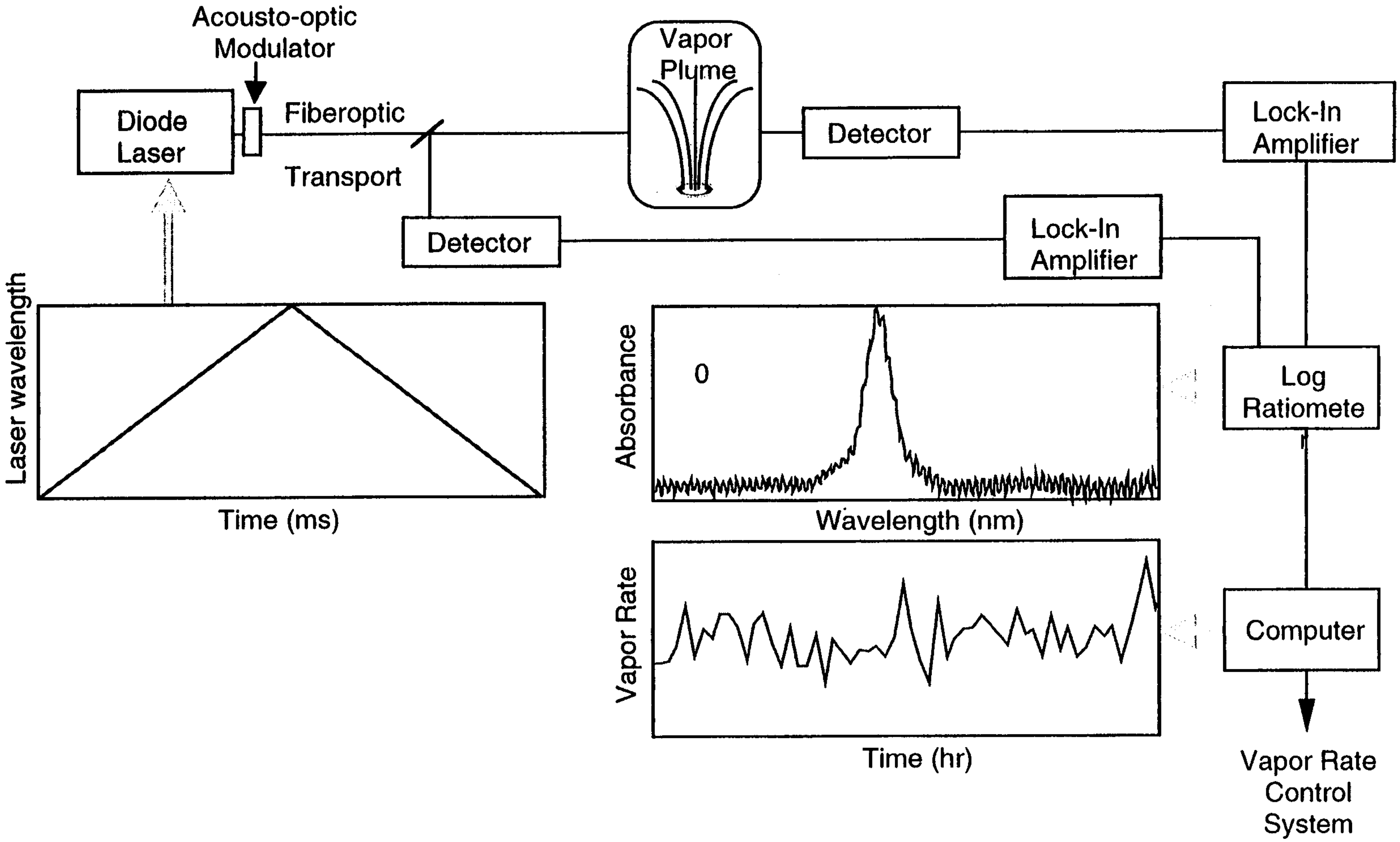
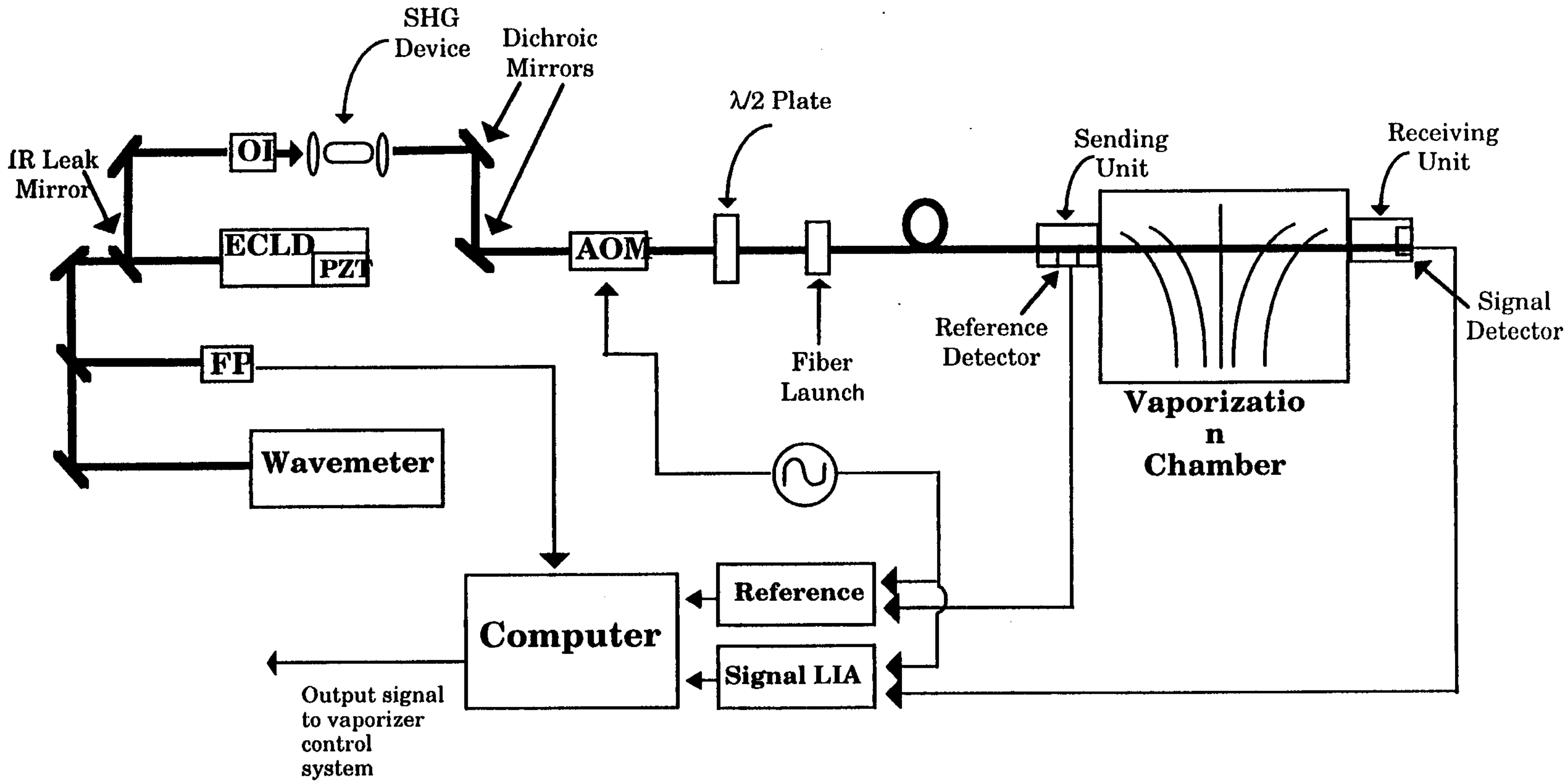


Figure 1



Titanium density monitoring system.
OI, optical isolator; AOM, acousto-optic modulator;
PZT, piezoelectric; LIA, lock-in-amplifier

Figure 2

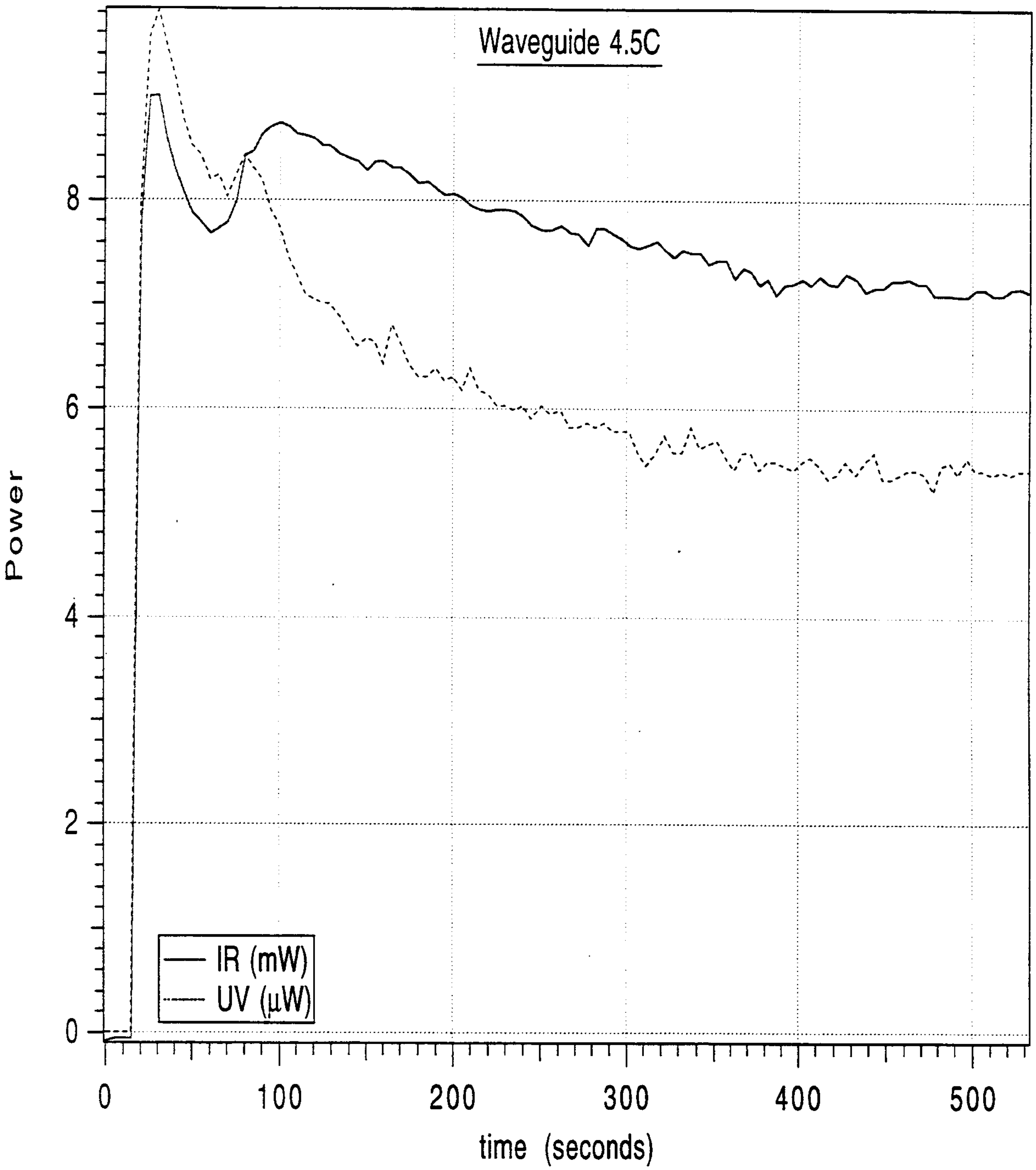

Waveguide 4.5C
Power
0
2
4
6
8
0
100
200
300
400
500
time (seconds)
IR (mW)
UV (µW)

Figure 3

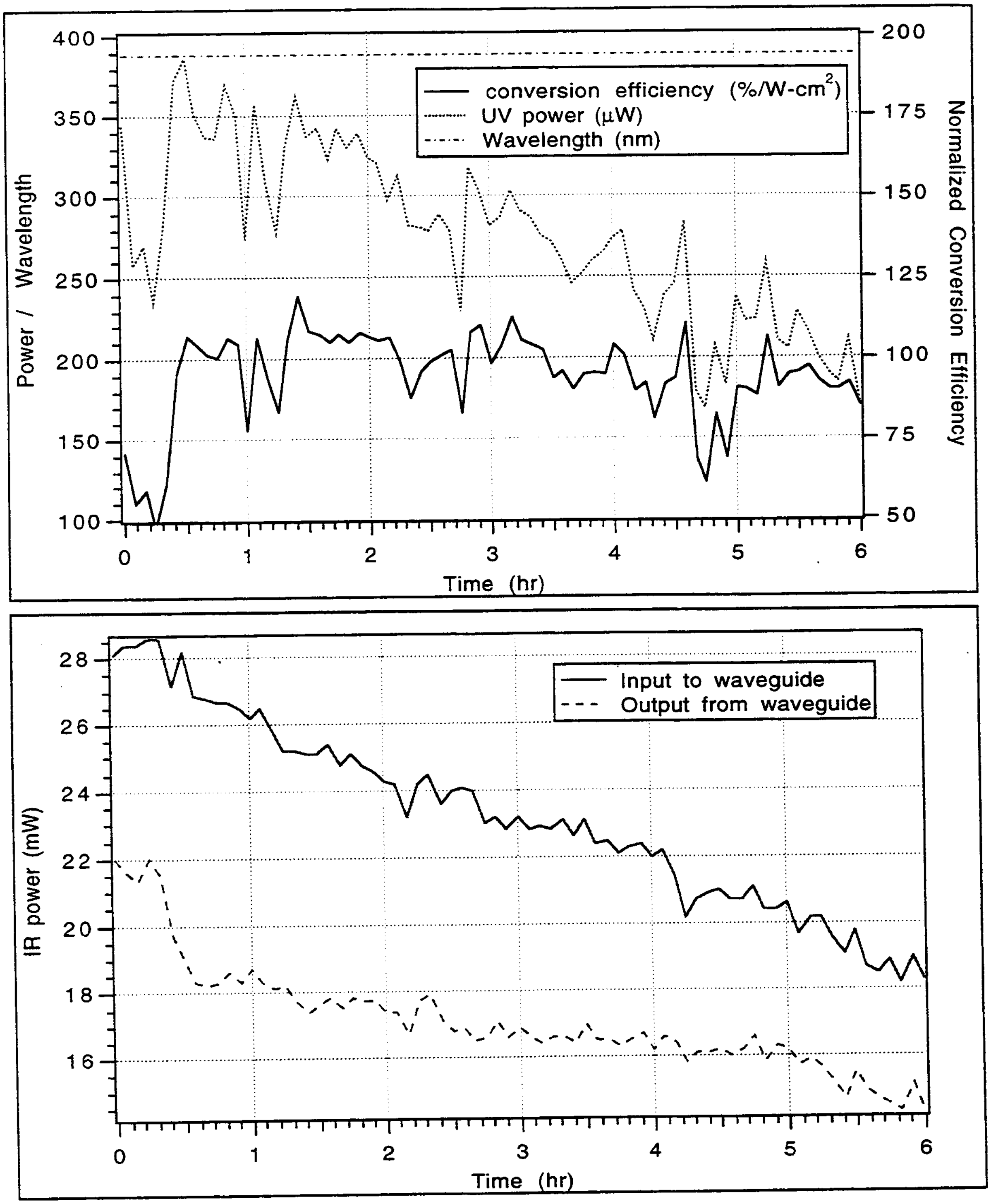
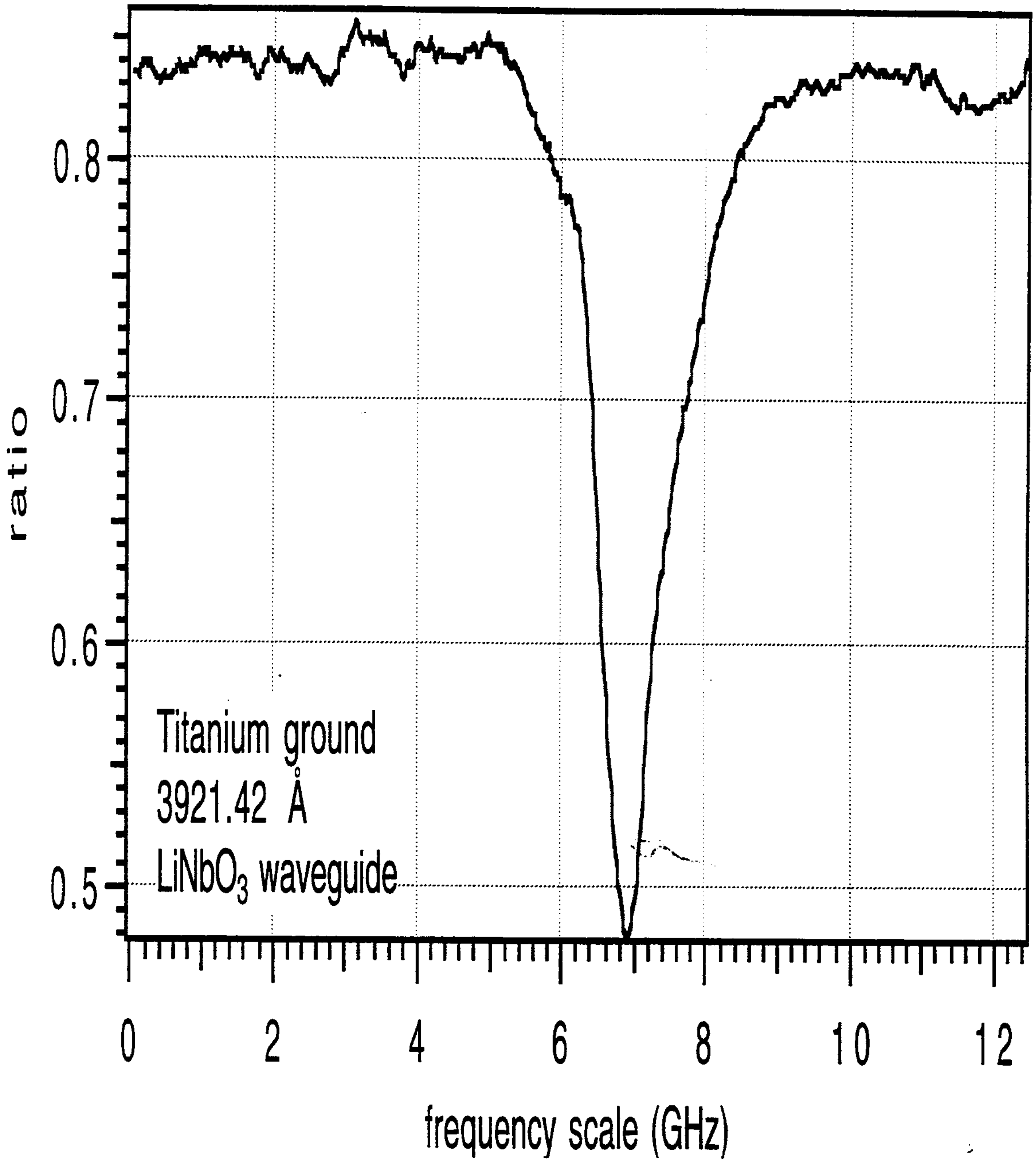

conversion efficiency (%/W-cm^2)
UV power (μW)
Wavelength (nm)
Power / Wavelength
Normalized Conversion Efficiency
400
350
300
250
200
150
100
200
175
150
125
100
75
50
0
1
2
3
4
5
6
Time (hr)
Input to waveguide
Output from waveguide
IR power (mW)
28
26
24
22
20
18
16
0
1
2
3
4
5
6
Time (hr)

Figure 4



ratio
0.8
0.7
0.6
0.5
Titanium ground
3921.42 Å
LiNbO$_3$ waveguide
0
2
4
6
8
10
12
frequency scale (GHz)

Figure 5

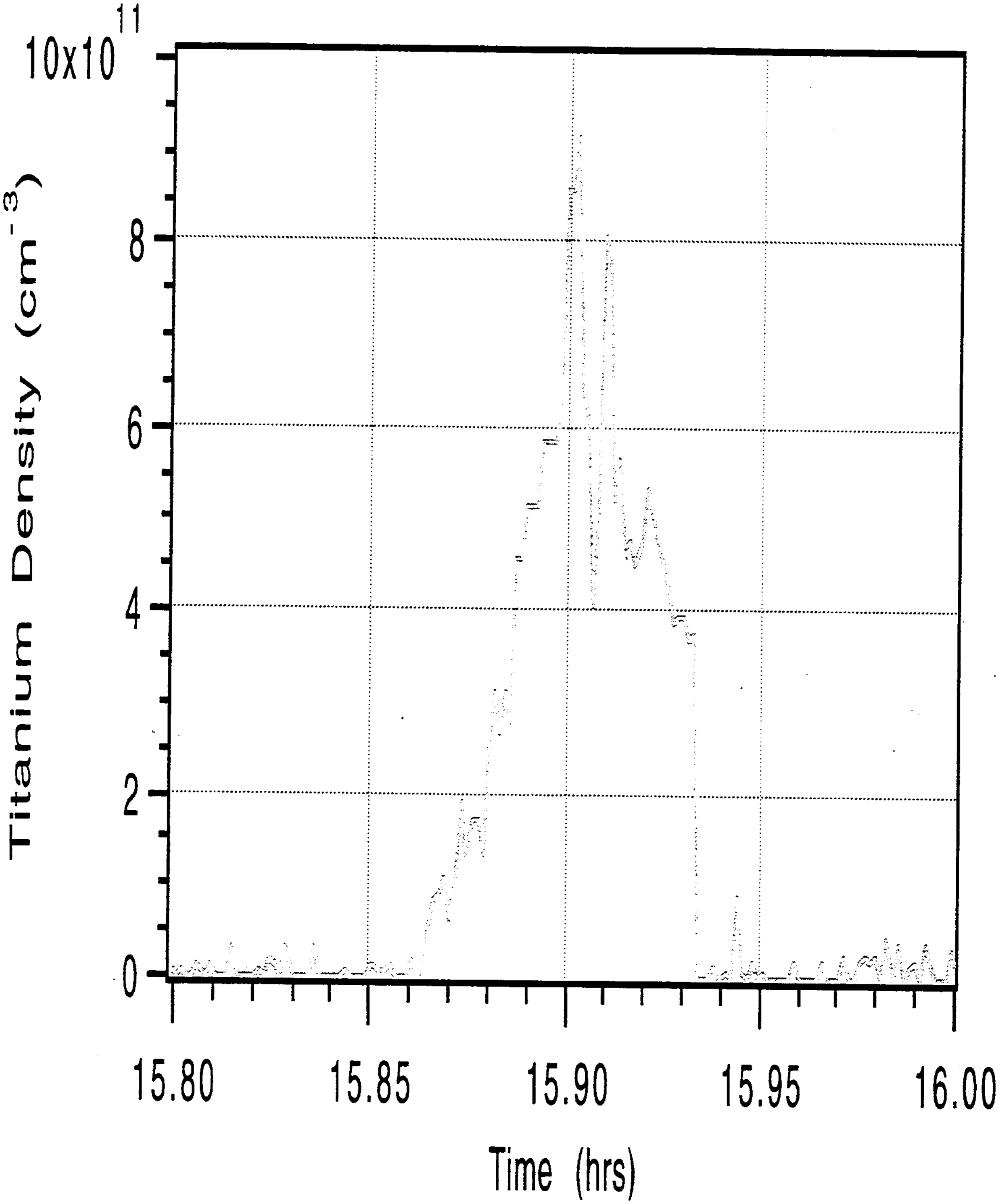

10×10^{11}
8
6
4
2
0
Titanium Density (cm^{-3})
15.80
15.85
15.90
15.95
16.00
Time (hrs)

Figure 6

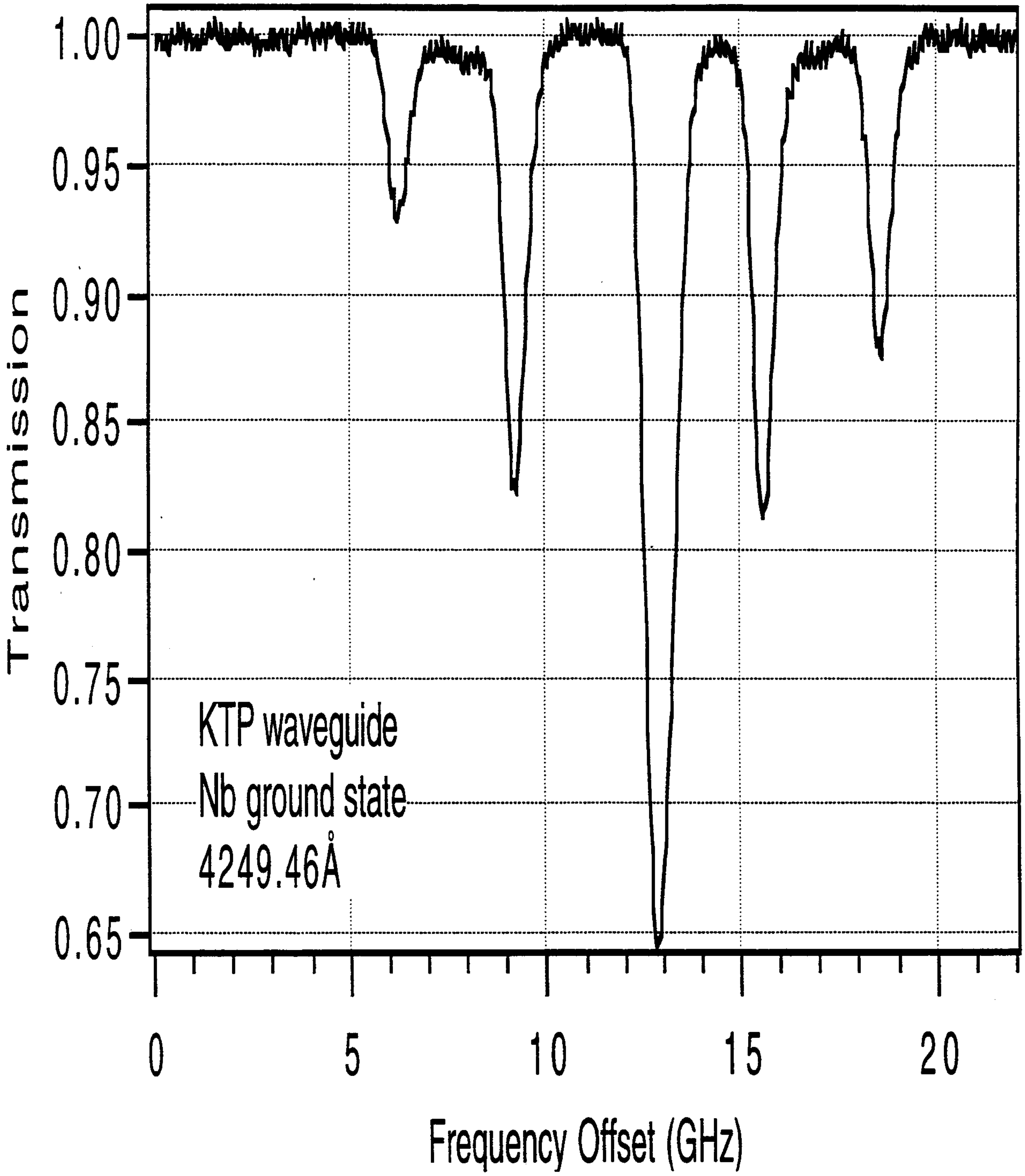

1.00
0.95
0.90
0.85
0.80
0.75
0.70
0.65
Transmission
KTP waveguide
Nb ground state
4249.46Å
0
5
10
15
20
Frequency Offset (GHz)

Figure 7