NASA-CR-205675

OPTICAL SENSORS BASED ON SINGLE ARM THIN FILM WAVEGUIDE INTERFEROMETER

Performance Report

1-st Year of Three-year Grant

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Period: August 1, 1996 to October 31, 1997

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B. Research Faculty

Dr. Martha Reddy (4 months, 75% time)

C. Post Graduates

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D. Graduate students involved

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D. Undegraduate students

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1. Goals and objectives established for the first year

The goals and objectives of the project in the initial proposal have been formulated as follows:

1-st Year (1997-1998)

1. Material selection. Materials for the waveguide interferometer will be selected from high temperature polyimide resins doped with thermally stable organic compounds. For specific extra high temperature applications, materials will be selected from the glasses prepared by sol-gel technique and/or possibly from ion-implanted monocrystals.

2. Thin film waveguide fabrication. Fabrication technique will include spin coating, UV curing, and/or ion implantation.

3. Thin film waveguide characterization. This phase of research will include modal spectroscopy based on prism coupling, propagation loss measurement with experimental set up based on imaging with digital CCD camera.

4. Experiments with novel single-arm dual-mode interferometry set up. Optical thin film slab waveguides will be studied with optical set up similar to that described in the proposal (Fig. 1 of the proposal). Testing the interferometer as a temperature and pressure sensor for the gaseous combustion products of aeropropulsion systems will be conducted. The gases to be tested will include CO, CO_2, NO, NO_2, and possibly others.

2-Nd Year (1998-1999)

1. Delineation of optical channel waveguides and their characterization.

2. Fabrication of optical coupling elements including gratings and inlets/outlets for optical fibers.

3. Testing the interferometer sensors in optical channel waveguide configuration.

3-Rd Year (1999-2000)

1. Development of purged gas chamber and precise heater for testing single-arm interferometric sensors.

2. Testing the waveguide coupled with optical fiber lines.

3. Estimations of the figures-of-merit of the devices. Conclusions on the feasibility of proposed sensors as elements of aeropropulsion control systems.

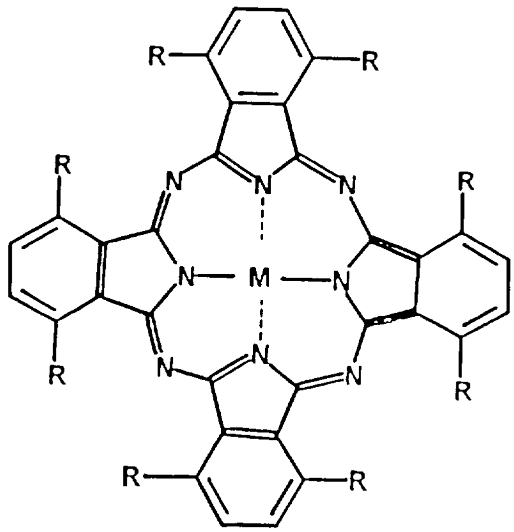
2. Actual accomplishments during the first year

2.1. Materials selection

The waveguide interferometric sensor is based on thin films of high temperature polyimide doped with metal substituted phtahlocyanines as indicator dyes. We have already shown that polyimide Ultradel 9020 from Amoco can be potentially used for planar waveguides delineated by direct writing with medium power UV source [1]. Metal substituted phthalocyanines are thermally stable organic compounds with distinctive optical absorption spectrum giving them deep blue or green color [2]. We performed preliminary study of a number of metal substituted phtahlocyanines (Fig.1) in order to investigate their potential use as indicator dyes. For example, 1,4,8,11,15,18,22,25-Octadecyloxy copper phthalocyanine changes significantly its optical absorption spectrum in a reversible manner after being exposed to various acid and alkali solutions. When several drops of acetic acid were added (3% acid concentration) to initially green solution of the phthalocyanine in ethanol, the color changed to brown. However, when several drops of this brown solution were added to acetone, the color changed back to green. This behavior is illustrated in Fig. 2. Experimental results are summarized in Table 1. Selected phthalocianines indeed perform as indicator dyes demonstrating change of optical absorption spectra after exposure to acid or alkali solutions. The same behavior accompanied by the refractive index change could be expected for phthalocyanines being exposed to NH_3, NO, NO_2, CO, CO_2, H_2S in the presence of water vapor.

2.2. Experimental gas chamber

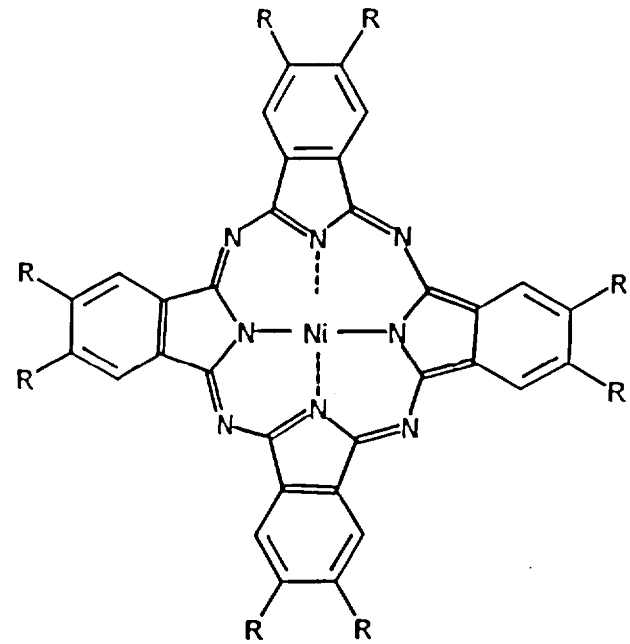
Experimental set-up for testing a single-arm dual-mode interferometric waveguide sensor is presented in Fig. 3. It includes gas chamber where the sensor is exposed to



M = Cu, Ni

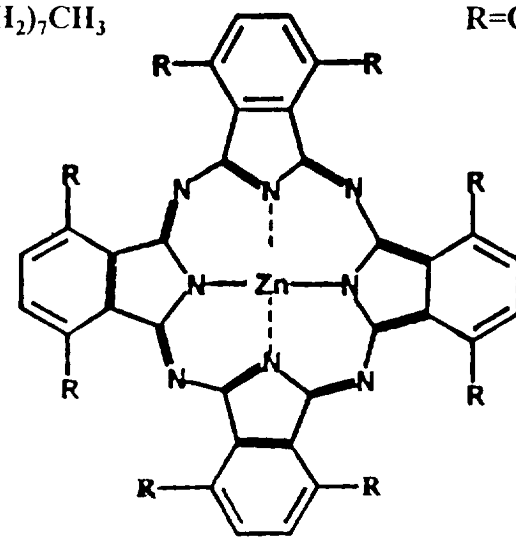
1,4,8,11,15,18,22,25-octaoctyloxy copper and nickel phthalocyanines

$R=O(CH_2)_7CH_3$



2,3,9,10,16,17,23,24-octadodecyloxy nickel phthalocyanine

$R=O(CH_2)_{11}CH_3$



$R = O(CH_2)_3CH_3$

1,4,8,11,15,18,22,25-octa-butoxy zinc phthalocyanine

Fig. 1. Metal substituted phthalocyanines which exhibit properties of indicator dyes to be used in waveguide sensor

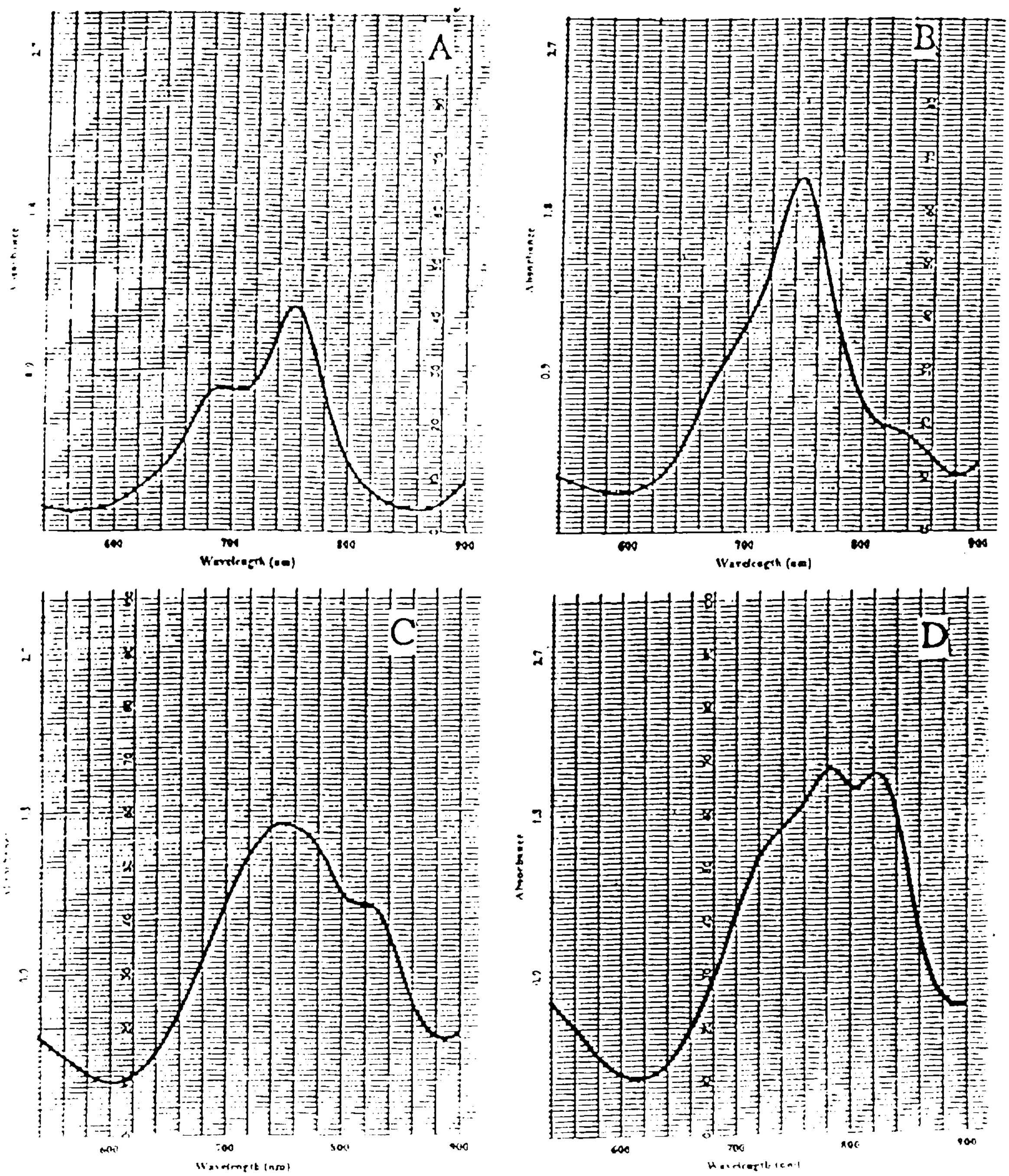


Fig. 2. Optical absorption spectra of 1,4,8,11,15,18,22,25-Octadecyloxy Copper Phthalocyanine in (a) ethanol solution (green color); (b) ethanol solution with 3% acetic acid added (brown color); (c) ethanol solution with 3% acetic acid deluted by acetone (green color); (d) ethanol solution with 1.8% chloroacetic acid added (brown color).

Table 1. Acid and solvent effects on phthalocyanines

Acid And Solvent Effects Of Phthalocyanines

Compound	Uncha.[f] In ACA[a]	Rev.[g] In ACA	Uncha.[h] In CACA[b]	Rev.[i] In CACA	Uncha.[j] In TCACA[c]	Rev.[k] In TCACA	Uncha[l] In CH[d]	Rev.[m] In CH	Uncha[n] In MEC[e]	Rev.[o] In MEC
1,4,8,11,15,18,22,25-Octabutoxy										
a) copper phthalocyanine	N[q]	Y[p]	N	Y	N	N	Y		Y	
b) zinc phthalocyanine	N	Y	N	Y	N	N	N	N	N	N
c) palladium phthalocyanine	Y		Y		N	Y	Y		N	Y
1,4,8,11,15,18,22,25-Octadecyloxy										
a) copper phthalocyanine	N	Y	N	Y	N	N	Y		Y	
b) zinc phthalocyanine	N	Y	N	Y	N	N	N	N	N	N
c) palladium phthalocyanine	Y		Y		N	Y	Y		N	Y

[a] ACA = Acetic acid
[b] CACA = Chloroacetic acid
[c] TCACA = Trichloroacetic acid
[d] CH = Chloroform
[e] MEC = Methylene chloride
[p] Y = Yes
[q] N = No

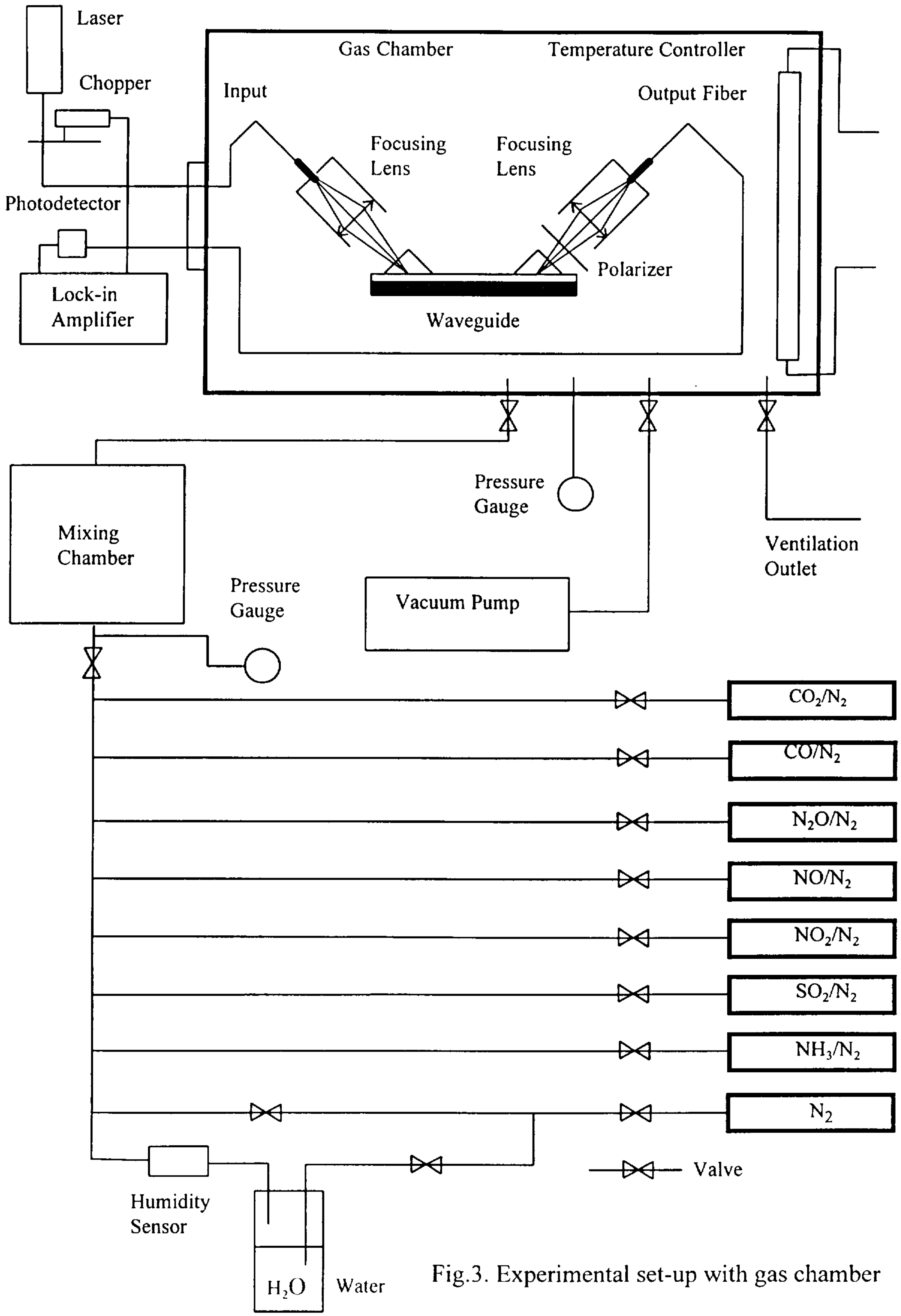
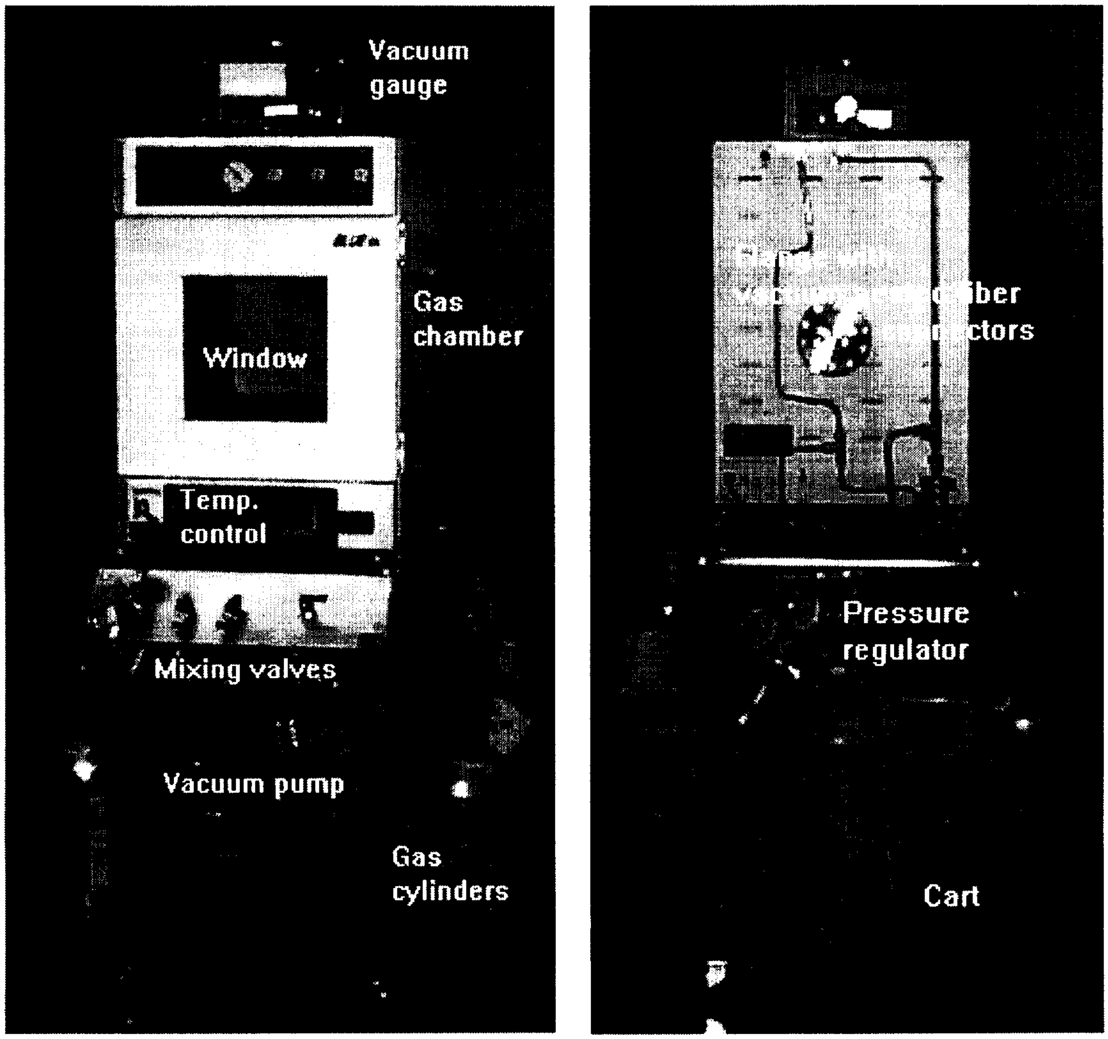


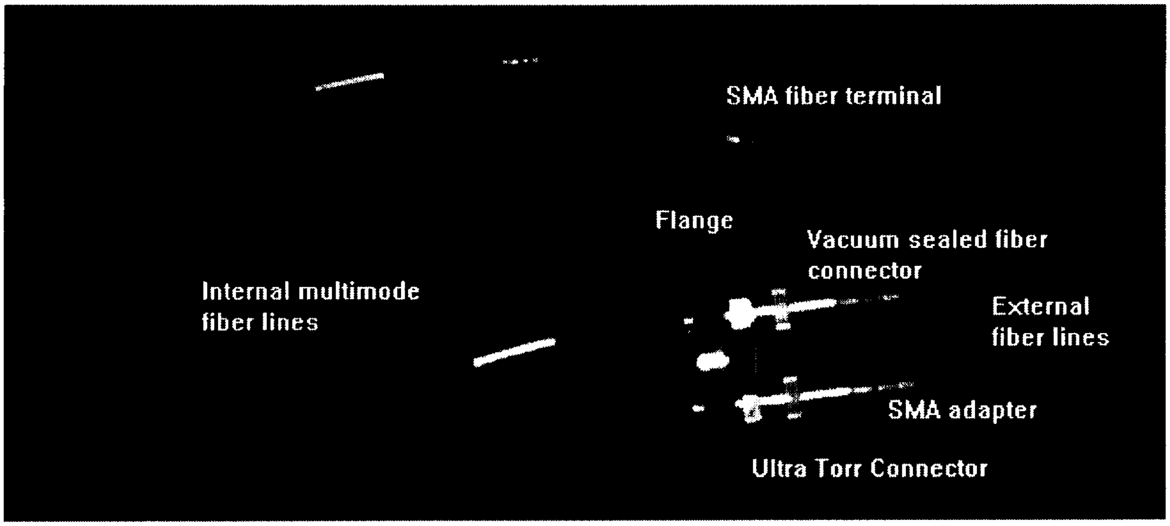
Fig.3. Experimental set-up with gas chamber

various gas mixtures. The gas supply unit includes a combination of gas cylinders and water bubbler connected through controlled pipe lines to a mixing chamber. The purpose of the mixing chamber is to prepare a mixture of different gases at a certain proportion before exposing the sensor to the mixture. This approach allows to investigate time response of the sensor to a particular gas mixture. The main gas chamber is equipped with an internal heater which provides the opportunity to characterize the performance of the sensor at elevated temperatures (up to 250^0C). The light from the external laser source (green He-Ne laser) is fed into the chamber through specially designed vacuum sealed multimode optical fiber connector. The multimode optical fiber line is coupled to the waveguide using focusing optics and a prism coupler. The same approach is used to outcouple the light from two propagating modes into the output multimode optical fiber line. The interference pattern is created by the light beams from the modes in the plane of the input terminal of the optical fiber line. The output light is fed through the second vacuum sealed optical fiber connector to a photo detector. Signal measurement is done using a chopper an lock-in amplifier. Fig. 4 and 5 show the view of the gas chamber and its parts. The vacuum chamber is mounted on a 4-wheel cart with a vacuum pump on the bottom and gas cylinders strapped to the sides and to the back. The mixing chamber (cylinder) is mounted below the gas chamber (is not seen). The pressure during the mixing and filling the gas chamber is measured by a vacuum gauge placed on the top of the chamber. Mixing gases with water vapor and filling the chamber is performed with four control valves on the front panel. Temperature control is performed with the electronic module on the front panel of the chamber. Feeding light in and out the chamber is done through a specially designed aluminum welded adapter (Fig. 4b and Fig. 5b) attached to the instrumental inlet on the back of the chamber. Vacuum sealed optical fiber connectors are inserted into the removable flange fixed to the adapter (Fig. 4a). The adapter prevents strong bending of the multimode optical fiber lines (with vacuum coating) coming to the breadboard with sensor set-up on it.

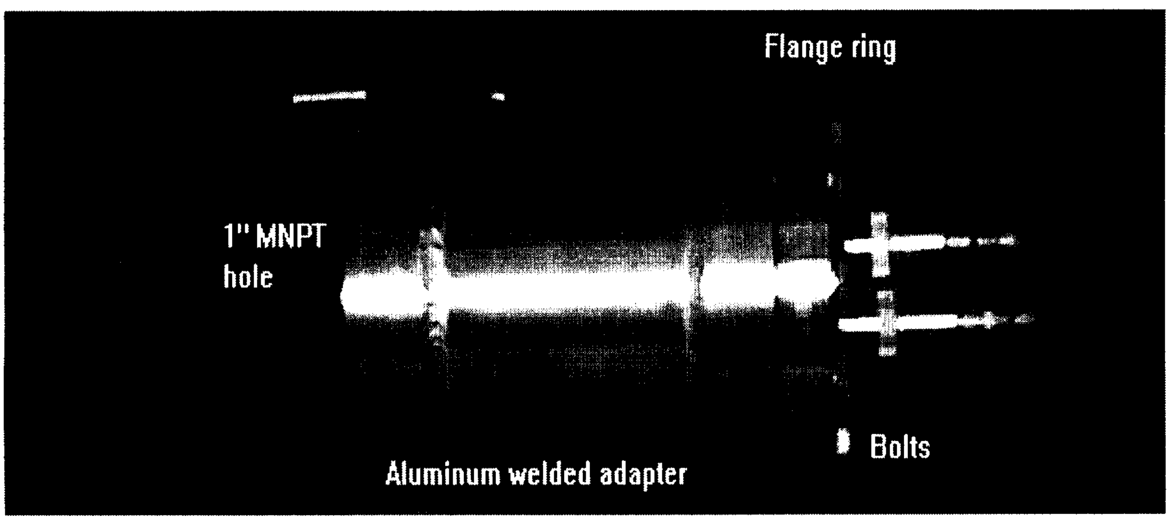


(a) (b)

Fig. 4 . Front (a) and back (b) view of the experimental gas chamber for studying waveguide interferometric sensor.



(a)



(b)

Fig. 5. General view of the vacuum sealed optical fiber lines used to connect the waveguide sensor to external light source and signal processing module: (a) vacuum sealed optical fiber connectors on the flange; (b) optical fiber connectors attached to the aluminum adapter which prevents critical bending of the optical fibers.

2.3. Preliminary results

We performed preliminary experiments with a planar waveguide made of polymer (particularly, PMMA) thin film. This experiments showed that the waveguide being exposed to atmospheric gases changes its refractive index. After annealing the refractive index returns to its previous level. Experimental data are presented in Fig. 6. In this case, a single mode slab waveguide made of PMMA doped with laser dye DCM is used. Annealing of the waveguide is done with UV radiation of a 500-W mercury arc lamp. The refractive index of the waveguide was measured with a prism coupler. The UV radiation bleaches the dye and gradually reduces the refractive index of the waveguide from 1.5025 down to 1.4995. At some instances the continuous exposure of the waveguide to UV radiation was interrupted and the waveguide was kept in open air for a certain period of time. This instances and time intervals are shown in Fig. 6 by arrows. The first 15 hours is the time elapsed between the moment when the waveguide was made and the moment of the first UV exposure. Keeping the waveguide in open air apparently increased its refractive index (increase is of the order of 0.001) while the heat generated by the UV radiation (even at doses less than 5 J/cm^2) was enough to remove the adsorbed gases from the waveguide and to return the index to the previous level. This preliminary experiment shows that reading the refractive index change in the waveguide using single-arm dual-mode interferometry could give the information about the presence of various gases in atmosphere. Quantitative study of the waveguide sensor will be accomplished when construction of the experimental gas chamber will be completed.

2.4. List of the accomplishments

The following accomplishments have been achieved during the first year:

1. Material selection. Metal substituted phtahlocyanines were selected and studied as potential indicator dyes that can be used in the waveguide sensor.

2. Design and construction of the experimental purged gas chamber. Experimental set-up includes additional mixing chamber which allows to expose the sensor to a certain

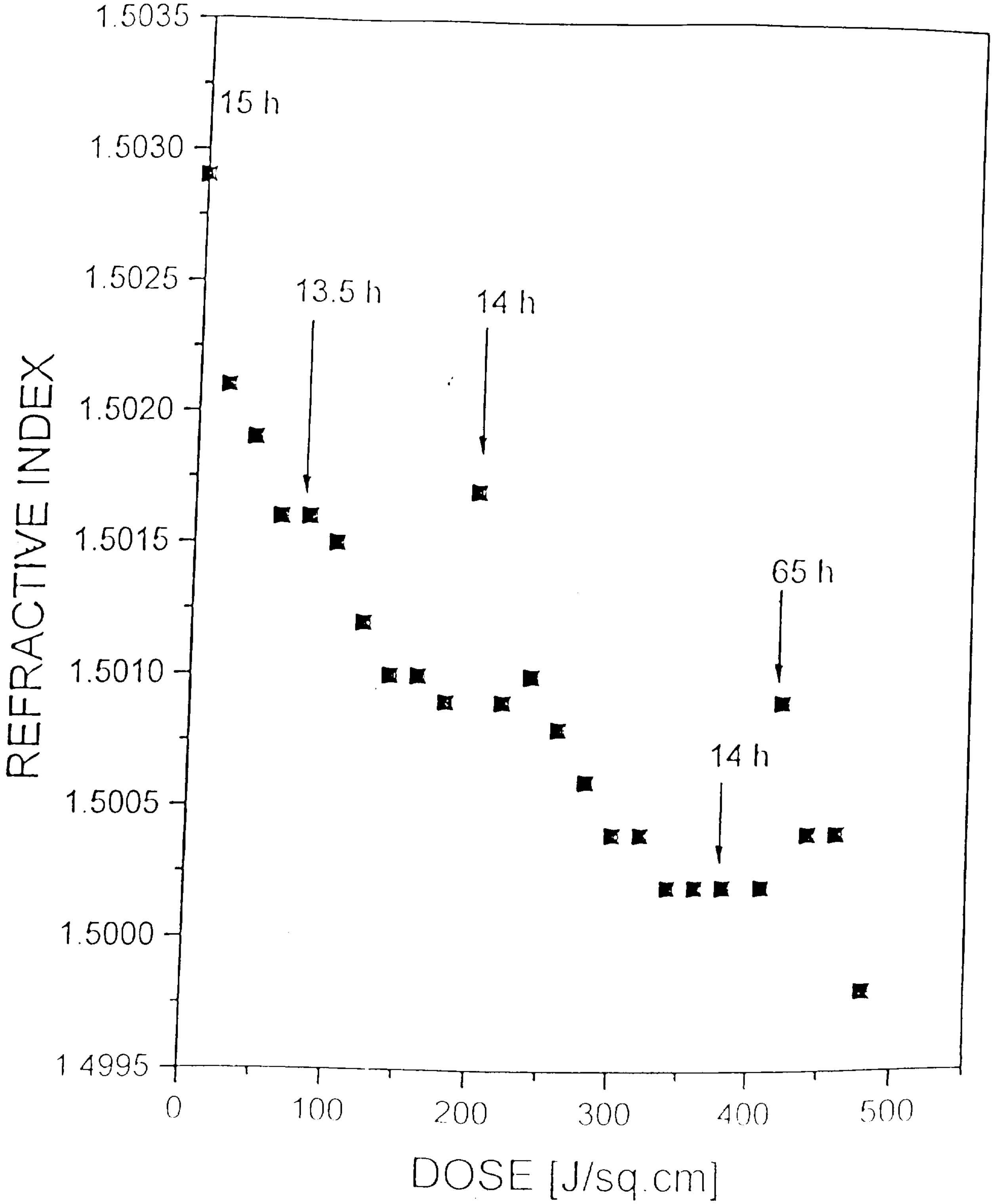


Fig. 6. Refractive index of a double mode DCM/PMMA waveguide on fused quartz substrate versus dose of UV exposure at 313 nm. Arrows show time intervals between exposures when the waveguide adsorbed ambient air.

gas mixture for a short period of time and therefore to characterize time response. It also includes vacuum sealed multimode fiber line which delivers light to and from the sensor inside the chamber to external signal processing equipment.

3. Preliminary study of the effects of gases adsorbed by polymeric waveguide on its refractive index. The obtained results showed that the refractive index is sensitive to the presence of the gases and that the single-arm dual-mode interferometry can be used in gas sensing. However, quantitative characterization of the interferometric waveguide sensor can be accomplished only after construction of the gas chamber.

3. Comparison of the accomplishments with the goals and objectives established for the first year

First accomplishment meets the first goal established for the first year. Goals 2 and 3 were met when the experimental planar waveguide structure was fabricated for preliminary experiments. The fabrication technique is spin coating. Characterization includes prism coupling to determine waveguide thickness and refractive index. Propagation losses will be determined using digital imaging of the trace of a propagating mode. Accomplishment 3 meets the goal 4 established for the first year. We did some preliminary experiments showing that the refractive index of the waveguide reacts to the presence of atmospheric gases.

Preliminary experiments also showed the importance of quantitative characterization of the sensor response to various gases such as CO_2, NH_3, NO, and NO_2. This characterization can be done with a purged gas chamber which was planned to be built during the third year. Taking into account all possible delays associated with the construction of the chamber, design and construction has been started at the firs year. 90% of this work has been already done. This the accomplishment 2 which meets the goal 1 for the third year. The construction will be completed by the end of 1997. During 1998, testing and characterization of the sensor will be done in the chamber which provides more realistic environment such as variable concentration of gases mixed at various proportions, presence of water vapor, and variable elevated (up to 250^0C).

4. Conclusions

All the goals of the research effort for the first year were met by the accomplishments. Additional efforts were done to speed up the process of development and construction of the experimental gas chamber which will be completed by the end of 1997. This chamber incorporates vacuum sealed multimode optical fiber lines which connect the sensor to the remote light source and signal processing equipment. This optical fiber line is a prototype of actual optical communication links connecting real sensors to a control unit within an aircraft or spacecraft.

Important problem which we are planning to focus during the second year is coupling of optical fiber line to the sensor (goal 2 for the second year). Currently this problem is solved using focusing optics and prism couplers. More reliable solutions are planned to be investigated.

4. References

1. S. Sarkisov, Z. Teague, P. Venkateswarlu, H. Abdeldayem, D. Frazier, and G. Adamovsky, Formation of graded-index waveguide in UV exposed polyimide, J. Appl. Phys., Vol. 81, No. 6 (1997), p. 2889-2891.

2. Phtahlocyanines: Properties and Applications, Editors: C.C. Leznoff and A.B.P. Lever, VCH Publishers , Inc., New York, 1989.

Appendix A. List of publications related to the Project

1. Sergey Sarkisov, Andre Taylor, and Putcha Venkateswarlu, Optical sensors based on single arm thin film waveguide interferometers, NASA Lewis Research Center HBCU Research Conference, Cleveland, Ohio, April 9-10, 1997, Abstracts, P.2.

2. Sergey Sarkisov, Andre Taylor, Putcha Venkateswarlu, and Grigory Adamovsky, Optical sensor based on single arm dual mode polymeric waveguide interferometer, Submitted to SPIE Meeting "Photonics West'98".