HASP 2008

Science Report on Payload # 7 of

University of North Dakota and University of North Florida

"Detection of Ozone Profile in Stratosphere Using ITO Gas Sensors on High Altitude Balloon"

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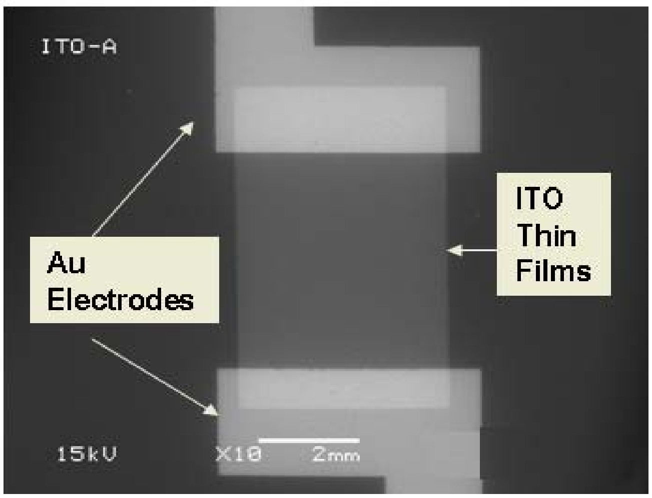
Introduction:

Osprey birds usually fly a few thousand feet high in the sky. UNF Osprey flew to a height of about 37000 meters during September 15 -16, 2008. UNF Osprey withstood severe cold temperatures down to – 56 ^oC, low pressures down to 10 mbar, plus vibrations and shock (10 g) during a flight of nearly 32 hours. UNF Osprey returned back to the ground successfully. UNF Osprey is an array of ozone gas sensors. With the support of High Altitude Student Platform (HASP) program of NASA, University of North Dakota (UND) and University of North Florida jointly collaborated on this scientific project on the detection of ozone gas in the stratosphere. HASP-NASA provided a platform for 12 payloads, 4 of which are large payloads and 8 which are small payloads. The maximum mass limit was 20 kg for a large payload, and 3 kg for a small payload. UND and UNF jointly have one small payload. The UNF responsibility was to provide an array of ozone gas sensors, while UND responsibility was to provide the micro controller circuit, software, and electronic communication circuits. The development of these ozone sensors are the byproduct of sensors developed for chemical agents and toxic chemicals, which are funded by the Department of Defense, US Army, Edgewood Chemical Biological Center, APG, MD.

HASP has onboard facilities of a computer, power supply batteries, GPS, video camera, and communication link for all payloads. UNF Osprey ozone sensors were tested and calibrated with a low concentration of ozone under low pressure at the NASA-Kennedy Space Center, UND and NASA-Columbia Scientific Balloon Facility (CSBF). During the first week of August 2008, the UND and UNF team met at the NASA-CSBF in Palestine, Texas for the integration of the ozone sensors and electronic circuits that made a complete UND-UNF payload. The payload was then fixed on to the platform of HASP. The NASA team conducted a series of tests on the UND-UNF payload for two days and gave green signal for flight. The UND-UNF payload successfully passed all the required tests, including high temperature and low temperature tolerance, low pressure and vacuum tolerance, power, computer link and data communications tests, and vibration and hang tests. Due to hurricane Gustav during the first week of September 2008, the date for launching of high altitude balloon was changed a few times and finally set for September 13. But due to the effects of another hurricane, Ike, the flight was scrubbed on September 13. Finally, the 400 foot high altitude balloon with the HASP payloads was launched successfully by NASA-CSBF on September 15, 2008 from Fort Sumner, New Mexico. The flight was originally planned for about 23 hours but was extended to about 32 hours due to favorable conditions. During the flight, the UNF Osprey ozone sensors array worked nicely and detected ozone in the stratosphere. The payload sent a data file every 7 minutes without any problems. The flight was terminated on September 16 at night. So the UNF Osprey ozone sensors detected the ozone profile of ozone layer from September 15 to 16, 2008. September 16 has been observed as the ***"International Day for the Preservation of the Ozone Layer"*** every year since 1995. That was the great coincidence. After the termination of the balloon flight, the payload landed safely on the ground using a parachute. The payload was recovered near the border of New Mexico and Texas. Some technical details, pictures and results of this flight are highlighted in this report.

Fabrication

Fig. 1 shows the top view of one typical ITO gas sensor having two gold electrodes for external elelctrical contacts. Fig. 2 shows the an array of 24 ITO gas sensors fabicated on one glass slides by vacuum deposition method at UNF. A interface cicuit board to interface an array is also shown in Fig.2 Fig. 3 shows the UNF Osprey Ozone sensors-2008 consisting of an array of 24 ITO gas sensors interfaced with a printed circuit board (PCB), flexible Kapton heater, electrical fan and 26 pins flat cable.



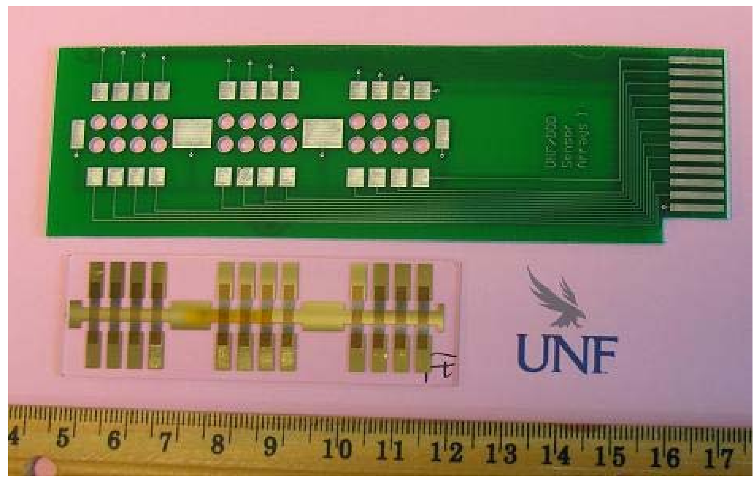


Fig.1 Top view of one ITO gas sensor

Fig. 2 An array of 24 ITO gas sensors with interface circuit board (PCB) (UNF Patent pending)

←Fig. 3UNF Osprey Ozone sensors-2008 consisting of an array of 24 ITO gas sensors with PCB, flexible heater, fan and flat cable

How does sensor sense?

<u>Interaction of oxidizing gas on surface of n-type ITO thin film sensor</u>

Upon adsorption of charge accepting molecules at the vacancy sites, namely from oxidizing gases such as ozone (O_3) and NO_2, these electrons are effectively depleted from the conduction band of ITO. Thus, this leads to an increase in the electrical resistance of n-type ITO.

For example ozone gas:

Oxygen vacancy (V) + Ozone (O_3) →Lattice Oxygen site (O_0) +O_2

Vacancies can be filled by reaction with either oxygen or ozone. Filled vacancies are effectively electron traps and as a consequence the resistance of the sensor increases upon reaction with oxygen or ozone.

<u>Interaction of reducing gas on surface of n-type ITO thin film sensor</u>

Oxygen vacancies on ITO surfaces are electrically and chemically active; these vacancies function as n-type donors decreasing the electrical resistivity of ITO. Reducing gases such as CO, H_2 and alcohol vapors result in detectable decreases in the electrical resistance of n-type ITO.

For example methanol:

CH_3OH (methanol) + O^- (chemisorbed ion on surface of ITO)

→ HCOH (Formaldehyde) + H_2O (water) + e^- (electron)

Vapors come in contact with surface and reacts with chemisorbed oxygen ions O- or O^{-2}, and re-injecting electrons in the conduction band.

Working Principle:

The electrical resistance of ITO increases in the presence of oxidizing gases such as ozone. Upon adsorption of the charge accepting molecules at the vacancy sites, namely oxidizing gases such as ozone, electrons are effectively depleted from the conduction band, leading to an increase in the electrical resistance of n-type ITO.

Calibration of ITO Sensors

The ITO Sensors array was tested and calibrated at different pressures at UND. These sensors were also calibrated at the Space Life Science Laboratory of Kennedy Space Center, NASA last year. An ozone gnerator was used as the source of ozone, which generated 0 to 12 ppm ozone gas with accuracy of 0.10 ppm. Fig. 4 shows the simultaneous testing of 3 sensor arrays and the ozone generator in the test vacuum chamber.

Fig. 4 Testing and calibration of UNF Ozone sensors arrays with ozone generator in the vacuum chamber at UND

Fig. 5 shows the calibration of the UNF Ozone sensor with ozone gas in the range of 0 to 10.3 ppm under low pressure. The usual variation of ozone in the stratosphere is about 0.5 to 10 ppm. It was observed that response of the sensors have two distinct linear variations: one from 0 to 4 ppm, and another from 4 to 10 ppm.

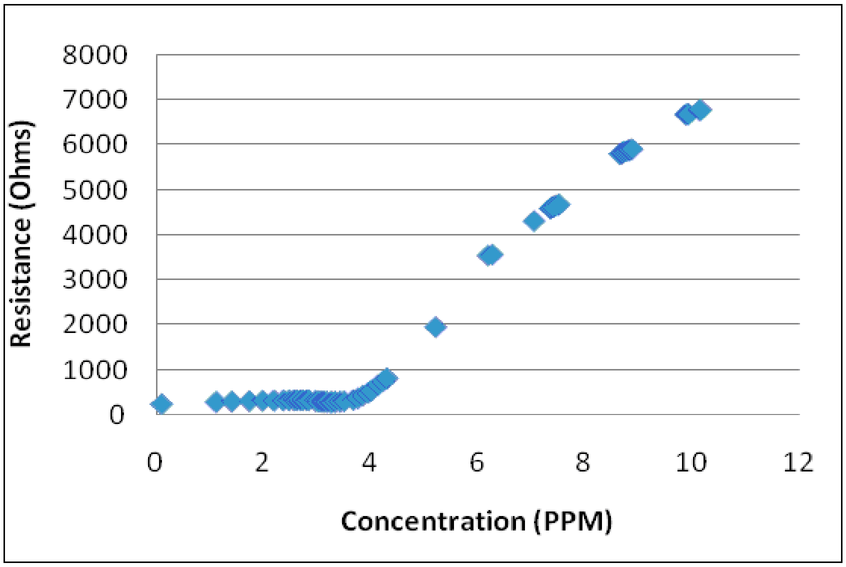


Fig.5 Calibration of ITO gas sensor with ozone gas (higher concentration range)

Fig. 6 shows the enlargement of the calibration of the sensor in the lower concentration ranges. The UNF Ozone sensor detected about 0.15 ppm, the lowest concentration of generated ozone.

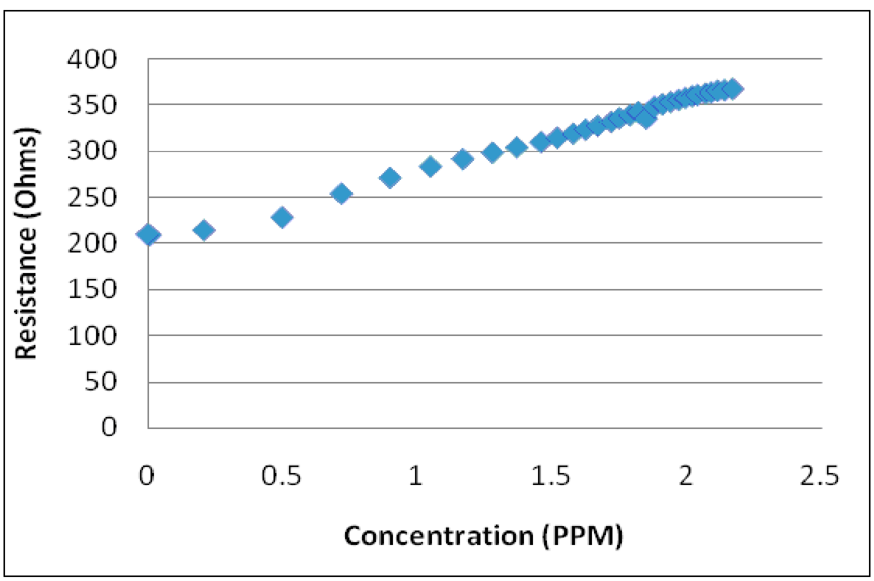


Fig. 6 Calibration of ITO sensor with ozone gas (lower concentration range)

Fig. 7 shows the response of the ITO sensor with an electronic circuit in terms of voltage with lower concentrations of ozone at low pressure. The linear trend line equation was used to develop the algorithm for the data analysis of measured data.

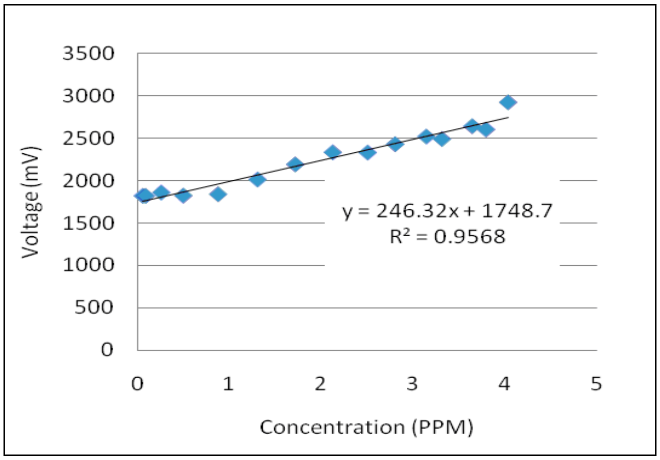


Fig.7 Calibration of ITO gas sensor with electronic circuit output with ozone gas (lower concentration range)

Fig 8 shows the cyclic response of the sensor system at the different concentrations of ozone in the 1 atm pressure in the test chamber. It was observed that the response of the sensor is faster at the lower concentrations of ozone.

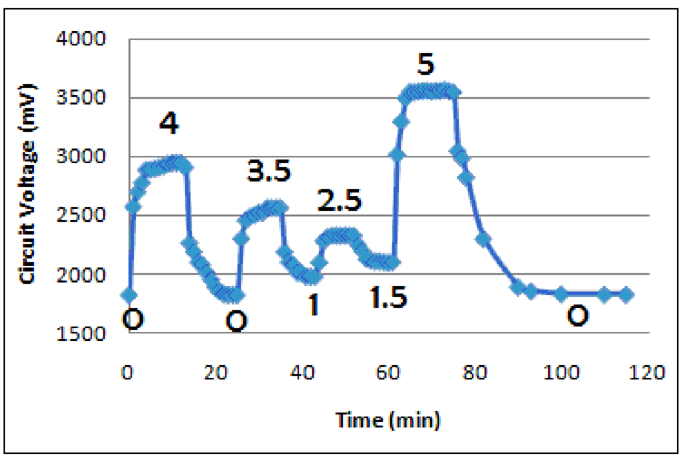


Fig. 8 Cyclic response of ITO sensor (printed number on each cycle represents the concentration of ozone in ppm)

Payload

Fig. 9 shows UNF sensors array on the PCB, and the UND electronic circuit.

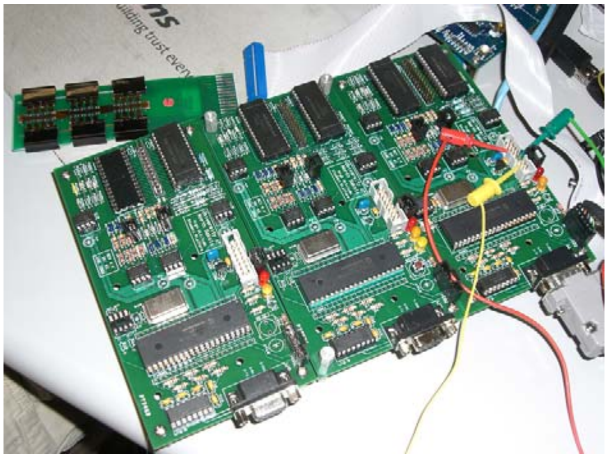


Fig. 9 UNF sensors array on PCB and UND electronic circuit

Fig. 10 shows the block diagram of circuits for the payload.

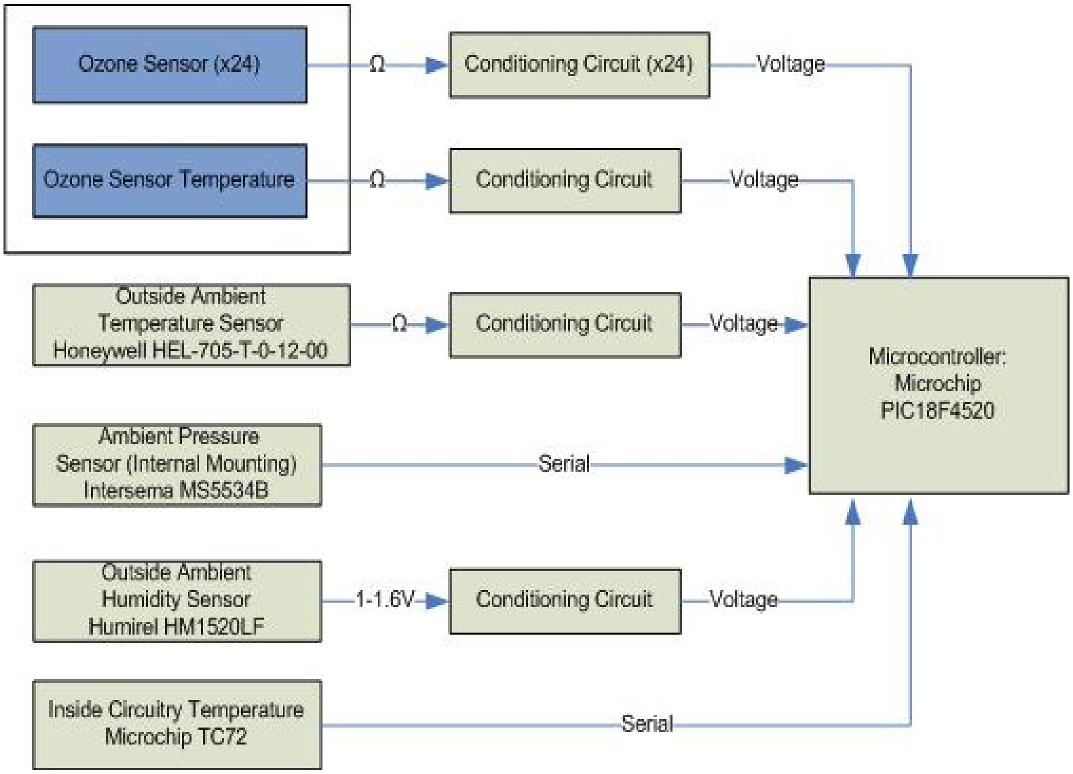


Fig.10 Block diagram of electronic circuits

Fig. 11 shows the flow chart for measurement and data collection.

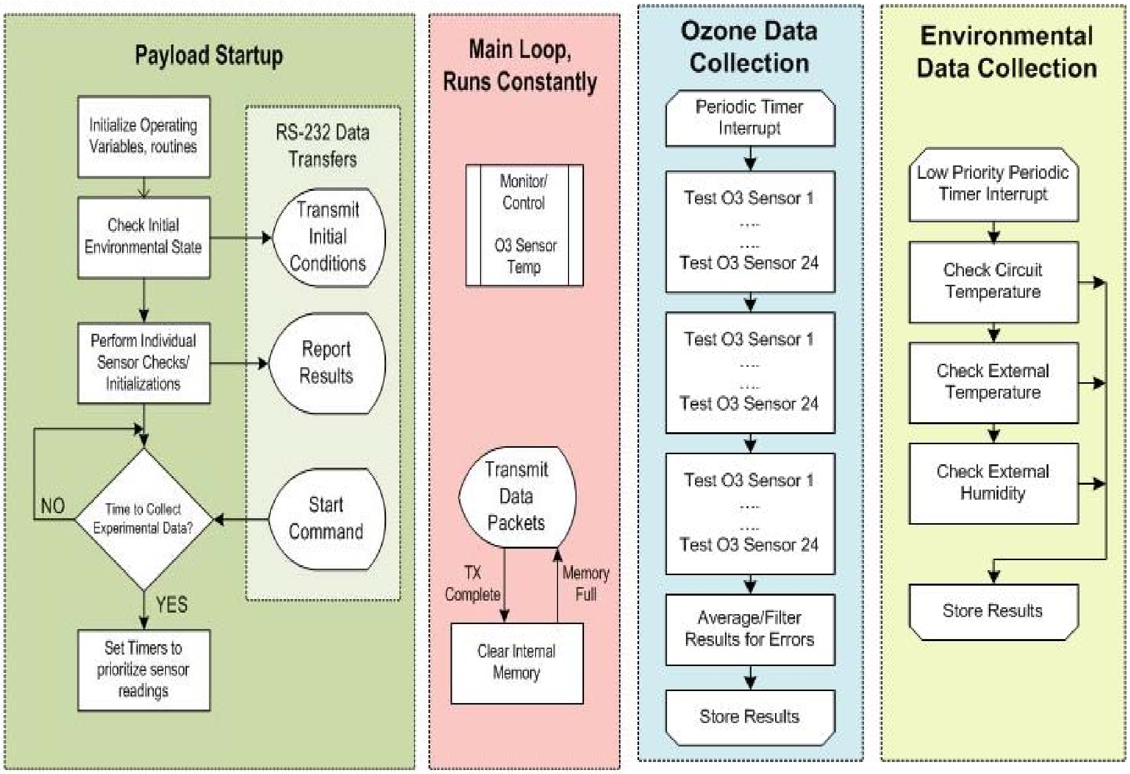


Fig. 11 Flow Chart for measurements and data collections

Fig. 12 shows the UND-UNF payload, which was mounted on the platform of HASP

Fig. 12 UND-UNF Payload (Payload #7) on the HASP Platform

The technical details of the payload on HASP are given below:

Payload Details:
Mass: 2.0 kg, Foot Print: 15 cm x 15 cm Height: 30 cm
Supplied Voltage: 29-33 VDC Maximum Current: 0.5 A
Max. Serial Downlink (bitstream): 1200bps, 2bytes/command
Serial Interface: 1200 baud, RS232 Protocol, DB9 Connector
Discrete Commands: Power ON / OFF
Analog Downlink: 2 Channels (0-5VDC)
Analog & Discrete Interface: EDAC 516-020
Downlink Rate: 36 kbps (Through website)

The UND-UNF payload was tested in the BEMCO chamber, which is shown in Fig. 13 (a) and (b) for high temperature (56^{o}C), low temperature (-60 ^{o}C), high pressure (1.5 atm), and low pressure (down to 1mbar). The payload was also tested with the shock and vibration test (10g vertical and 3 g horizontal), and also a hang test.

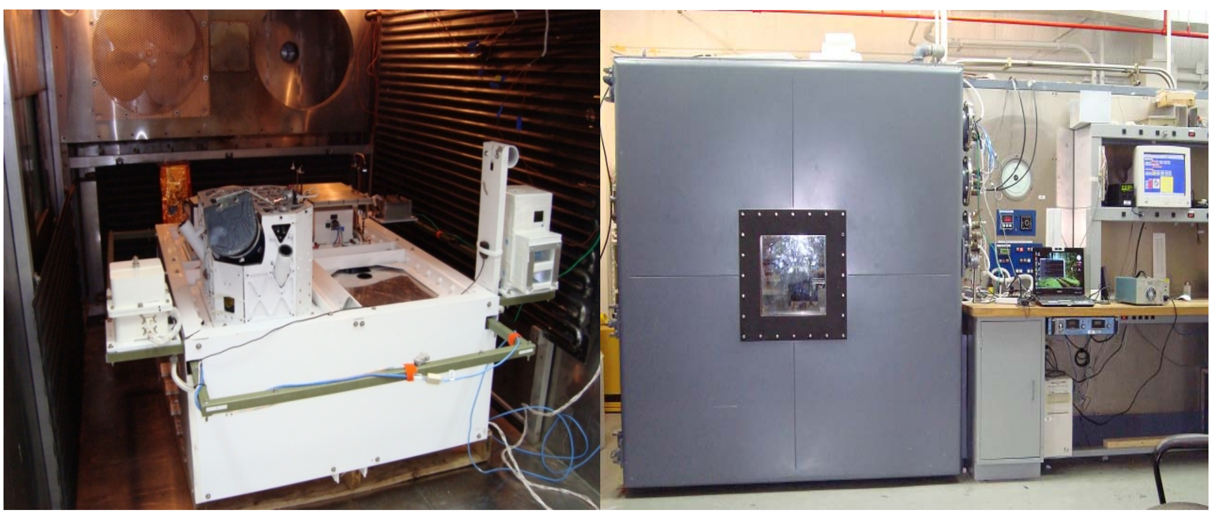


Fig. 13 (a) UND-UNF payload in the thermal and vacuum chamber and (b) Thermal and Vacuum Testing of UND-UNF Payload in the large chamber at NASA-CSBF, Palestine, TX

The NASA team approved the UND-UNF payload for launch after passing of all the required tests. Fig. 14 shows the hanging of all the payloads on the HASP platform by the moving truck “Big Bill”. The payload was connected with the high altitude balloon and the parachutes.

Fig. 14 “Big Bill” with HASP moving for launch on the runway at Fort Sumner, NM

The balloon parameters are given in the table below.

Balloon Parameters (Courtesy: HASP)

Balloon Type	Zero pressure, 1 cap CSBF #979)
Balloon Size	11.82 million cubic feet
Parachute Diameter	79 feet
HASP Weight	411 pounds
SIP Weight	589 pounds
Balloon Systems	458 pounds

Ballast	542 pounds
Altitude with Ballast	122,500 feet
Altitude without Ballast	126,000 feet
Ballast for Drive-Up	140 pounds
Ballast for Sunset	259 pounds

The high altitude balloon filled up with helium gas is shown in Fig. 15.

Fig. 15 High altitude balloon filled up with helium gas (Courtesy: HASP)

Fig. 16 shows the picture of high altitude balloon attached with parachutes and HASP payloads.

Fig. 16 Balloon attached with parachutes and HASP payloads (Courtesy: HASP)

Fig. 17 shows the launching of balloon attached with the HASP payloads.

Fig. 17 Launching of balloon attached with parachutes and HASP payloads (Courtesy: HASP)

The onboard “Cosmo Cam” video camera with GPS System provided real time video, as well as data for altitude, latitude, longitude, pressure and temperature during the flight. Fig. 18 shows one of the pictures taken by the “Cosmo Cam” camera, which shows the UND-UNF payload with other payloads.

Fig. 18 UNF Osprey flying at altitude of about 37 km (Picture taken by “Cosmo Cam” video camera).

Fig. 19 shows the HASP balloon flight profile. The altitude profile was measured by the average of four GPS instruments. The flight duration was 31.8 hours, while the average float altitude was 120, 080 feet.

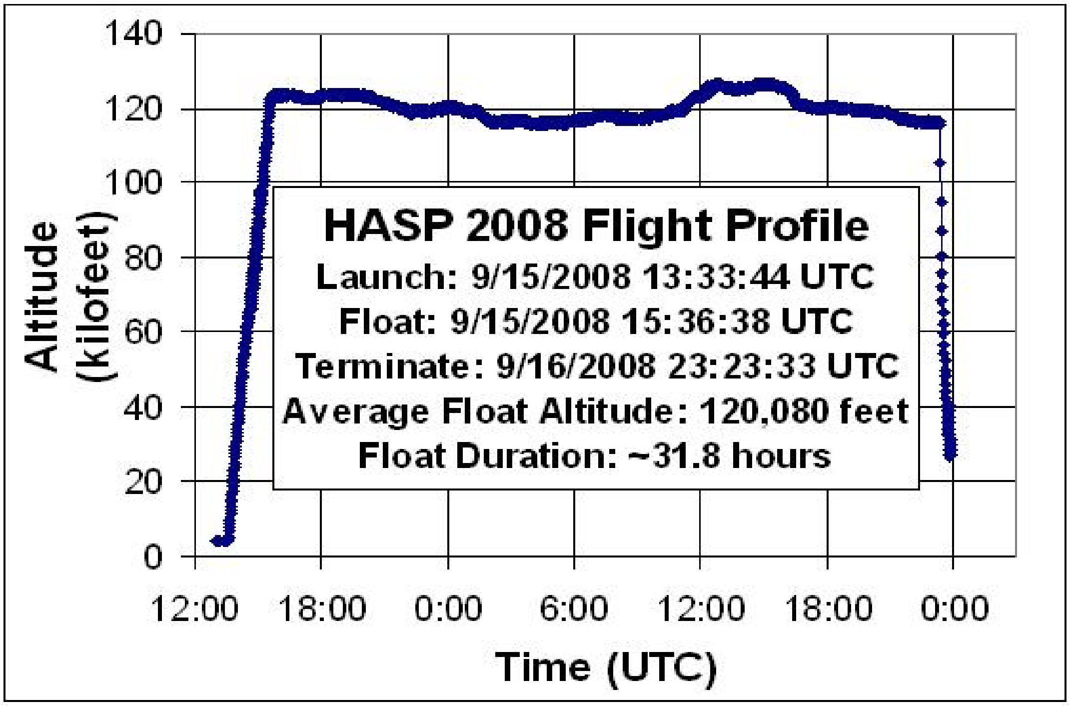


Fig. 19 HASP balloon flight profile (Courtesy: HASP)

Fig. 20 shows the flight path of the balloon on the Google map. The flight was started from Forth Sumner, and terminated near the border of Texas and New Mexico. The flight got additional flight time period due to the convoluted path of balloon. The reason for the convoluted path was the very indeterminate high altitude winds that occur during "turn-around" conditions.

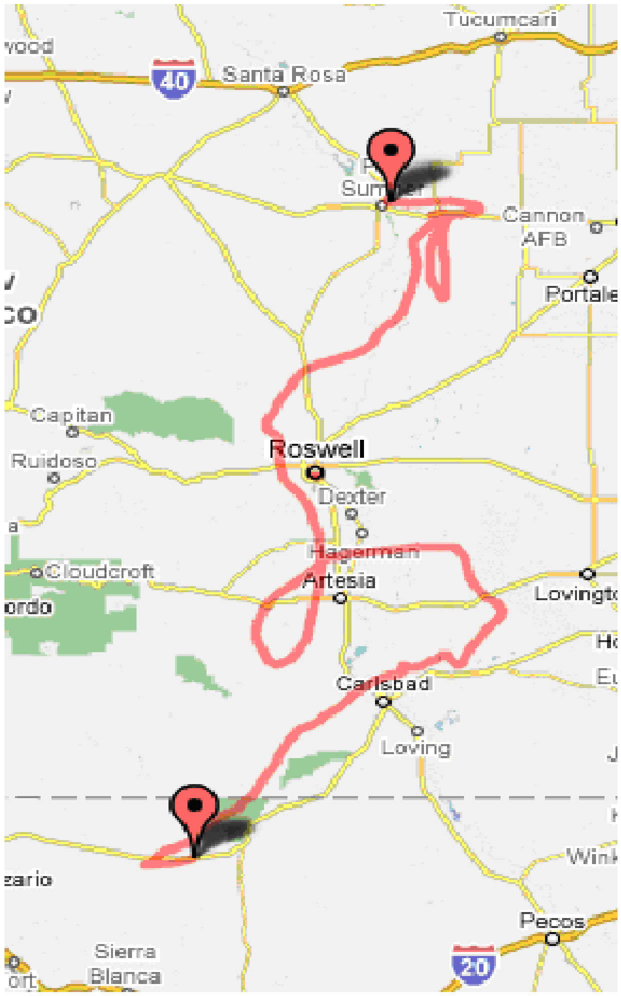


Fig. 20 The path of HASP balloon flight on the Google map

During the flight, UNF sensors measured the ozone profile. The payload sent data files of 2KB every 7 minutes during the flight time through the NASA-HASP computer and was uploaded on the HASP website. We downloaded all the data files, and applied the calibration algorithm using software program. We found that the sensors, hardware and software worked very smoothly. There was no need to give special commands to reboot the payload system. Fig. 21 shows the variation of the ozone concentration measured by the UNF sensors with the altitude measured by the GPS. Each data point of the profile is an average of 24 sensors.

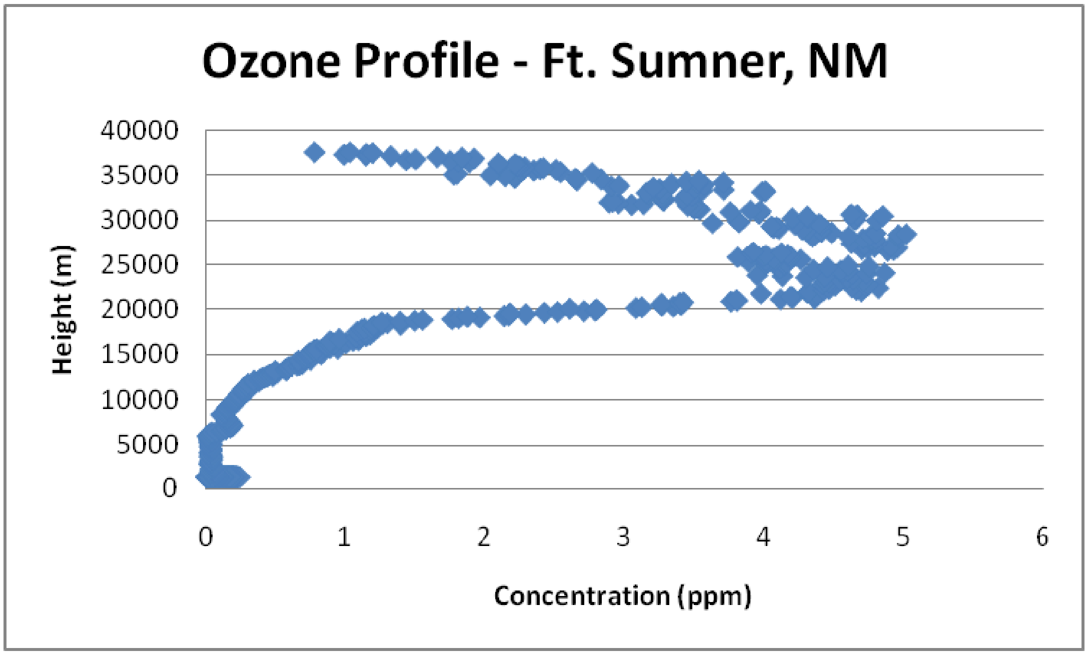


Fig. 21 Experimentally measured ozone profile by UND-UNF sensors in the stratosphere

The measured profile matched the theoretically predicted profile, which is shown in the Fig. 22 for comparison.

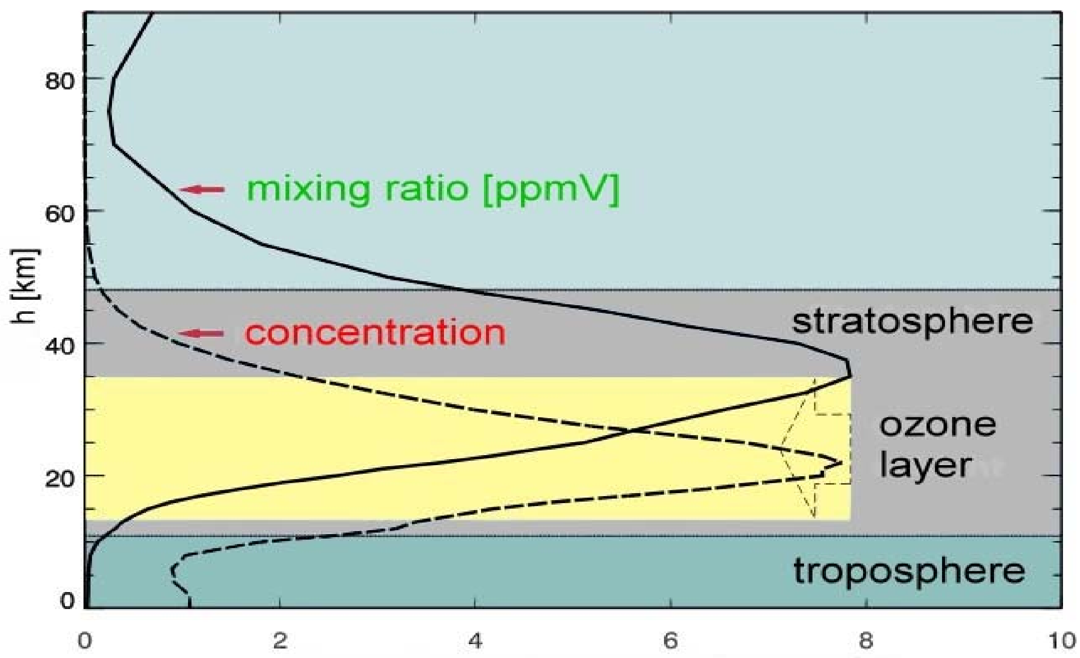


Fig. 22 Theoretical ozone profile in stratosphere **(http://www.atmosphere.mpg.de/enid/1yy.html)**

This profile clearly indicates that the UND-UNF payload successfully measured the ozone profile in the stratosphere. The measured maximum value of ozone was a few ppm less than that of the expected theoretical value. Possibly, the outward convective lifting by the feeder bands from hurricane Ike produced low mixing ratios. NASA scientists reported earlier that ozone levels drop when hurricanes are strengthening (http://www.nasa.gov/vision/earth/environment/ozone_drop.html). Our results were supported by NASA scientists.

Scanning electron microscopy surface of sensors before and after flight

Two identical sensors arrays were fabricated under identical conditions at same time. One was used for flight, while other was kept for standby. The surface morphology of standby ITO thin film gas sensor before flight was examined using scanning electron microscope (SEM, FEI Quanta 200) at UNF. Fig. 23 shows the scanning electron micrograph of ITO thin film sensor before flight. The average grain size of film is about 75 nm. The most of grains were none overlapping to each other.

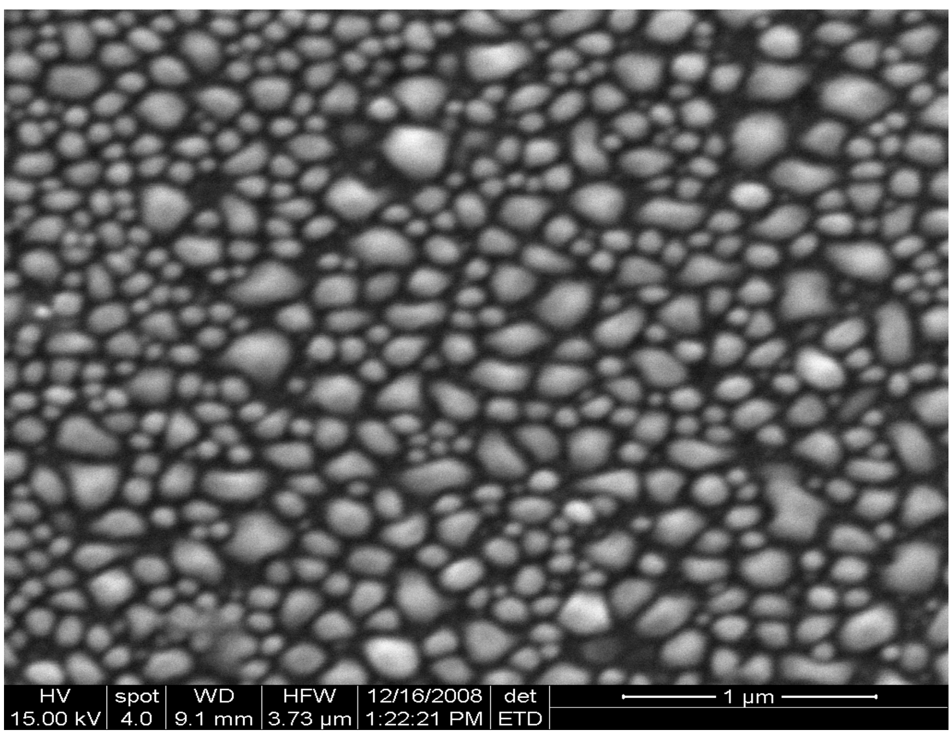


Fig. 23 Scanning electron micrograph of surface of ITO thin film gas sensor before balloon flight

After the termination of flight, the HASP payload was recovered and was returned to UND. UNF got the sensors box from UND. The surface morphology of recovered ITO thin film gas sensor after flight was examined using SEM. Fig. 24 shows the scanning electron micrograph of ITO thin film sensor, which shows that the average grain size of film is about 75 nm.

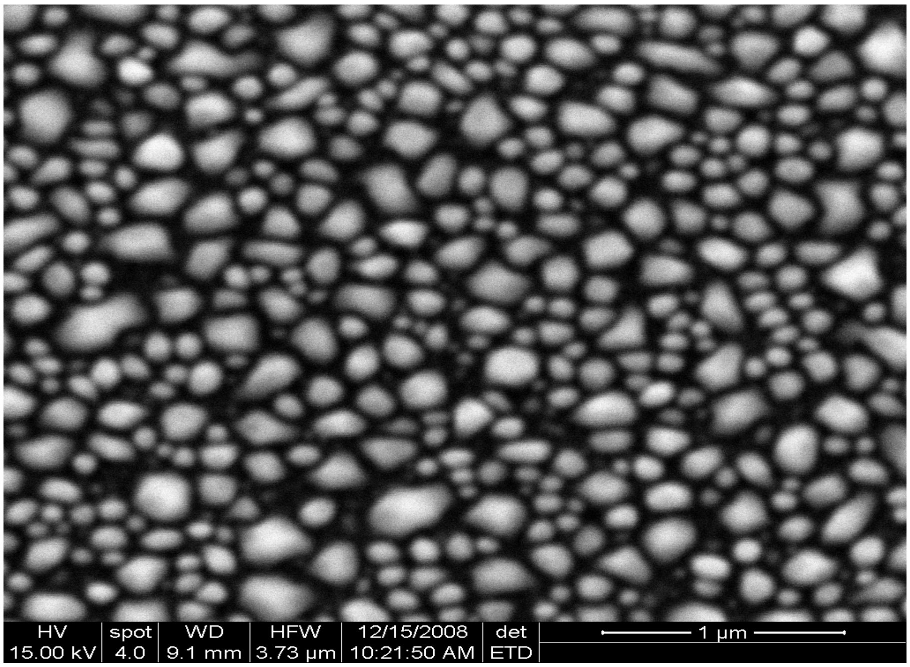


Fig. 24 Scanning electron micrograph of surface of ITO thin film gas sensor after recovering of payload

It was found that there was no change of morphology of the surface of ITO thin film gas sensor after flight of about 32 hours. However, some patches of moisture condensation after termination of flight or landing on ground or during payload transportation and also dust particles on the surface were observed under low magnification of SEM, which is shown in fig. 25. Fig. 25 shows the condensation of moisture on the surface of sensors by gray color patches and dust particle by one white color particle on the surface. The moisture condensation on the surface of sensor during the flight can be ruled out because of the internal heater maintain the constant temperature as well as internal fan circulate air molecules uniformly inside the payload. Moisture patches and dust particles can be avoided by improving the packaging of sensors as well as payload design.

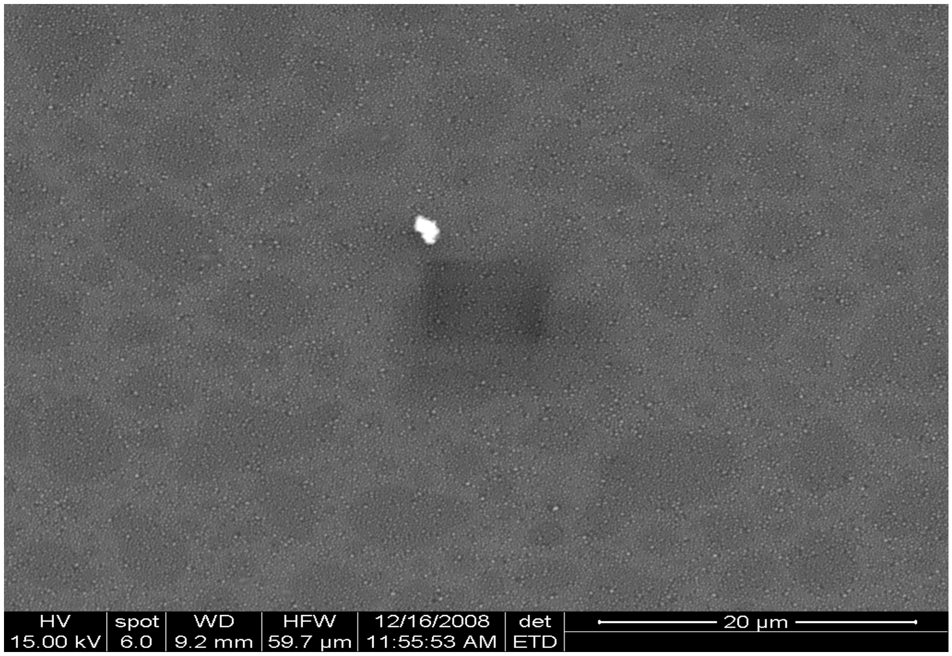


Fig. 25 Low magnification scanning electron micrograph of surface of ITO thin film gas sensor after recovering of payload.

The chemical composition of sensors was determined by energy dispersive analysis of x-rays (EDAX) attached with SEM. Fig. 26 shows the EDAX spectrum of sensors, while fig. 27 shows the quantity analysis of ITO thin films. It was observed that the composition remain nearly same before and after flight. No significant change was observed.

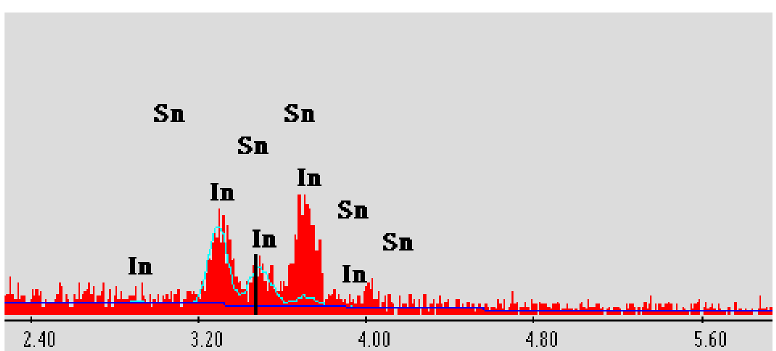


Fig. 26 EDAX spectrum of surface of ITO thin film gas sensors

Matrix Correction: ZAF

Element	Wt%	At%
InL	87.92	88.27
SnL	12.08	11.73

Fig. 27 Quantity analysis of surface of ITO thin film gas sensors

SEM and EDAX confirmed that ITO thin film gas sensors have good surface as well as chemical stability. The sensors after recovering were found in working condition and still in working condition on today (18^{th} Dec., 2008). In addition, it was observed that the average resistance of array of 24 sensors before flight was 235±3 ohms, while 255±3 ohms after flight. The little increase of resistance of sensors after recovering may be due to increase of contact resistance between gold pad of sensors and interface PCB via copper adhesive tape and patches of water condensation or damage of surface by striking of dust particles.

Conclusion:

(i) Nanocrystalline ITO thin film gas sensors array made by UNF was successfully calibrated with ozone gas at UND, integrated with electronic circuits by UND, pass all the HASP tests and detected ozone in the stratosphere during HASP flight without any problem.

(ii) The measured ozone profile matched with the expected profile, but the magnitude of maximum value of ozone was found to be lowered by few ppm because of the effect of IKE hurricane.

(iii) UND-UNF payload need further improvement in the design, adding the different types of ozone sensors for the comparison and need one more opportunity for the HASP flight

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(iv) Kennedy Space Center, NASA

(v) University of North Dakota, Grand Forks, ND

(vi) University of North Florida, Jacksonville, FL

(vii) Department of Defense, US Army, Edgewood Chemical & Biological Center (ECBC), APG, MD