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Generation of Pulsed, Energy-Selected Metal Atom Beam by Laser Vaporization of Metal Compounds

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12 November 1991



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**CHARLES P. PIKE, Chief
Spacecraft Interactions Branch**

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Preface

This report describes the laser vaporization experiment located in the Spacecraft Interactions Branch. The purpose of the experiment is to determine thermochemical properties of materials used in space research. The laser vaporization apparatus is a modified high temperature mass spectrometer in which the Knudsen cell assembly is replaced by a disc of a precursor material that is irradiated by a pulsed laser beam. The beam of laser-vaporized material is crossed by a pulsed, supersonic molecular beam of oxide molecules. The laser-produced beam and collision products are characterized by time-of-flight spectroscopy.

Generation of Pulsed, Energy-Selected Metal Atom Beams by Laser Vaporization of Metal Compounds

1. INTRODUCTION

Materials used in low earth orbit are exposed to an atomic oxygen rich environment.¹ At orbital velocities, oxygen atoms collide with surfaces perpendicular to the velocity vector with a mean kinetic energy of 4.9 eV. Studies of materials subjected to this environment have indicated that these atom-surface collisions result in effects similar to those observed in laboratory studies of high temperature vaporization and oxidation.² Materials in these studies are found to erode, disappear completely, or gain mass due to the formation of an oxide coating. In many instances, thermochemical properties such as heats of formation and bond energies of the species involved in the degradation process have not been experimentally determined. Estimates based on trends in the periodic chart have been applied. A better data base is needed to understand the vaporization or decomposition of these materials in the space environment.

For over thirty years, high temperature effusion beam mass spectroscopy has proven to be an invaluable tool in the study of high temperature reaction equilibria. In such an experiment, the measured abundances of the species present in the beam effusing from a Knudsen cell yield respective partial pressures which enable the determination of the equilibrium constant K_{eq} of the reaction being investigated.³ This technique, however, is restricted by the limit in accessible temperature and the accuracy in determining the temperature inside the cell.

The use of an intense laser pulse to vaporize a sample inside a mass spectrometer is well known.^{4,5} The first theoretical consideration of plasma production and heating by means of a laser beam was presented by Basov and Krokhin in 1963.⁶ Since that time, a considerable amount of work, both theoretical and experimental, has been devoted to the study of the interaction of laser beams with solid surfaces. Much of the characterization of the energetics of the ablated material has been done by time-of-flight spectroscopy.

In 1974, Friichtenicht proposed a technique for the production of an atomic beam with chemically significant amounts of kinetic energy, in the 1 to 10 eV range.⁷ This technique involved the essentially instantaneous vaporization of a thin film of appropriate material in vacuum by irradiation with the output from a Q-switched laser. The dense cloud of material expands and becomes collisionless, after which the beam species follow straight-line trajectories. Time-of-flight spectroscopy is used to determine the atom's kinetic energy.

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1. Because of the large number of references they are not listed here, see page 17.

This method of reaction beam production has been used by Wicke⁸ and others⁹ to study the reaction dynamics of various metals with oxidizing gases. For the reaction:



bond energies can be determined by observing a collision energy threshold for the production of MO. An observed center of mass kinetic energy threshold (KE_T) for oxide formation yields the bond dissociation energy of the metal oxide using conservation of energy:

$$D(MO) = D(XO) - KE_T \quad (2)$$

If the metal oxide is formed considerably above threshold, chemiluminescence of the metal oxide is frequently observed yielding spectroscopic and structural information.

The instrumentation designed to study these types of reactions is described in Section 2 of this report. In Section 3 the time-of-flight calibration of the pulsed valve as well as some preliminary results of metal and metal oxide vaporization will be discussed. In Section 4, an evaluation of the instrument's performance to date and future work using the instrument will be discussed.

2. INSTRUMENTATION

2.1. Overview of the Apparatus

The apparatus consists of a modified Nuclide high temperature mass spectrometer (Model 12-60), a Nd:YAG laser, and associated optics and electronics, which are shown in Figure 1. The mass spectrometer is evacuated using 4- and 8-inch diameter cryopumps (CTI Cryogenics) and one 4-inch diameter ion pump (Varian). A pulsed Nd:YAG laser is used to vaporize a sample. The laser-produced beam travels 0.279 m to the ion source where it is ionized by electron impact. The resulting ion beam is accelerated by a 4500 V potential drop in the ion source into the 60°, 12-inch radius magnetic sector mass filter. The mass-selected ions are then detected by a microchannel plate. The distance from the ion source to the microchannel plate is 1.373 m for a total time-of-flight distance of 1.652 m. The output of the detector is amplified by a fast preamplifier and counted by a multichannel scaler in a time-of-flight mode. The major components of the instrument are described below.

2.2. Laser and Beam Alignment

The laser used for vaporization of the sample is a Quantel Model YG661S Nd:YAG laser. The laser is an oscillator-amplifier system in which two 6mm YAG rods are pumped by one flashlamp. The 1064 nm fundamental output can be frequency doubled (532 nm) or tripled (355

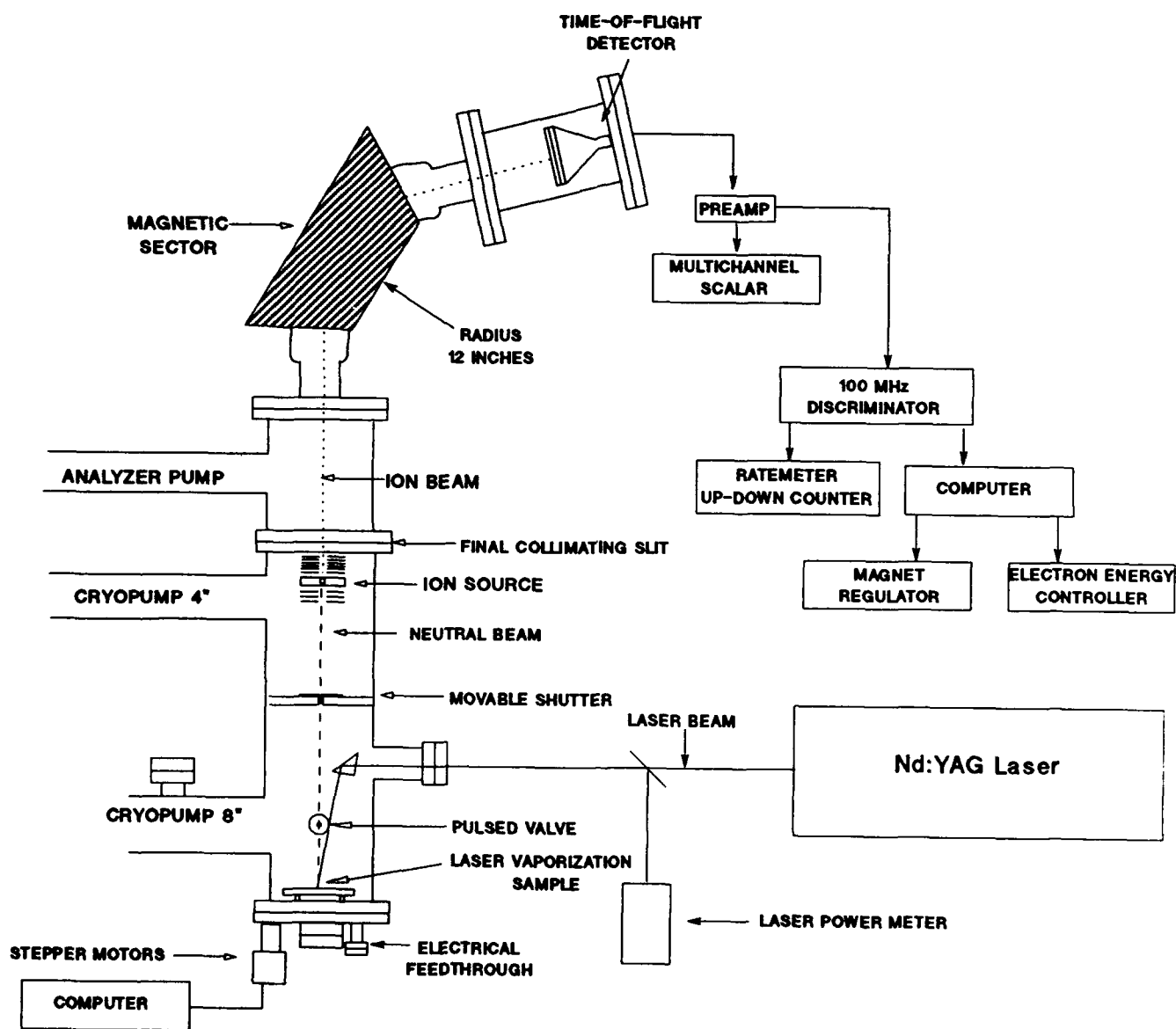


Figure 1. Laser Vaporization Time-of-Flight Mass Spectrometer

nm) using one or two KDP crystals. The pulse width of the beam is approximately 10 ns and it has a variable repetition rate of 2, 5, or 10 Hz, controlled through the LU660 logic unit. The LU660 also serves as the trigger for both the pulsed valve and time-of-flight data acquisition. The maximum laser pulse energy is 270 mJ/pulse (2nd harmonic) with a beam diameter of 5 mm. Laser power is varied rotating a polarizer positioned prior to the first prism.

The laser beam is directed to the sample by the use of five prisms, four outside of the vacuum chamber and one inside. A fraction of the beam is picked off prior to the first prism into a calorimeter (Scientech, Inc.) for power monitoring during signal acquisition. The fifth and final prism is located inside the mass spectrometer on a rotary feedthrough with a worm gear reduction for more precise movement. The prism is adjusted to direct the laser beam onto the center of the sample holder. This can be monitored by a photodiode that detects laser light passing through a pin hole in the sample center (See Section 2.3 - Sample Holder). The total power reduction through the complete optical system is approximately 50 percent. Higher power densities are obtained using a 1000 mm focal length lens.

2.3. Sample and Sample Holder

The sample to be vaporized is held in a stainless steel platform mounted on an X-Y positioner (See Figure 2). The sample is either a $\frac{5}{8}$ in. diameter, $\frac{1}{16}$ in. thick metal disk or a powder that has been pressed into a $\frac{1}{2}$ in. diameter pellet. A fresh surface is presented to each pulse of the laser beam by moving the sample while the target spot remains fixed. An X-Y positioner is moved inside the mass spectrometer by two stepper motors mounted on feedthroughs. The stepper motors are controlled by software that creates a 10 mm square area for laser irradiation. With a beam diameter of approximately 1 mm, 100 laser shots can be taken before the same area is irradiated again, greatly increasing the lifetime of the sample. Calibration samples have a 0.020 in. hole in the center.

2.4. Pulsed Valve

A pulsed valve (General Valve Corp.) is used in this experiment to produce a supersonic molecular beam of an oxidizing gas perpendicular to the laser produced beam. It is mounted on an adjustable sleeve inside the first chamber of the mass spectrometer at a known distance between the sample and the ion source. Gas is supplied to the valve through a gas manifold. A backing pressure of 600-800 Torr is normally applied. The pulsed valve parameters are controlled by the IOTA ONE pulse driver system (General Valve Corp.). The pulse width used in the experiment is approximately 180 μ s and is adjustable. The pulsed valve driver is triggered by a TTL level pulse from the Q-switch of the LU660 for synchronization with the laser vaporized beam.

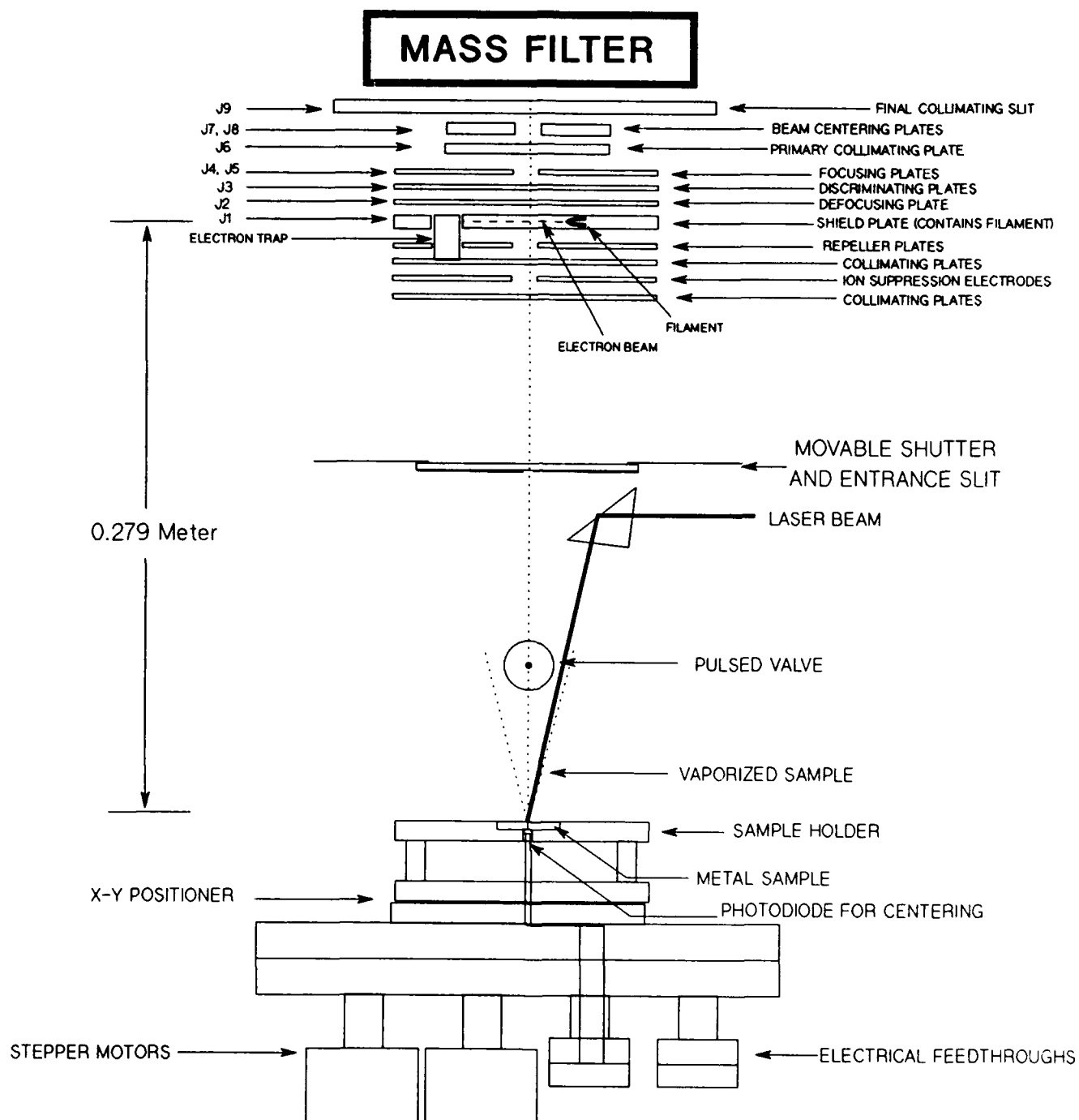


Figure 2. Sample Holder/Ion Source Assembly

2.5 Ion Source/Magnetic Sector

The ion source and magnetic sector used in this experiment have been described previously and will be outlined briefly.¹⁰ The neutral beam enters the ion source chamber through a slit in a movable shutter. The ion source consists of collimating plates, a repelling electrode, and electron impact ionization source including a thoriated iridium filament (located in J1) and an electron trap, and eight other accelerating and focusing plates, J2-J9. The atomic or molecular beam is ionized by a beam of electrons from a hot filament emitted perpendicularly to the beam. The emission current from the filament is variable and can be set as high as 5.0 mA for increased ionization efficiency. Filament emission current is controlled by a feedback circuit that adjusts the voltage across the filament to maintain a constant emission current. The electron energy is controlled by a 12-bit digital output by combining an output port of the CTM-05 (Keithley-Metrabyte) and the 8-bit LPT2 parallel port. This output is connected to the digital input of the electron energy control unit, where the digital data lines are coupled to the ion source high voltage through optoisolators.

After passing through the adjustable object-defining slit in plate J9, the ion beam enters the 60°, 12-in. radius, magnetic mass analyzer. The transmitted mass-to-charge ratio is controlled either directly by adjusting the magnet current or indirectly by adjusting the induction. The magnetic induction is measured by a temperature-regulated Hall probe. The desired induction is obtained by adjusting the magnet regulator. The magnet regulator provides the magnet current necessary to produce a Hall voltage equal to a reference voltage representing the selected mass. For a fixed radius of curvature and accelerating potential, the mass-to-charge ratio is directly proportional to the square of the magnetic field. This is shown by the equation:

$$m/e = (r^2 \cdot B^2)/2V$$

where m is the mass, e the charge on the ion, r the radius of curvature, B the magnetic field and V the accelerating potential. Since the radius of curvature is fixed (12-in.) and the acceleration potential is constant at 4500 V, masses are selected by varying the magnetic field. Electronic sweeping of the magnetic field by an AT computer is discussed in Section 2.8.

2.6 Time-of-Flight Detector

Ions that have passed through the 60° magnetic sector and detector slit are accelerated by a negative 2000 V potential applied to the upper chevron of a stacked pair of microchannel plates (TOF-2003, Galileo Electro-Optics Corp.). A microchannel plate (MCP) is an array of miniature electron multipliers oriented parallel to one another.¹¹ Each electron multiplier is fabricated from a lead doped glass, treated to optimize secondary emission characteristics and to give the channel walls semiconducting properties, which allows charge replenishment from an external voltage source. Electrical contact to each channel is provided by a metallic coating on the front and rear surfaces of each MCP. The contacts also serve as input and output electrodes. The detector is well suited for time-of-flight measurement due to its fast rise time (500 ps) and narrow (≈ 1 ns), distortion free output pulses.

2.7 Data Acquisition/Multichannel Scaler

The negative pulses from the microchannel plate are passed through a fast preamplifier (Stanford Research Systems, Inc.). The amplified pulses are counted by a Stanford Research Systems SR430 Multichannel Scaler. Data acquisition is started by a TTL level pulse from the laser's Q-Switch SYNC/BUSY output. During each time bin, input signal pulses are counted. At the end of the time bin, the counter data is stored in memory and the counter is reset for counting during the next time bin. After the record is complete, the data is added to the accumulation of all previous records. After accumulation is completed, the memory is re-armed for the next trigger pulse. Once the total number of records is completed, advanced functions such as point smoothing, data storage to the internal 3.5 in., 720 kB disk, and hard copies to the attached HP Laserjet III can be accomplished.

2.8 Mass Scanning and Appearance Potential Data Acquisition

The discriminator output of the multichannel scaler is input to a ratemeter for visualization of count rates. The signal is also fed into a modified AT computer used for mass scans and appearance potentials. The software and hardware used to perform mass scans and appearance potentials has been described previously¹⁰ and will be outlined briefly. The output of the multichannel scaler's discriminator is input into a counter on a timer-counter computer interface card (Metrabyte CTM-05). A timer is programmed to produce a constant square wave signal which is connected to the count-enable gate of the signal counter. The dwell time of each counting period is predetermined by the user. The scanning of the magnetic field is done by a voltage ramp generated by a 12-bit digital-to-analog convertor (DAC) computer interface card (Metrabyte DAC-02) with an output range of 0 to +10 V. The resulting analog output of the DAC is adjusted by a variable resistor of a summing amplifier network, allowing the total scan range to be varied. The ramp is added to the mass offset voltage already preset by the user. The amplifier and feedback circuit of the magnet regulator produces whatever output is necessary to make the Hall voltage equal to the reference voltage. A typical mass spectrum is shown in Figure 3.

When scanning of the electron energy is required, a voltage ramp that floats at the ion source acceleration potential must be generated. To do this, the computer produces a 12-bit digital output by combining the output port of the CTM-05 and the 8-bit LPT2 parallel port. The output is connected to the digital input of the Nuclide electron energy control unit, where the digital lines are coupled to the ion source high voltage through optoisolators. The analog ramp is then generated with a digital-to-analog convertor floating at the acceleration potential. The electron energy can be scanned up to the controller's limit of 81.915 V, corresponding to a digital input of 4095 with 20 mV/channel. A typical electron energy scan is shown in Figure 4.

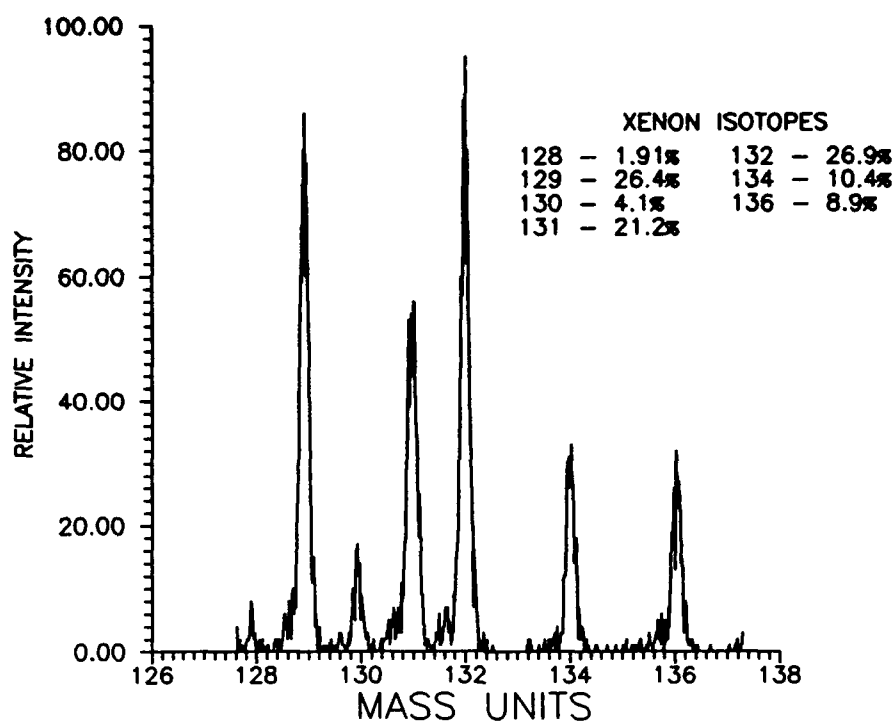


Figure 3. Xenon Mass Spectrum

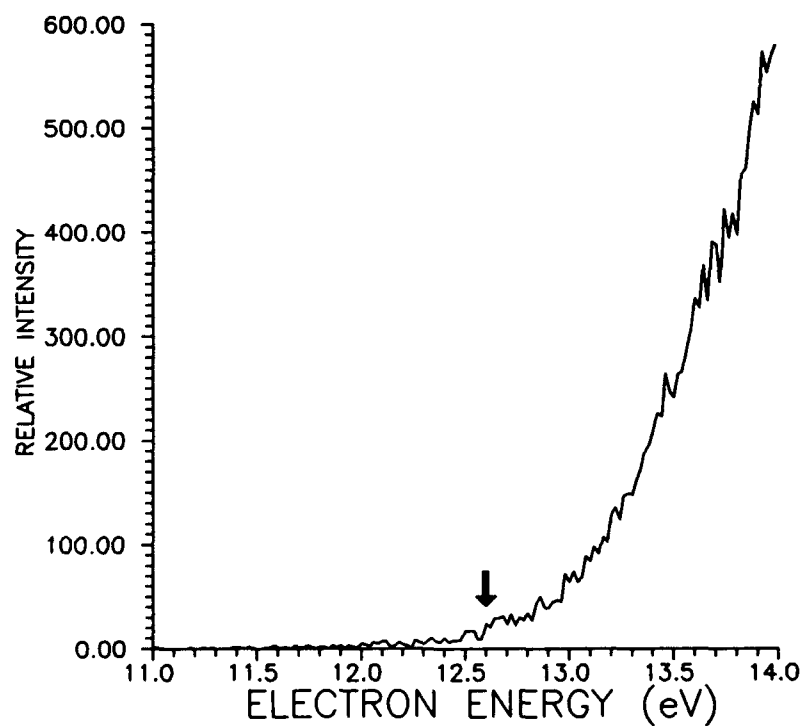


Figure 4. Ionization Potential of H₂O

The AT computer and interface cards are also programmed to operate as a multichannel scaler if mass scans are needed. The purpose of the multichannel software is to:

1. Accept the data acquisition parameters and control commands of the user.
2. Read the counter and store the counts in memory.
3. Control the experiment by driving the digital-to-analog converters.
4. Display the experimental data during acquisition.
5. Store the complete data to disk.
6. Manipulate the data and create hard copies.

The source code is a combination of FORTRAN 77 (main program), MACRO ASSEMBLER (computer interfaces), and C (color graphics and routines). During acquisition, the program reads the counter contents, adds the acquired counts to the memory location of the current channel, increments the digital output of the DAC, and reads the keyboard for commands.

There are four possible acquisition modes. First, in the regular mass scan mode, the digital input of the DAC is incremented by a fixed amount following each count period. Second, since m/e is proportional to B^2 , the X-axis of the mass spectrum will not reflect a linear mass scale. A linear mass scale can be obtained if the digital ramp is incremented according to a square root function. Third, when it is required to obtain data for mass peaks separated by more than a few mass units, data acquisition time is decreased by "hopping" the magnet's induction over areas of insignificant signal. Fourth, an electron energy scan can be performed as described previously.

3. CALIBRATION/INITIAL RESULTS

3.1. Pulsed Valve Characterization

The characterization of the pulsed valve was the first step in testing the time-of-flight experiment. A pulsed valve is used to create a supersonic molecular beam. The valve was mounted on a blank flange at the approximate level of the sample platform to be used in the subsequent laser vaporization experiment, on axis with the entrance slit to the ion source. The calibration involved the use of several different types of gases (O_2 , N_2O , Ar, Kr, and Xe), different backing pressures (100 torr - 1 atm) and different pulse durations to test both the characteristics of the pulsed valve and the vacuum system's capability to pump increased gas loads. A characteristic TOF spectrum is shown in Figure 5. The parameters for this spectrum were 800 Torr backing pressure, 180 μs pulse width, and 10 Hz repetition rate, with 500 scans. It was found that a 180 μs pulse width gave the narrowest distribution.

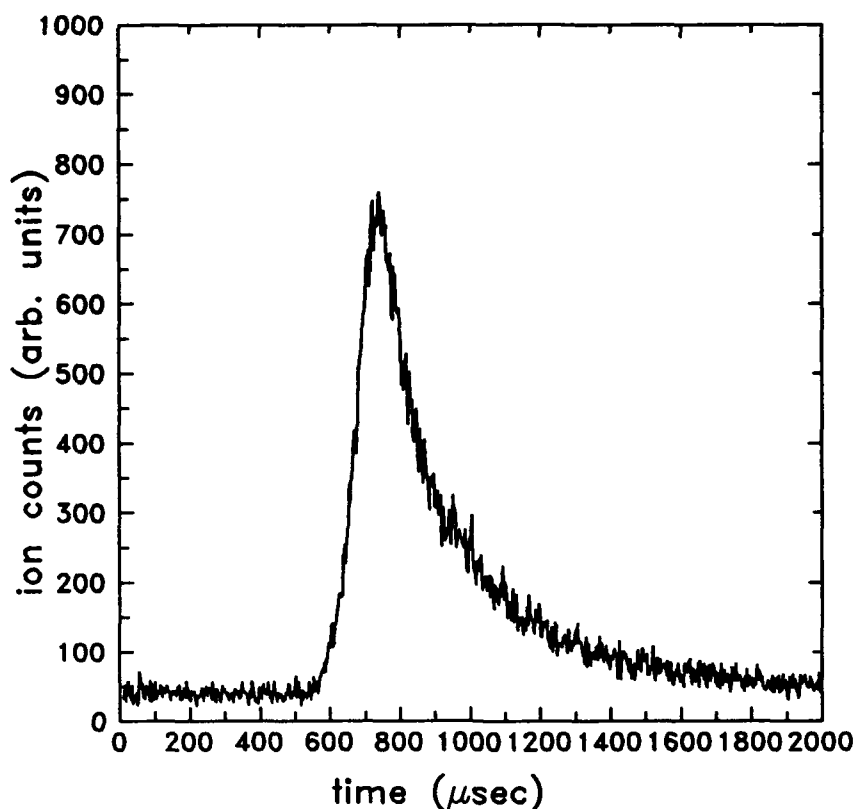


Figure 5. Argon Time-of-Flight Spectrum

3.2. First Laser Vaporized Metal Time-of-Flight

Following the characterization of the pulsed valve, the internal prism mount was installed for the first metal vaporization studies. The first laser vaporized samples to be tested in the mass spectrometer were solid zinc and copper samples. The laser beam was not focused at this time. The object of the initial study was first, to find what type of material would give the most intense neutral atomic beam, and second, to see what effect laser power had on the neutral beam's energy. Zinc was used since it had been studied before.⁸ As with similar experiments used for atomic spectroscopy, the signal was most intense when the sample was first irradiated but dropped off significantly after time. One possible explanation for this is that an oxide coating on the metal disk is less reflective and heat conducting than the metal itself and therefore attains a higher temperature resulting in greater vaporization of the sample.

To test this theory, a pressed pellet of ZnO was vaporized. The ZnO provided a more intense atomic zinc beam for longer acquisition periods. Other samples were then tested to determine the best metal compound for the production of the most intense laser produced beam. Characteristic spectra of Zn, ZnO, and ZnS are shown in Figures 6 through 8. Laser power is the

power measured after the first prism and is shown for comparison only. In conjunction with the different compounds tested, different laser powers were also tested. The results of the test on ZnS is shown in Figures 9 and 10.

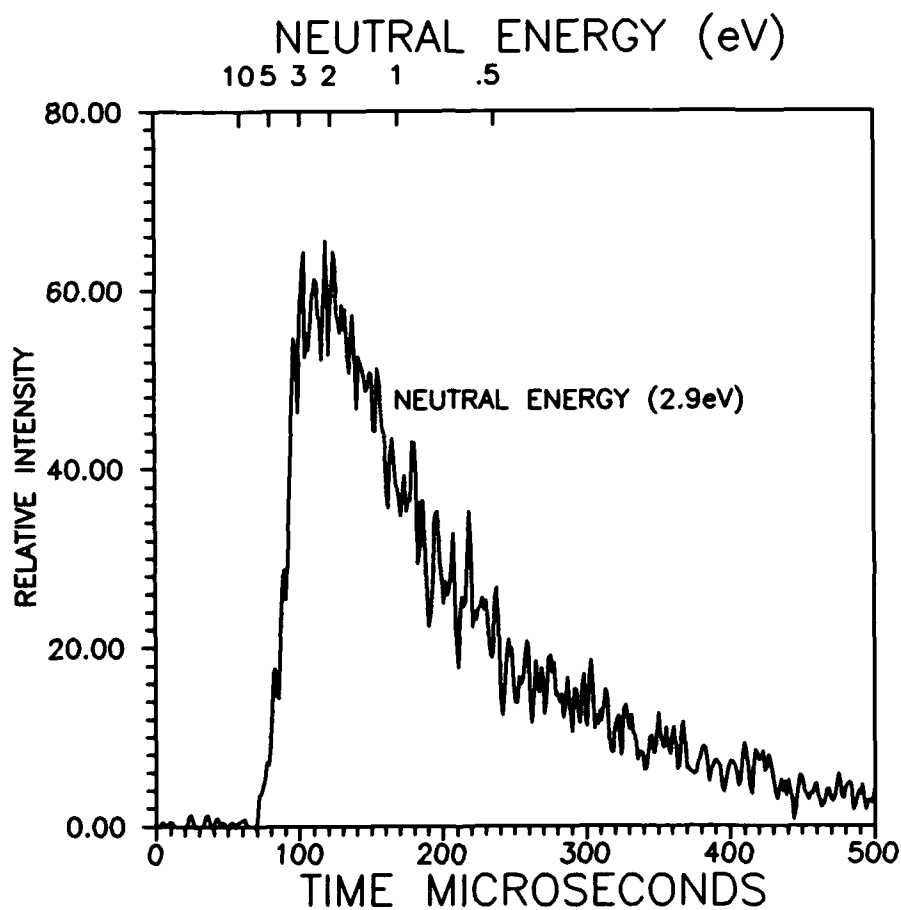


Figure 6. Zn Time-of Flight Spectrum using Zn Metal

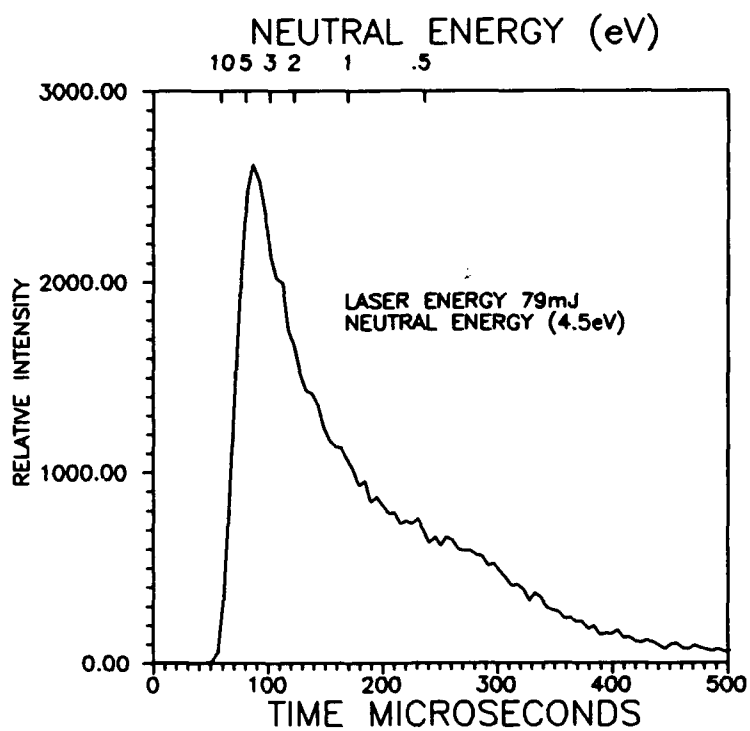


Figure 7. Zn Time-of-Flight Spectrum Using ZnO

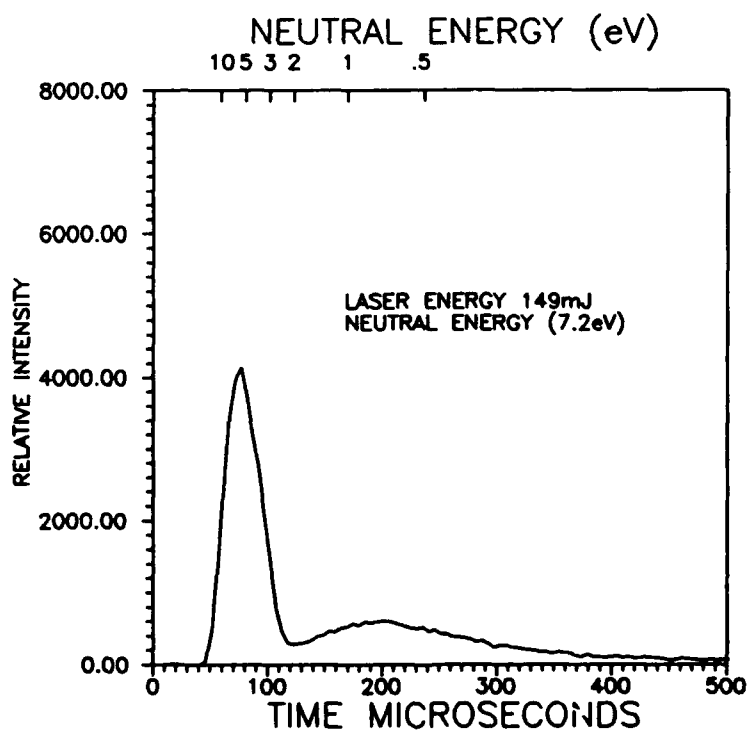


Figure 8. Zn Time-of-Flight Spectrum Using ZnS

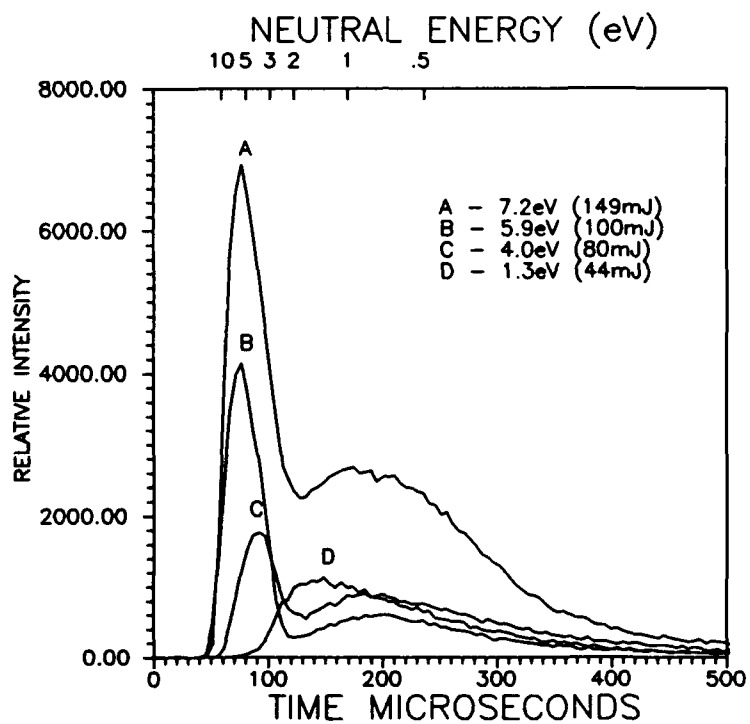


Figure 9. Effect of Variation of Laser Power on Zn Time-of-Flight Spectrum

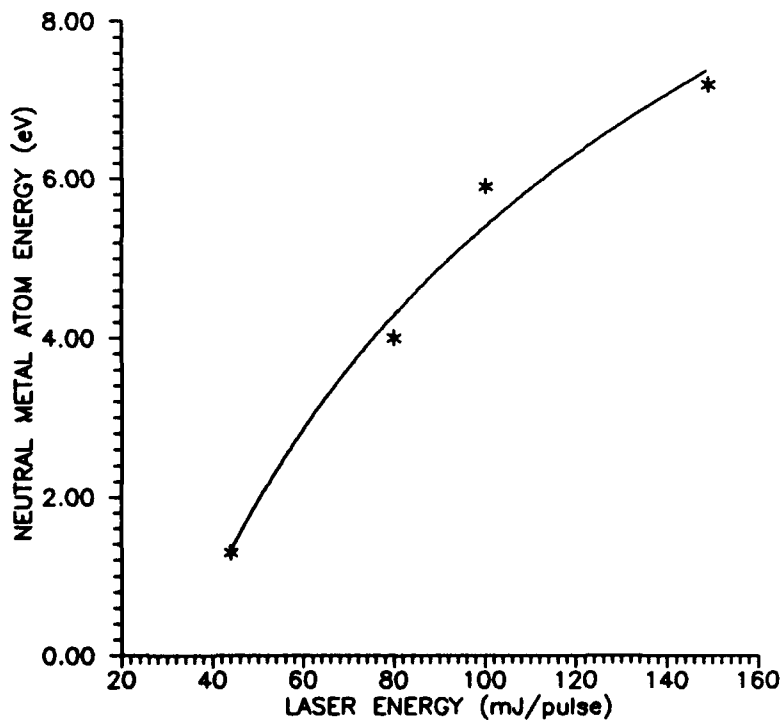


Figure 10. Laser Energy vs. Neutral Metal Atom Energy

4. CONCLUSIONS

4.1. Analysis of the Instrument and Experimental Method

We have described in this Technical Report the modifications to the high temperature mass spectrometer in order to study the thermochemical properties of metals and metal compounds as well as associated high energy kinetics. Replacement of the Knudsen cell assembly with a laser vaporized source should allow the study of even the highest melting point materials. Laser stability is critical in the study of the energetics of the vaporized metal. Variations during data collection greatly widens the time distribution and increases the error possible in the determination of bond energies. The use of disks and pressed pellets greatly increases the lifetime of the sample over that of thin films.

4.2. Future Plans

We plan to continue the study of the interactions of the laser with metals and metal compounds including metal oxides, sulfides, etc., to determine the effect of different bonding energies to the production of the metal atomic beam. We also plan on continuing to study the cross reaction of the laser produced beam with oxygen containing gases by use of the pulsed valve. A optical multichannel analyzer will be added in order to study chemiluminescence from the reaction products formed in an excited state. This spectroscopic data will also yield thermochemical properties of the metal oxides, as well as dynamic information on the reaction.

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