NOTES

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A high-temperature pulsed solenoid valve for supersonic jet introduction up to 550 °C

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A pulsed solenoid valve which can operate up to 550 °C has been designed for supersonic jet introduction. This valve uses a novel design where the nozzle head, which serves as the hot oven, and the solenoid operator are separated and the latter is maintained at a relatively low temperature using water cooling. A long steel plunger forms a metal-to-metal seal at the orifice which is broken when the magnetic field generated by the solenoid pulls back the plunger. Thus, although the oven operates at the high temperatures required for nonvolatile materials, the solenoid operator and electrical parts remain relatively cool.

The supersonic jet technique has become a powerful method for producing internally ultracold molecules for gas phase spectroscopy. In work utilizing pulsed laser sources as the spectroscopic probe, pulsed valves have often been used as a means of also pulsing the molecular beam. The result is a substantial reduction in the duty cycle necessary for pumping compared to a continuous jet and thus a relatively simple pumping station can be used for such jet experiments. A number of pulsed valves have been used in the past based upon solenoid valves,[1-11] piezoelectric devices,[12] or the magnetic repulsion principle.[13-16] Each design has its own particular advantages or disadvantages in terms of its operating parameters including pulse width, repetition rate, and the maximum operational temperature and pressure.

One of the main problems in the design of these pulsed injection sources has been to develop valves compatible with samples that need to be heated to > 150 °C. The design of such valves depends mainly on developing vacuum seals and electrical components that can operate at elevated temperatures. There have been several such valves developed which are described in the literature.[5-9] These valves are generally based on modified solenoid valves or Bosch fuel injectors. Most notable is the valve design of Even and co-workers[9] which uses a glass fiber-insulated coil and Kapton polyimide seals to reach an operating temperature of 470 °C with a pulse width of 200 μs.

In this note, we report the design of a novel high-temperature nozzle that can operate up to 550 °C. The unique feature of this valve is that it separates the pulsed orifice from the solenoid operator which is water cooled. The result is that the nozzle head can be heated to very high temperatures while the electrical components remain relatively cool.

The high-temperature pulsed valve consists of two parts: the nozzle head which contains the high-temperature oven and the nozzle body which contains the solenoid operator. The two components are separated by a water cooling system.

The stainless-steel head of this high-temperature pulsed nozzle functions as a hot oven. The sample is placed into the oven through an $\frac{1}{8}$-in.-diam aperture (see Fig. 1). After the sample has been introduced, this small sample inlet is sealed with a copper gasket by a screw. A gas outlet for the hot oven is present so that the carrier gas can be flowed through continuously while baking the nozzle in order to clean the oven after each experiment. In addition, this forward gas flow prevents clogging of the plunger in the water-cooled region which may occur because of sample condensation. Such condensation may occur due to vapor feedback from the nozzle's hot oven to its cold body. The nozzle head is heated with a thermocoax heater (Omega Engineering, Inc.). The temperature of the nozzle head can be monitored by the use of a Chromel–Alumel thermocouple (Omega Engineering, Inc.). The nozzle head can be heated at least up to 550 °C during operation.

The cylindrical orifice is 800 μm in diameter $\times$ 2 mm in length cut into a stainless-steel plate which is sealed to the nozzle head using a copper gasket and knife edge seal. Variable size orifices can be used with this nozzle to obtain the desired molecular-beam throughput. The connection between the nozzle head and the body is cooled by circulating

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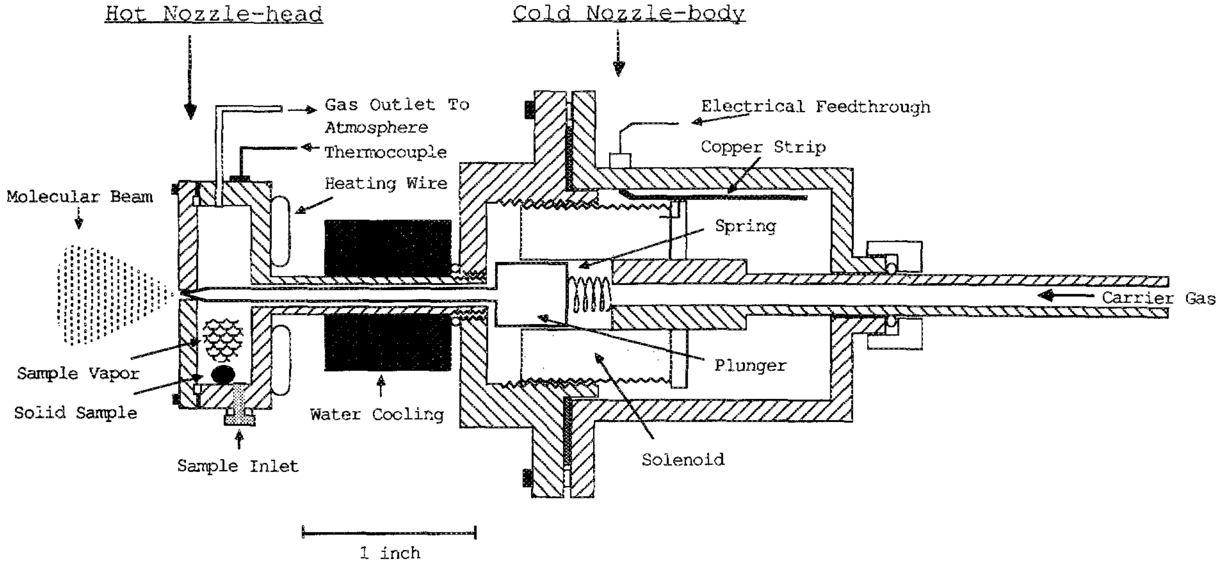

FIG. 1. The design of the high-temperature pulsed nozzle.

tap water as indicated in Fig. 1. The nozzle head is screwed into the nozzle body and a nitrile O-ring (Parker Hannifin Co. No. 2-011) can now be used to seal the joint since the temperature at the joint remains as low as the cooling tap water.

The main components of the nozzle body are the plunger and the plunger driving system (see Fig. 1). The plunger is constructed of a long stainless-steel needle (2.125 in. long and 0.0625 in. in diameter) which screws into a steel base. This needle is guided by the tube which connects the nozzle head and the body so that the plunger needle forms a metal-to-metal seal against the orifice. The leakage from this metal-to-metal seal is negligible compared to the gas emitted per pulse. Most of the plunger remains at room temperature due to the water cooling even when the nozzle head is heated up to 550 °C, although the plunger needle inside the nozzle head does become hot. This important feature allows us to use the solenoid of an automobile fuel injection valve to drive the plunger without substantial modification. A commercial Honda solenoid is used to drive the plunger and a stainless-steel spring is used to hold the plunger against the orifice in order to form the seal. During the operation, the plunger base is attracted by the electromagnetic force generated in the solenoid when energized and the gas is allowed to pass through the orifice. When the solenoid is turned off, the spring returns the plunger to its original position.

The electrical feedthrough used to provide the current is constructed of machinable ceramic insulation that is threaded so that it screws into the stainless-steel body. A stainless-steel rod is epoxied into the ceramic using "Torr Seal" (Varian). A copper strip screwed on the stainless-steel rod is used to contact the lead of the solenoid. The electrical circuit used with this valve has been reported elsewhere.[10] The highest potential available from this circuit is 400 V, although usually about 50 V is used to drive the solenoid.

In Figs. 2(A)–2(C) is shown a series of gas pulse profiles emitted from the high-temperature pulsed nozzle, corresponding to different driver currents at several different temperatures. These pulse profiles are obtained by monitoring the molecular ion signal intensity produced by resonant two-photon ionization (R2PI) in a time-of-flight mass spectrometer, using a boxcar integrator as a function of time delay between the nozzle firing and the ionization laser pulse. In these particular experiments, aniline was seeded into Ar carrier (20 psi) in order to generate the set of pulse profile curves at 25 °C, whereas carbazole and coronene were seeded in Ar in order to generate the profiles at 200 and 450 °C, respectively. These compounds were chosen since they have a sufficient vapor pressure at these temperatures to provide a detectable R2PI signal which reveals the profile of the Ar pulse, but do not provide too much vapor pressure that might saturate the Ar pulse and interfere with the supersonic jet flow. The wavelength of the ionization laser beam was 266 nm where these compounds absorb and ionize readily and the laser power density was $\sim 5 \times 10^5$ W/cm^2. The pulse width of the gas pulse at 37.5-A driver current (profile) is ~ 225 μs FWHM with an ~ 70-μs (10%–90%) rise time and an extended fall-off time of ~ 175 μs. The nozzle pulse reaches a flat top with 130 μs width, indicating that choked flow is reached and thus optimal jet cooling should be achieved. The pulse width can be adjusted for longer pulse operation by adjusting the distance between the solenoid and the orifice. As this distance is increased, the pulse width becomes longer.

The pulse characteristics of the valve appear to be quite repeatable up to an operational temperature of at least 450 °C since the solenoid remains relatively cool. In addition, the electrical conditions needed to drive the circuit do not change substantially as the temperature increases. At each temperature, the pulse profile basically saturates at 37.5 A/pulse and the pulse profile does not change significantly as the current is increased further. The valve has been

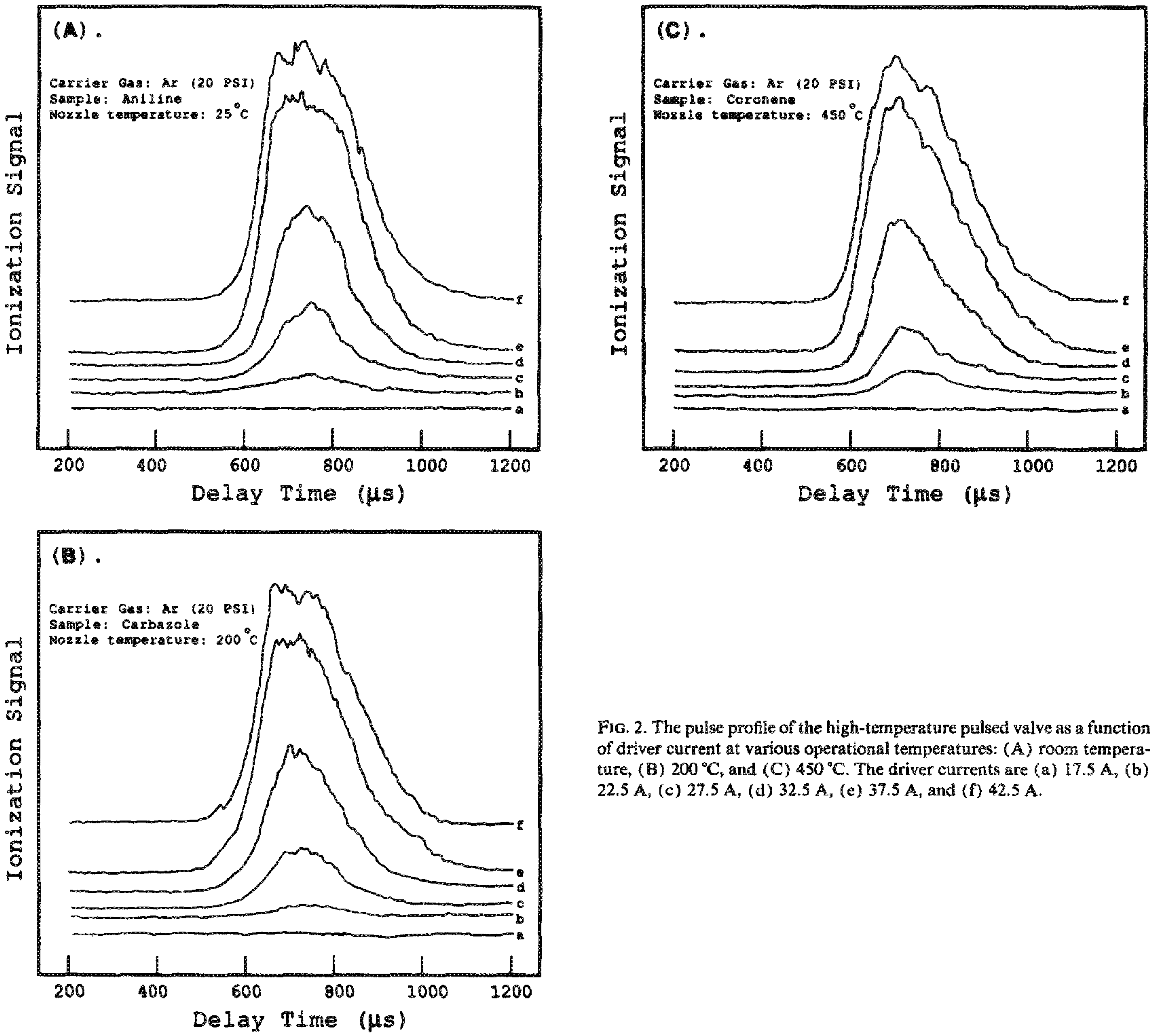

FIG. 2. The pulse profile of the high-temperature pulsed valve as a function of driver current at various operational temperatures: (A) room temperature, (B) 200 °C, and (C) 450 °C. The driver currents are (a) 17.5 A, (b) 22.5 A, (c) 27.5 A, (d) 32.5 A, (e) 37.5 A, and (f) 42.5 A.

operated reliably up to 550 °C for several hours. However, at 550 °C there are few stable organic molecules with which this valve can be tested reliably.

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