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Critical parameter determination of sonic flow controller diamond microtubes and micronozzles

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In this article, the authors measure throughput of sonic diamond microtubes and micronozzles that can work as passive gas flow controllers and flow meters under choking conditions. The behavior of the outlet pressure through the microdevices using an experimental setup with constant volume and constant temperature was determined in order to obtain the critical throughput, the critical mass flow rate, and the discharge coefficients of the diamond sonic microdevices. © *2007 American Vacuum Society.* [DOI: 10.1116/1.2790924]

I. INTRODUCTION

The micromachining technology and flow metrology research area have promoted major advances on flow rate meter fabrication, specially designed to operate under small flow for several technological applications.[1] Classical small restriction elements in the milimetric range such as thin circular orifices and classical Venturi tubes operating in molecular and viscous regimes to measure small flow has been analyzed previously.[2,3] Recent works have shown that micrometric restriction elements such as microtubes and micronozzles operating under choking conditions can be used as passive flow controllers and flow meters because of precise flow control without moving parts, avoiding wear effects and thermal perturbation.[1,4–7] The choking conditions are achieved when the flow velocity in the throat nozzles is equal to the local speed of sound and the mass flow rate becomes constant despite the outlet pressure variation. The critical flow parameters are those related to the choking conditions. In previous works, we showed a method to fabricate diamond sonic microtubes and micronozzles by replication of tungsten molds.[5–7] We discussed in these works that the strategic choice of diamond to fabricate the microdevices is an important step toward achieving properties such as mechanical stability, precise temperature control, usability in corrosive environments, and stability of the opening size and throat diameter for a large temperature range.[8,9] In this article, we determined the behavior of pressure through the microdevices as a function of time taking constant volume and temperature to obtain the throughput, the critical mass flow rate, and the discharge coefficients of the diamond sonic microdevices operating under choking conditions.

The throughput (Q) of gas (PV value of the gas per time) under constant volume and temperature is determined by

$$Q = \frac{d}{dt}(P_{\mathrm{vc}}V_{\mathrm{vc}}) = V_{\mathrm{vc}}\frac{dP_{\mathrm{vc}}(t)}{dt}, \tag{1}$$

where P_{vc} and V_{vc} are the pressure and volume of vacuum chamber discharge.[10,11]

The mass flow rate ($\dot{m}$) (mass per time) using a Boyle-Mariotte approach is determined by

$$\dot{m} = \frac{dm}{dt} = \frac{M}{RT}V_{\mathrm{vc}}\frac{dP_{\mathrm{cv}}(t)}{dt} = \frac{M}{RT}Q, \tag{2}$$

where M is the molar mass, R is the universal gas constant, and T is the absolute temperature of the gas.[10–13]

The discharge coefficient C_d is dimensionless and determined by

$$C_d = \frac{\dot{m}_{\mathrm{expt}}}{\dot{m}_{\mathrm{ideal}}}, \tag{3}$$

where $\dot{m}_{\mathrm{expt}}$ is the experimental mass flow rate and $\dot{m}_{\mathrm{ideal}}$ is the mass flow rate for an ideal gas flux given by

$$\dot{m}_{\mathrm{ideal}} = A^{*}C^{*}\frac{P_0}{\sqrt{\frac{RT_0}{M}}}\ (\mathrm{g/s}), \tag{4}$$

where A^{*} is the throat area, $C^{*}=0.6847$, P_0 and T_0 are the pressure and temperature in the inlet region (stagnation conditions), R is the universal gas constant, and M is the mo-

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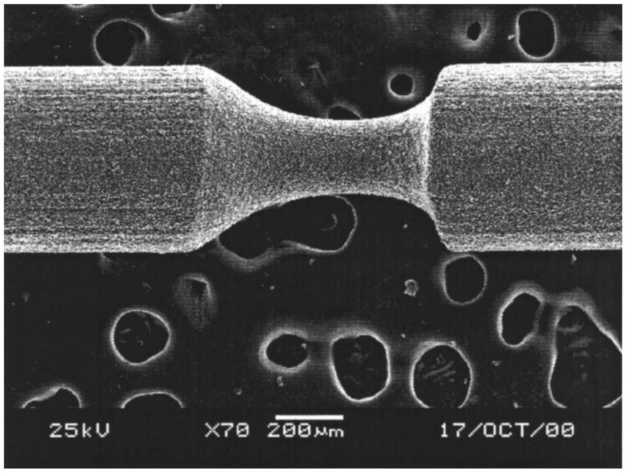

FIG. 1. Scanning electron micrograph of a convergent-divergent shaped tungsten wire coated with a diamond film.

lecular mass, following the ISO 9300 form.[14] A discharge coefficient different from unity indicates that the viscous effects near the walls affect the actual mass flow. For instance, for a smaller than 1 discharge coefficient, mass losses are present in the flow field (friction losses at the walls), when compared to the predicted flow behavior.[12–14]

II. EXPERIMENTAL PROCEDURES

The method used to fabricate diamond sonic microtubes and micronozzles has been described previously.[5–7] One of the diamond sonic micronozzle fabricated and tested in this work is shown in a scanning electron micrograph in Fig. 1. This is a device with a total length of 4810 μm, the convergent-divergent nozzle length is 700 μm, with inlet and outlet diameters (d) of 500 μm and a throat diameter (d^*) of 160 μm. The production of diamond micronozzles was based on the molding process. The fabrication technique begins with a tungsten wire chemically etched in order to obtain the convergent-divergent geometry that is later used as a mold for the diamond coating. The final step is to etch the *sacrificial* tungsten material, leaving the self-standing diamond micronozzles.[5–7] We characterized three tubes and three micronozzles where their geometric parameters can be seen in Table I. Our devices have a flow restriction area with diameters in the range of 1300–160 μm. Device dimensions were measured by scanning electron microscopy of the molds used to fabricate the diamond microdevices.[5–7,15]

TABLE I. Geometric parameters of the microdevices.

Devices	Length of devices (μm)	Length of nozzle (μm)	Inlet or outlet diameter (d) (μm)	Throat diameter (d^*) (μm)
Tube01	47 000	···	1300	···
Tube02	49 000	···	612	···
Tube03	5 000	···	500	···
Nozzle01	4 740	490	500	410
Nozzle02	4 770	760	500	240
Nozzle03	4 810	700	500	160

The experimental setup used to test the microdevices is shown in Fig. 2. The gas used to perform the flow measurements was nitrogen (N_2) and the temperature, the inlet pressure, and the discharge volume were maintained constant during the experiments. Three vacuum chambers were used to perform the experiments. The vacuum chamber CV1 positioned in the inlet region (upstream) was used to control the flow coming from the N_2 gas cylinder and to adjust the inlet pressure. The flow parameters in the inlet region can be considered as in stagnation conditions. In the outlet region (downstream), we have two vacuum chambers, CV2 to perform big flow rates and CV3 to perform small flow rates. There are several shut-off valves along the line to select a convenient flow experiment for each microdevice. The discharge chambers (CV2 and CV3) were pumped by a mechanical vacuum pump, with the final pressure equal 2 $\times 10^{-2}$ torr, and selected shut-off valves were activated to start the gas expansion. Two types of pressure meters (Pirani and Hg column) were used to monitor the gas expansion along the line. The volumes of the discharge chambers CV2 and CV3 were measured by a direct measurement method where the dimensions of the pipeline and pressure meters were considered. The volumes of the chambers are $V_2 = 61.8 \pm 0.4$ l and $V_3 = 2.08 \pm 0.05$ l. The microdevice characterization as flow controllers and flowmeters was performed under choking conditions. Outlet pressures as function of time were measured for each microdevice for three different inlet pressures 0.5, 1, and 1.5 atm. We plotted outlet pressure as function of time to fit and to determine the slope in the constant region of the curve. The linear region of the curve describes the operation in choking conditions. We used the Boyle-Mariotte approach to determine the critical throughput and the critical mass flow rate and we used the ISO 9300

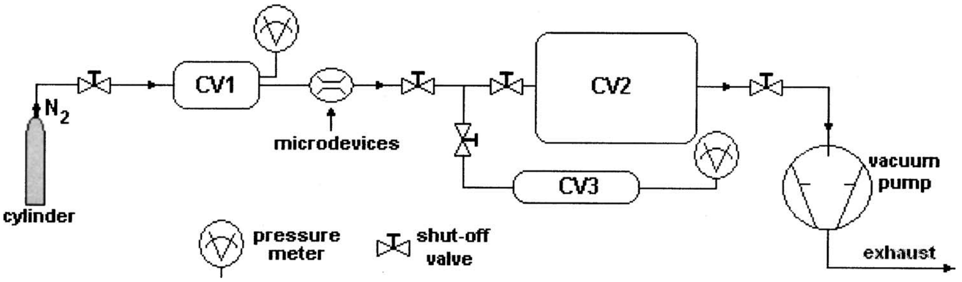

FIG. 2. Experimental gas flow test setup.

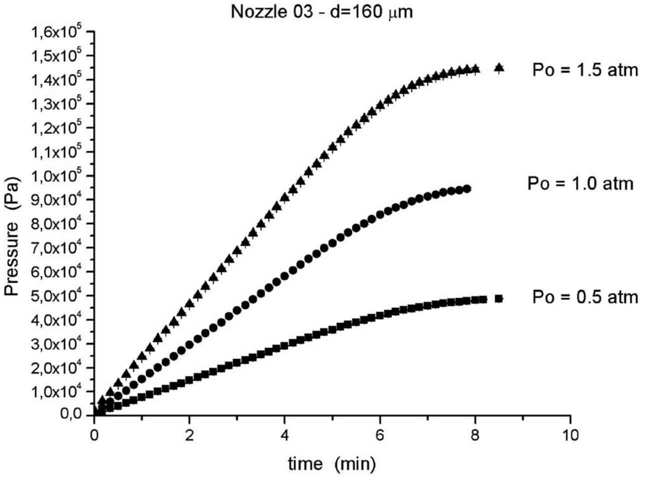

FIG. 3. Outlet pressure as a function of time for the smallest micronozzle ($d = 160\ \mu$m).

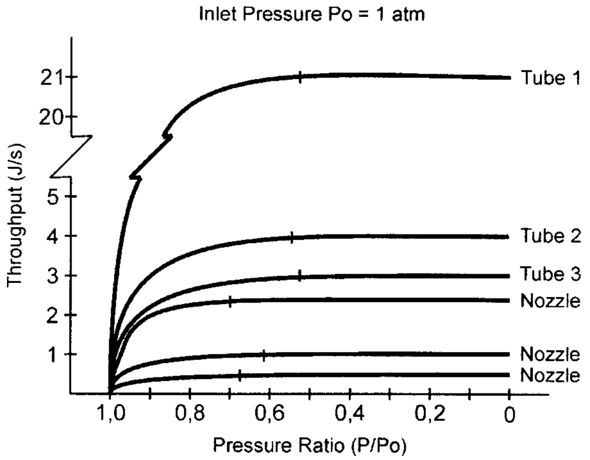

FIG. 4. Throughput as a function of pressure ratio for several microdevices (inlet pressure $P_0 = 1$ atm).

approach to determine the discharge coefficients of each microdevice.[10–14] The Boyle-Mariotte approach is based on an experimental procedure using a vacuum chamber with a constant volume in which the pressure changes with respect to time. This situation can be described by Eq. (1).

The important region of the curves for the application of these microdevices as passive gas flow controllers and flowmeters is the one with constant slope. In this case, the throughput is constant and can be calculated considering the slope of the curve of outlet pressure versus time. With this method, it was possible to determine with good precision the critical throughput and the critical pressure ratio.

III. RESULTS AND DISCUSSION

The behavior of the outlet pressure (P) as a function of time for the smallest micronozzle (Nozzle03, with $d^* = 160\ \mu$m) in the three different conditions of the inlet pressure is shown in Fig. 3. The linear part of the curves shows the operation in choking conditions. We can see that the slope of the curves (source of the choking parameters) increase with the inlet pressure.

The choking conditions for several microdevices can be clearly seen with the average behavior of throughput as a function of pressure ratio (P/P_0) shown in Fig. 4. As we can see, the choking conditions are obeyed for all microdevices and the throughputs increase with the diameter of the microdevices. We fitted the linear part of the curves of outlet pressure for all samples in the three different inlet pressure conditions in order to determine the slope of the curves to calculate the critical flow parameters related to the choking conditions. The critical throughput (Q^*), the critical mass flow rate ($\dot{m}^*$), the critical pressure ratio (P/P_0), and the discharge coefficient (C_d) calculated can be seen in Table II. Note that the critical throughput and the critical mass flow rate decrease with diameter reduction but increase with inlet pressure enhancement. The critical pressure ratios increase with diameter reduction and inlet pressure enhancement, varying from 0.56 (Tube01) to 0.63 (Nozzle03) for the smaller inlet pressure (0.5 atm) and varying from 0.67 (Tube01) to 0.69 (Nozzle03) for the higher inlet pressure (1.5 atm). The discharge coefficients have the same behavior as the critical pressure ratio, varying from 0.772 (Tube01) to 1.23 (Nozzle03) for the smaller inlet pressure (0.5 atm) and varying from 0.830 (Tube01) to 1.32 (Nozzle03) for the higher inlet pressure (1.5 atm). Our experiments were performed with the Reynolds number in the order of magnitude of 10^3.[12–14] The throat geometries of the micronozzles do not obey the ISO 9300 (Ref. 14) and, because of that, our discharge coefficient is not necessarily expected to be close to 1. Our purpose was to determine the experimental parameters of flow choking conditions in order to show that our devices can work as passive gas flow controllers and flowmeters. The studies of the discharge coefficient with the exact geometry samples described in the ISO 9300 can be seen in recent works.[16–19]

IV. CONCLUSIONS

Diamond microtubes and micronozzles have been characterized as flow passive devices using nitrogen gas in a setup with constant volume and temperature. The critical parameters such as critical throughput, critical mass flow rate, and discharge coefficient were determined for all devices. The experimental results showed that the critical throughput and the critical mass flow rate decrease with the diameter reduction but increase with the inlet pressure enhancement. The critical pressure ratios increase with diameter reduction and inlet pressure enhancement. The discharge coefficients have the same behavior as the critical pressure ratio, varying from 0.772 (Tube01) to 1.23 (Nozzle03) for the smaller inlet pressure (0.5 atm) and varying from 0.830 (Tube01) to 1.32 (Nozzle03) for the higher inlet pressure (1.5 atm). These results show that the diamond microtubes and micronozzles can work as flow controllers and flowmeters under choking conditions.

TABLE II. Critical flow parameters.

Devices	Inlet pressure P_0 (atm)	Critical throughput Q^* (J/s)	Critical mass flow rate $\dot{m}^*$ $[\times 10^{-3}$ (g/s)$]$	Critical pressure ratio P/P_0	Discharge coefficient C_d
Tube01	0.5	10.0±0.3	113±4	0.56±0.03	0.772±0.019
Tube02	0.5	1.69±0.07	19.0±0.9	0.525±0.016	0.54±0.03
Tube03	0.5	1.61±0.05	18.0±0.6	0.59±0.03	0.63±0.04
Nozzle01	0.5	1.26±0.03	14.1±0.4	0.64±0.03	0.97±0.06
Nozzle02	0.5	0.497±0.012	5.56±0.17	0.62±0.03	1.1±0.1
Nozzle03	0.5	0.247±0.010	2.76±0.12	0.63±0.03	1.23±0.16
Tube01	1.0	21.2±0.7	241±9	0.52±0.03	0.84±0.04
Tube02	1.0	4.15±0.13	47.1±1.8	0.54±0.03	0.74±0.04
Tube03	1.0	3.20±0.10	36.3±1.3	0.524±0.016	0.85±0.04
Nozzle01	1.0	2.51±0.10	28.2±1.2	0.71±0.06	0.99±0.07
Nozzle02	1.0	0.98±0.03	10.9±0.4	0.64±0.04	1.1±0.1
Nozzle03	1.0	0.495±0.014	5.59±0.19	0.68±0.03	1.29±0.17
Tube01	1.5	31.0±1.0	352±13	0.67±0.06	0.83±0.03
Tube02	1.5	6.5±0.2	74±3	0.53±0.02	0.77±0.04
Tube03	1.5	5.00±0.10	56.3±1.6	0.59±0.04	0.87±0.03
Nozzle01	1.5	3.83±0.15	42.9±1.8	0.624±0.019	1.01±0.07
Nozzle02	1.5	1.49±0.10	16.7±1.1	0.69±0.04	1.14±0.12
Nozzle03	1.5	0.763±0.019	8.6±0.3	0.69±0.03	1.32±0.17

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[1] C.-M. Ho and Y.-C. Tai, Annu. Rev. Fluid Mech. **30**, 579 (1998).
[2] W. Jitschin, M. Ronzheimer, and S. Khodabakhshi, Vacuum **53**, 181 (1999).
[3] W. Jitschin, Vacuum **76**, 89 (2004).
[4] S. S. Mammana, M. C. Salvadori, K. Kawakita, M. T. Pereira, and M. Cattani, J. Vac. Sci. Technol. B **21**, 2034 (2003).
[5] B. T. Arnberg, ASME J. Basic Eng. **84**, 447 (1962).
[6] S. Silva, M. C. Salvadori, K. Kawakita, M. T. Pereira, W. Rossi, and M. Cattani, Diamond Relat. Mater. **11**, 237 (2002).
[7] S. Silva, M. C. Salvadori, K. Kawakita, M. T. Pereira, R. M. Galvão, and M. Cattani, J. Micromech. Microeng. **12**, 108 (2002).
[8] M. Seal, *Applications of Diamond Films and Related Materials: Proceedings of the Third International Conference*, NIST Special Publication 885, Washington, 1995, pp. 3–10.
[9] P. W. May, Philos. Trans. R. Soc. London, Ser. A **358**, 473 (2000).
[10] *Foundations of Vacuum Science and Technology*, edited by J. M. Lafferty (Wiley, New York, 1998).
[11] A. Berman, *Total Pressure Measurements in Vacuum Technology* (Academic, Orlando, FL, 1985).
[12] R. W. Fox and A. T. McDonald, *Introduction to Fluid Mechanics* (Wiley, New York, 1981).
[13] J. D. Anderson, Jr., *Modern Compressive Flow* (McGraw-Hill, New York, 1990).
[14] ISO9300, "Measurement of gas flow by means of critical flow Venturi nozzles." International Organization for Standardization (www.iso.org-ICS:17.120.10-ISO9300:2005), Switzerland, 2005.
[15] M. C. Salvadori, V. P. Mammana, O. G. Martins, and F. T. Degasperi, Plasma Sources Sci. Technol. **4**, 489 (1995).
[16] N. Bignell and M. Takamoto, Flow Meas. Instrum. **11**, 255 (2000).
[17] M. Hayakawa, Y. Ina, Y. Yokoi, M. Takamoto, and S. Nakao, Flow Meas. Instrum. **11**, 279 (2000).
[18] M. Ishibashi and M. Takamoto, Flow Meas. Instrum. **11**, 305 (2000).
[19] M. Ishibashi and M. Takamoto, Flow Meas. Instrum. **11**, 293 (2000).