Paper

Development of an Anti-Corrosive Integrated Mass Flow Controller

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The development of an anti-corrosive integrated mass flow controller made by micromachining is described. Stainless steel (SUS316, SUS316L) was used as a material to prevent corrosion. The mass flow controller consists of a thermal flow sensor and a flow control valve driven by a unimorph actuator. The valve could control the flow rate from 0 to 90 SCCM at 75 kPa of N2 gas when the voltage of the piezoelectric disk was varied from 75 V to –75 V.

Keywords: Anti-Corrosive, Mass Flow Controller, Piezoelectric Disk, Flow Sensor

1. Introduction

It is required to control flow of reactive gas in advanced semiconductor process such as MOCVD, MOMBE, ALE and RIE etc. Flow control systems that contain mass flow controllers have been used for such processes. However, the dead volume in the gas tube line as well as the slow response of the usual mass flow controllers prevents a rapid change of gas flow [1].

Integrated mass flow controller fabricated by silicon micromachining has been developed [2]. By integration of a flow control valve and a flow sensor, not only dead volume was greatly reduced but also the response to gas flow was improved. In order to remove the absorbed water to prevent the deposition of the reaction product inside the mass flow controller, a bakable type has also been reported [3]. The integrated mass flow controller can be attached directly to the chamber for semiconductor processes to minimize the dead volume. Recently, MEMS mass flow controller made of silicon called "Fluistor" has been developed and manufactured [4]. The MEMS mass flow controller is composed of a thermo-pneumatic valve and a micro flow sensor. Dead volume is reduced by integration of flow control systems but the response of gas flow is inadequate because of using the thermo-pneumatic valve. Furthermore, the MEMS mass flow controller is not available for fluoride gases that corrode silicon. In order to use the integrated mass flow controller for fluoride gases, an anti-corrosive material must be chosen for its material.

This paper describes the development of an anti-corrosive integrated mass flow controller made of stainless steel for advanced semiconductor process.

2. Design

Figure 1 illustrates the structure of the anti-corrosive mass flow controller. Stainless steel (SUS316, SUS316L) is used as a material to prevent corrosion. The structure has a flow control valve driven by a unimorph actuator and a thermal flow sensor. This mass flow controller can control gas flow with high speed and high accuracy because both the flow control valve and the flow sensor can respond quickly.

The structure is composed of three parts of stainless steel. A flow channel, the groove for a gasket and the nozzle of a valve, which are made by using mechanical milling, are

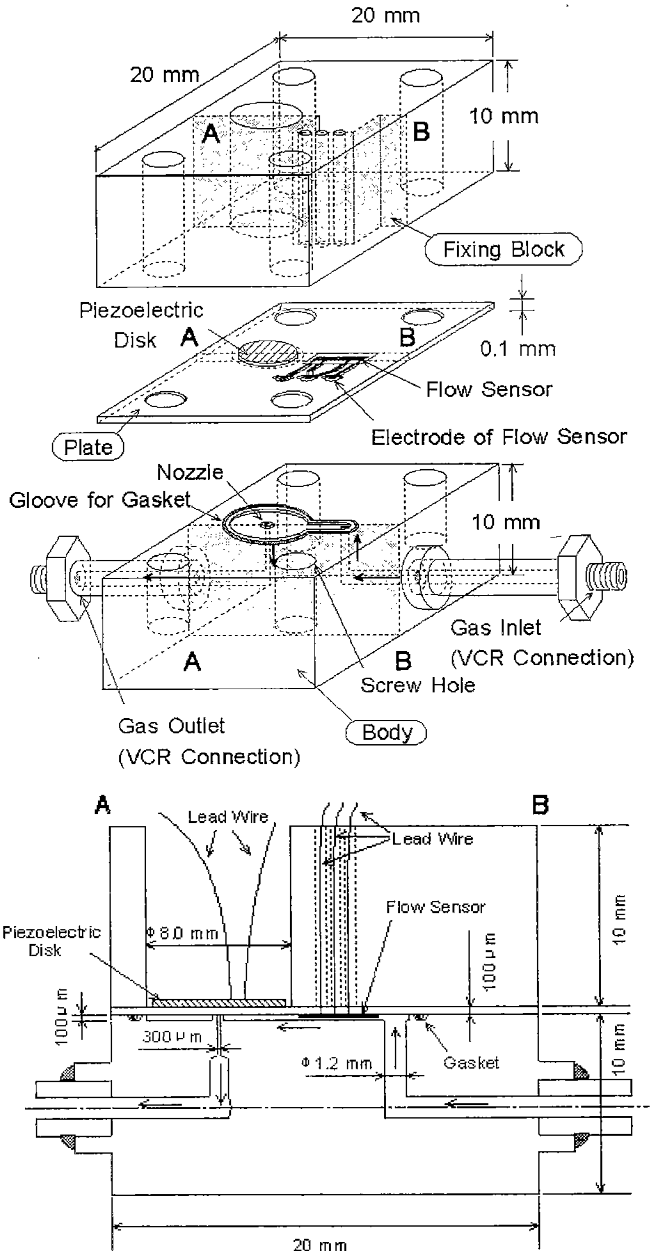

Figure 1 The structure of the anti-corrosive integrated mass flow controller

formed in the "body". The flow control valve and a micro flow sensor made by using micromachining are formed in the "plate". The "fixing block" is used for tightening the plate to the body. This block has holes for taking out lead wires from the flow sensor and the piezoelectric disk.

The "fixing block" and the "plate" are screwed to the "body" using four bolts. The flow channel between the "body" and the "plate" is sealed with the gasket that is placed in 85 μm deep groove on the "body". The gasket is made of a 100 μm thick gold plate because gold is very deformable by mechanical force.

2.1 Valve

Fugure 2 shows the schematic diagram of the flow control valve. The piezoelectric disk ((diameter 6 mm, thickness 0.1 mm) C-91, Fuji Ceramics Corp.) is bonded on the stainless steel plate, that is, an unimorph actuator is formed as the flow control valve. This valve responds with high speed and high accuracy compared with a thermo-pneumatic valve [4]. Epoxy resin was used for bonding the piezoelectric ceramics because it was very easy to handle. If the valve is used at high temperature, it is better to replace the epoxy resin with polyimide or ceramic adhesive.

The unimorph actuator shown in Fig.3 was fabricated in order to evaluate the displacement of the valve by using laser Doppler vibrometer (MLD-102 · MLD-701, NIHON KAGAKU ENGINEERING Corp.). The actuator was same size as the designed valve. Figure 4 indicates that the actuator was displaced from –12 μm to 8 μm when the voltage of the piezoelectric disk was varied from –75 V to 75 V. The displacement is sufficient to control gas flow.

2.2 Flow Sensor

Schematic diagram of the flow sensor is shown in Fig. 5. The sensor has 0.1 μm thick Ni heaters that can be also used as a temperature sensor. The heaters are sandwiched between 5 μm thick SiO2 layers being suspended as a bridge [5]. The sensor forms a differential pair to achieve high sensitivity. The structure is located in a gas flow channel to provide a reproducible flow response [6]. The stainless steel islands, which is isolated from the stainless steel "plate", are used as electrical terminals for taking out the signal of the sensor.

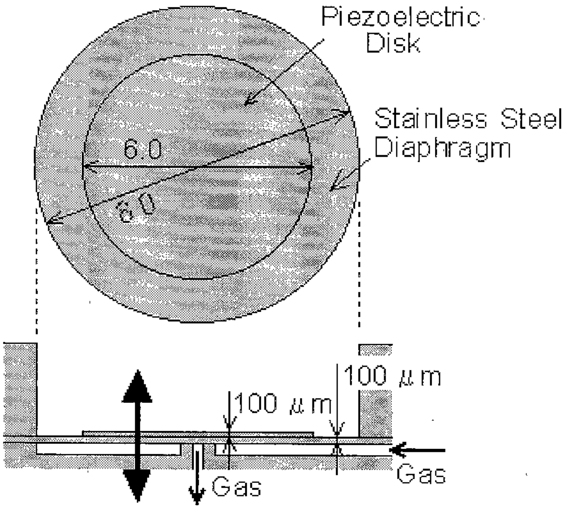

Figure 2 Schematic diagram of the valve

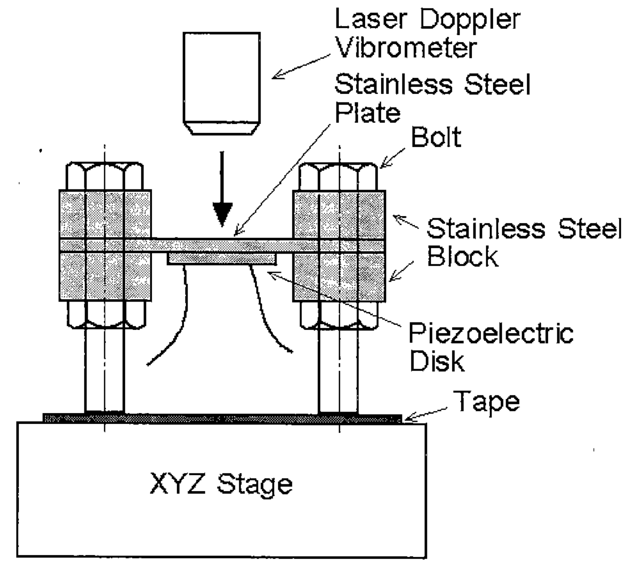

Figure 3 Measurement system of the unimorph actuator

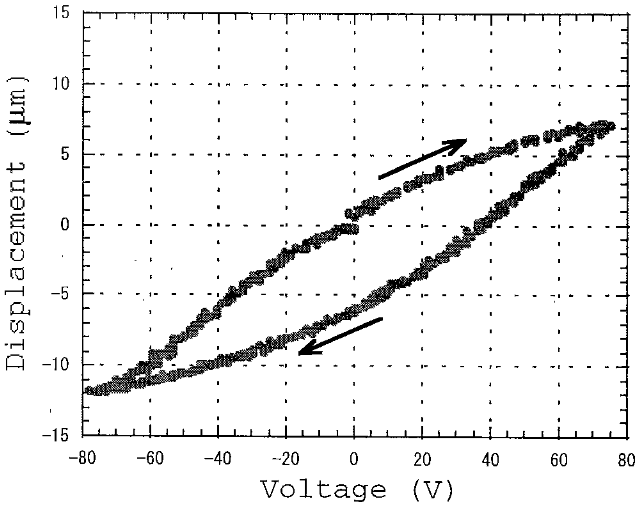

Figure 4 Displacement of the unimorph actuator

3. Fabrication

3.1 Flow Sensor

Figure 6 indicates the fabrication process of the flow sensor. SiO2 film deposited by TEOS-CVD (Tetra Ethoxy Silane - Chemical Vapor Deposition) forms the bridge. TEOS-CVD can deposit the thick SiO2 film of high quality at low temperature, so it is suitable for depositing SiO2 film on the stainless steel that has large thermal expansion coefficient. Ni heaters made by sputterdeposition are sandwiched between the SiO2 films. The stainless steel islands were formed by etching the stainless steel "plate".

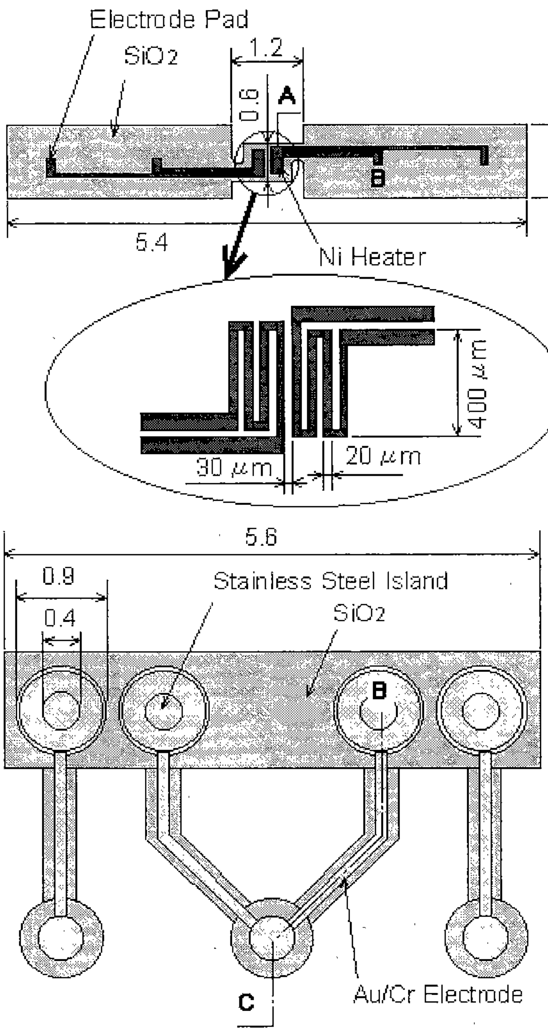

Figure 5 Schematic diagram of the flow sensor (Top: Heater, Bottom: Terminals)

Finally the sacrificial Si layer was removed by XeF_2 and the flow sensor was separated to the stainless steel "plate" in order to reduce the heat capacity of the flow sensor.

① One side polished stainless "plate" (SUS316L) 100 μm in thickness was used.
② Si was sputterdeposited and etched by CF_4 plasma etching.
③ SiO_2 film was deposited by plasma TEOS-CVD and contact holes were patterned by buffered HF.
④ Ni film was sputterdeposited and heater pattern was formed by etching in HNO_3.
⑤ SiO_2 film was deposited by plasma TEOS-CVD.
⑥ Au/Cr was sputterdeposited as a seed layer for electroplating. After photoresist was patterned, Ni film was deposited by electroplating for etching mask.
⑦ Vertical etching through the SiO_2 film was made by CCP-RIE (Capacitively Coupled Plasma-Reactive Ion Etching). Ni and Au/Cr were removed after the etching.
⑧ SiO_2 film was patterned with buffered HF.
⑨ The stainless steel "plate" was etched using $FeCl_3$ as

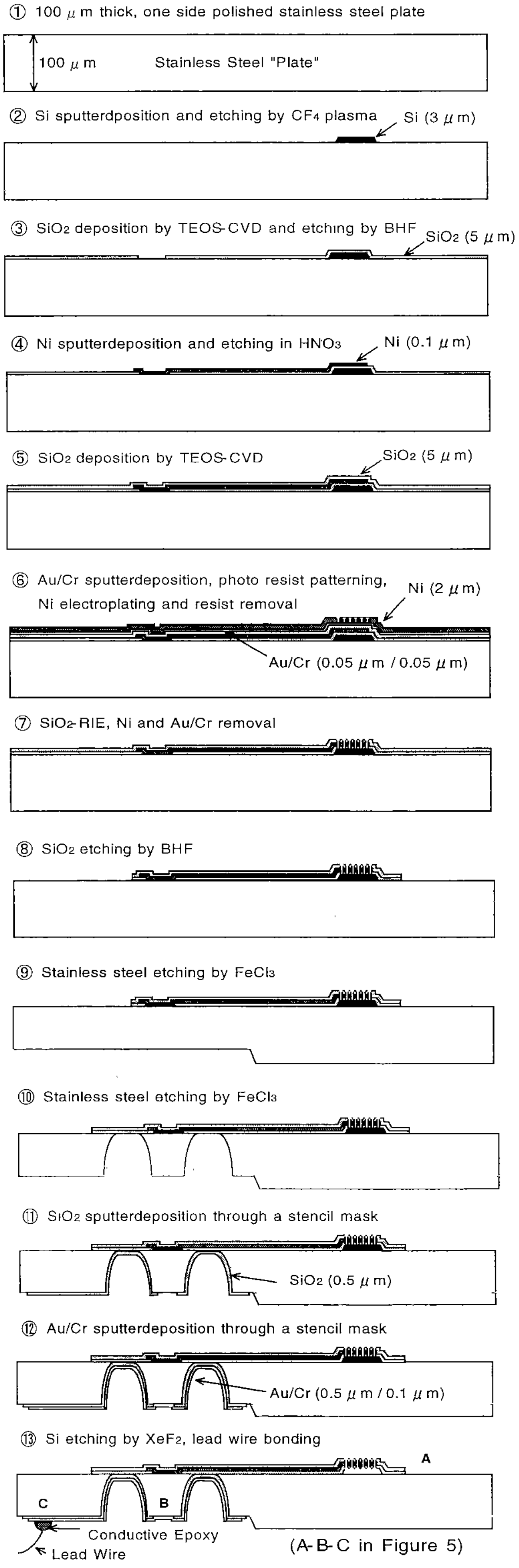

Figure 6 Fabrication process of the flow sensor

an etchant to form 5 μm gap.

⑩ Penetrating etching through the stainless steel "plate" was made by $FeCl_3$.
⑪ SiO_2 film was sputterdeposited through a stencil mask.
⑫ Au/Cr films were sputterdeposited through a stencil mask.
⑬ Sacrificial Si layer was removed by XeF_2 and finally lead wires were bonded.

Figure 7 shows the fabricated flow sensor. It was confirmed that the SiO_2 bridge structure and the stainless steel islands could be made.

3.2 Gold Gasket

The fabrication process of the gold gasket is shown in Fig.8.

① Gold plate 100 μm in thickness was used.
② Cr was sputterdeposited and patterned by using Cr etchant.
③ Gold plate was etched by using Au etchant.
④ Photoresist and Cr were removed.

Figure 9 shows the fabricated gold gasket. In order to deform the gasket easily, the width of the gasket was made as narrow as possible.

4. Characteristics

Figure 10 shows the photograph of the fabricated

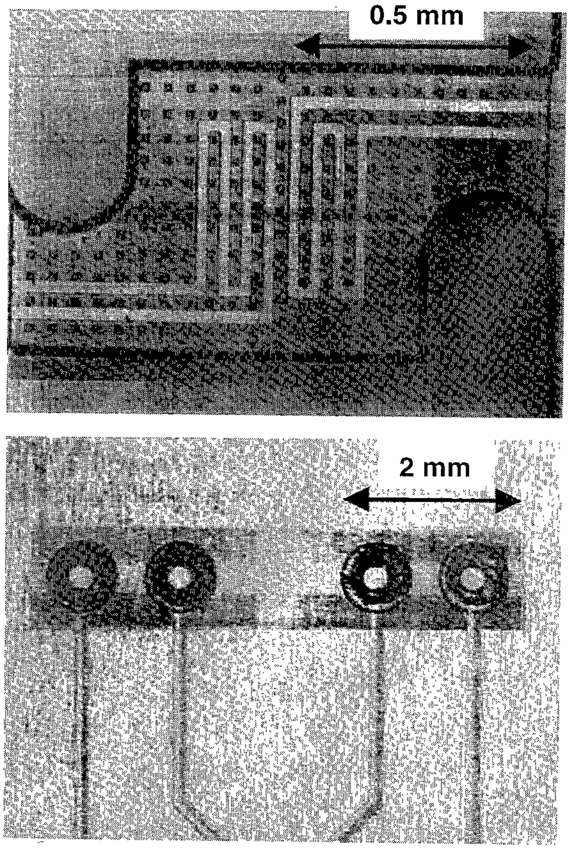

Figure 7 Photograph of the flow sensor (Top:SiO_2 bridge, Bottom:Terminals)

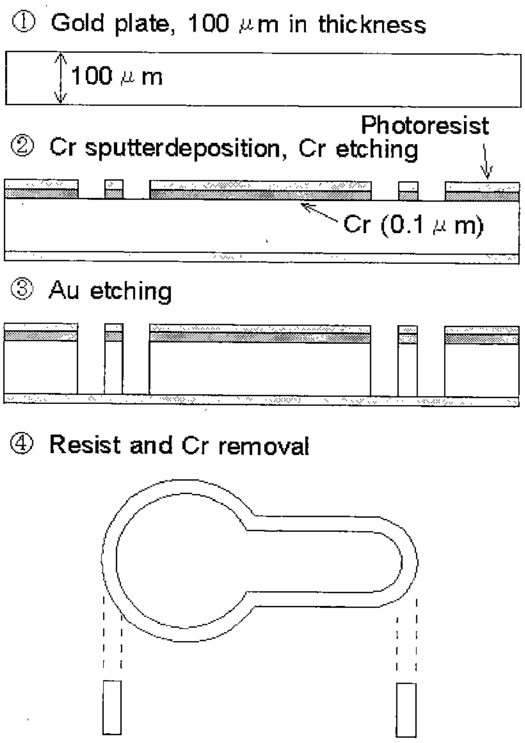

Figure 8 Fabrication Process of the gold gasket

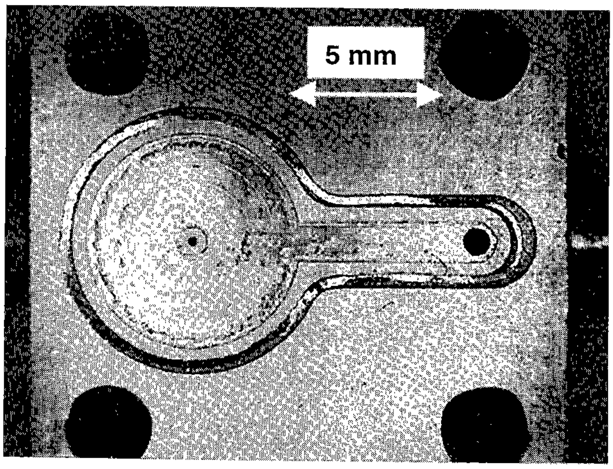

Figure 9 Photograph of the gold gasket in the groove of the stainless steel "body"

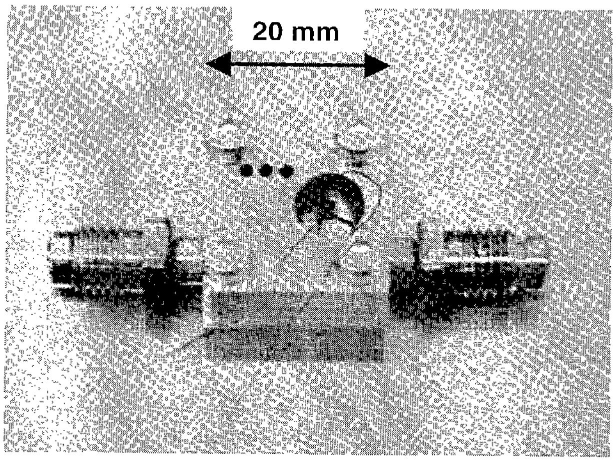

Figure 10 Fabricated controller

controller. The leakage through the gold gasket to the outside the controller was less than 10^{-7} atm·cc/sec. The result shows that the fabricated gold gasket had good property of hermetic sealing. In order to measure the flow characteristics of the fabricated controller, the measurement system was made as shown in Fig.11. The flow characteristics of the valve is Fig.12. This result shows that the flow rate could be controlled from 0 to 90 SCCM at 75 kPa of N2 gas when the voltage of the piezoelectric disk was varied from 75 V to –75 V. Although large hysteresis was found in Fig. 12, this phenomenon seemed to be the characteristics of the piezoelectric disk. The hysteresis is not a serious problem because the gas flow can be controlled by a closed loop system using the flow sensor. The leakage through a valve seat was measured as shown in Figure 13. First the small chamber was evacuated by a rotary pump. Next the stop valve V3 was closed and the increasing pressure of the chamber δP was measured using pressure gauge. The leakage could be calculated as follows [3].

$$(Leakage) = \frac{\delta P \times (Chamber\,Volume)}{(Time)}$$

Figure 14 indicates that the leak rate of the valve seat was about 0.1 SCCM. The leakage can be reduced by polishing the stainless steel plate.

5. Conclusions

We have developed a micromachined anti-corrosive integrated mass flow controller using stainless steel as a material for advanced semiconductor process. The fabricated device could be controlled small amount of gas. Although the flow sensor could not be operated this time, it is the next step to characterize the flow sensor. The future work is to characterize the flow control system and to apply to practical use.

Acknowledgements

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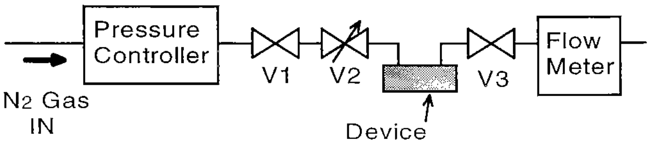

Figure 11 Measurement system of the flow characteristics

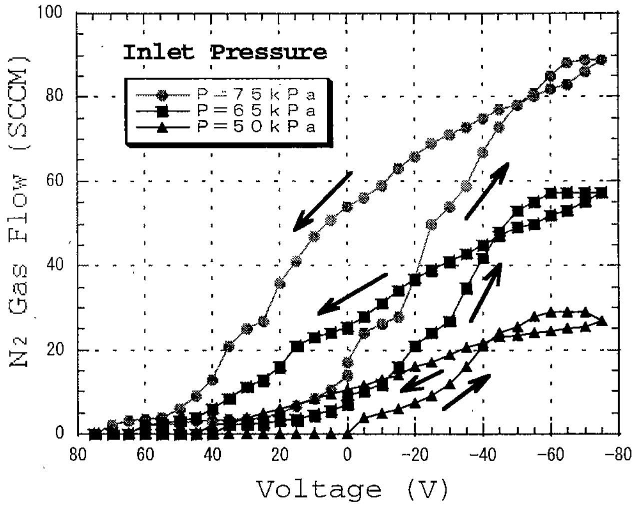

Figure 12 Flow characteristics of the valve

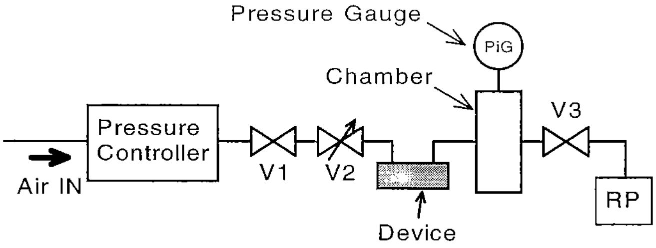

Figure 13 Measurement system of the leakage through a valve seat

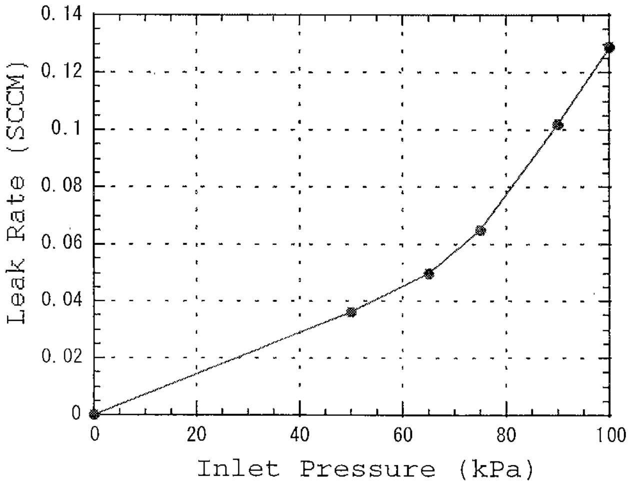

Figure 14 Leak rate through the valve seat

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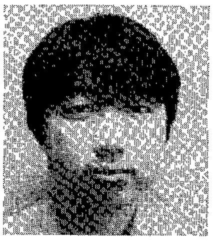

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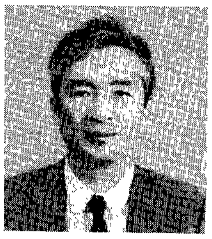

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