Fabrication of High-Density Electrical Feed-Throughs by Deep-Reactive-Ion Etching of Pyrex Glass

Xinghua Li, Takashi Abe, Yongxun Liu, and Masayoshi Esashi

***Abstract*—This paper describes the fabrication technology for high-density electrical feed-throughs in Pyrex glass wafers. Small through holes (40–80 μm in diameter) in Pyrex glass wafers have been fabricated using deep-reactive-ion etching (DRIE) in a sulfur hexafluoride (SF_6) plasma. The maximum aspect ratios obtained were between 5 and 7 for a hole pattern and 10 for a trench pattern. Through the wafer etching of a hole pattern of 50 μm diameter was carried out using 150-μm-thick Pyrex glass wafers. The electrical feed-throughs in the wafers were fabricated by filling the through-holes with electroplated nickel. We were able to successfully bond the glass wafer to silicon by anodic bonding after removing the electroplated nickel on the surface of the wafer by chemical–mechanical polishing (CMP). The electric resistance of the feed-through was estimated by a 4 point wire sensing method to be about 40 mΩ per hole. The heat cycles test shows that the resistance changes were within 3% after 100 cycles. The fabrication of high density electrical feed-throughs is one of the key processes in the field of MEMS. Probable applications of this technology are in electrical feed-throughs between logic elements and microprobe arrays for high-density data storage and for packaged devices. [877]**

***Index Terms*—Chemical–mechanical polishing (CMP), deep-reactive-ion etching (DRIE), high-density electrical feed-throughs, Pyrex glass.**

I. Introduction

PYREX glass is widely used in the field of MEMS because it can be bonded to silicon by anodic bonding. The microfabrication of Pyrex glass is one of the key processes in MEMS. However, it is difficult to achieve fine (<50 μm in size) and high aspect ratio (>10) structures with regular etching methods such as wet chemical etching [1], electrochemical discharge drilling [2], ultrasonic drilling [3], or powder blasting [4]. Only by laser drilling [5] can fine electrical feed-throughs less than 100 μm in diameter be realized. However, this technology is not suitable for batch fabrication because it takes too long to process. Recently, deep-reactive-ion etching (DRIE) of Pyrex glass has been developed in our laboratory [6], [7]. There are many problems in the RIE of materials like glass, which contains atoms such as lead, sodium and aluminum. The etched surface is protected by nonvolatile halogen compounds produced during etching (NaF, AlF_3, etc). Thus the etching characteristics depend on the composition of glass. Pyrex glass has a relatively small mass ratio (6.3 wt%) of these components (Na_2O, Al_2O_3). In our laboratory, etching was carried out using a magnetically enhanced inductively coupled plasma RIE system [8]. According to our previous study, physical etching conditions such as a high self-bias voltage (−390 V) and a low pressure (0.2 Pa) are required to etch glass. For glass etching, the removal of nonvolatile products on the surface is important to obtain high etch rates and high aspect ratio structures. A vertical etch profile was achieved when the mask opening was small (20–30 μm for the trench pattern) because the deposition of nonvolatile products on the side-wall was reduced. Small holes having high aspect ratios and batch fabrication were realized using DRIE technology.

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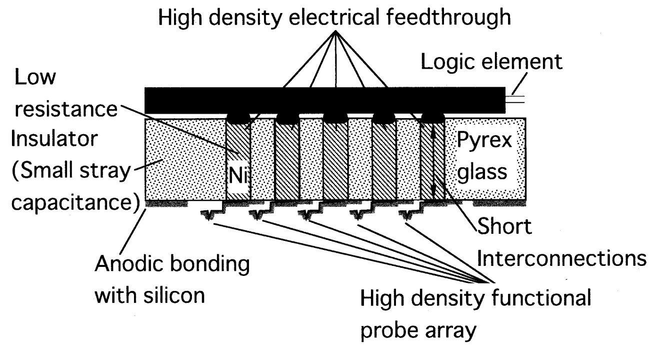

Fig. 1. Schematic illustration of an electrical feed-through from microprobe array.

In this paper, we applied the DRIE of Pyrex glass to the fabrication of high density electrical feed-throughs. A fabrication technology for high density electrical feed-throughs for integrated sensors is extremely desirable [9]. As the number of electrical connections increases, the interconnection between logic elements and individual sensors becomes complex owing to geometrical limitations. For example, consider the fabrication of 10 000 electrode pads/cm^2 for a microprobe array. Neither two-dimensional wiring nor regular etching techniques are applicable to the fabrication. The fabrication of high density electrical feed-throughs in Pyrex glass is one solution to overcome such limitations. Our technology based on DRIE also reduces the complexity of the process. Fig. 1 represents the proposed electrical feed-through structure from a microprobe array to a logic element. As indicated in the figure, there is good process compatibility between the technology and the probe-based technology using silicon micromachining, because it is easy to bond silicon to Pyrex glass. Short interconnections between the logic elements and the high density microprobe array are also realized by this method. Compared with a high-density electrical feed-through fabricated in silicon, the electrical feed-through proposed in this study has advantages such as small leakage

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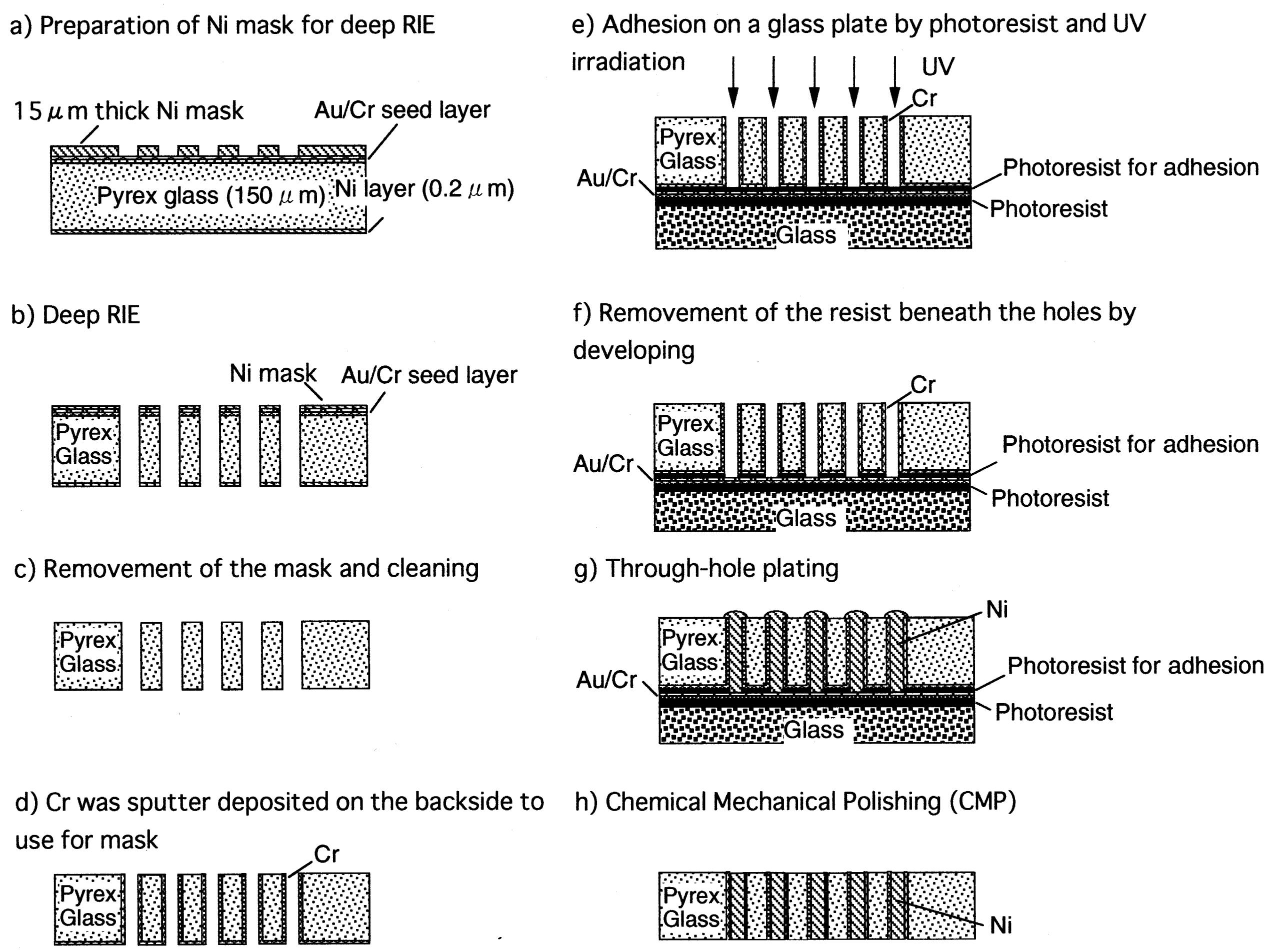

Fig. 2. Process steps for fabricating Pyrex glass for high-density electrical feed-throughs.

current and small stray capacitance due to the use of glass as the insulator. These features are indispensable for a high frequency-electronic system.

II. Experimental

The Pyrex glass wafers used for the fabrication of high-density electrical feed-throughs were 150 μm in thickness. These glass wafers (Asahi Techno Glass Corporation) used in this experiment had the composition, in terms of mass ratio, of $SiO_2 : B_2O_3 : Al_2O_3 : Na_2O = 81.0 : 12.7 : 2.3 : 4.0$. The glass wafers were obtained by wet chemical etching in an aqueous solution of HF (16 vol.%) and ethanol (0.5 vol.%). The addition of a surfactant (ethanol) to remove bubbles and particles from the surface resulted in a smooth etched surface.

The process flow for the fabrication of Pyrex glass wafers with electrical feed-throughs is shown in Fig. 2. A thick nickel-film (15 μm) was selectively electroplated on the exposed part of Au/Cr seed layer using thick photoresist (PMER, Tokyo Ohka Kogyo Co., Ltd.) as a mold (a). The DRIE was carried out at a pressure of 0.2 Pa, with a self-bias voltage of -390 V and a stage temperature of 293 K using a DRIE system developed in our laboratory (b). The details of this process have been described in previous papers [6], [7]. After the nickel mask and the Au–Cr were removed (c), Cr was deposited on the Pyrex glass wafer by sputtering from the backside. The Cr functions as a photo mask, and it also plays an important role in the adhesion of metal in the through holes (d). Positive photoresist was used to stick the Pyrex glass wafer to a Au–Cr seed layer deposited on a glass plate (e). It should be noted that the glass plate was covered by positive resist prior to the deposition of seed layer. By exposing the plate, the photoresist at the bottom of the holes was removed by developing (f). Then the holes were filled with nickel by periodic reverse plating [5] (g). In this process, a nickel sulfamate bath was used. The pH and the temperature of the electrolyte bath were controlled to be about 4 and 55 °C, respectively. An aspirator was used to remove air bubbles inside the through holes. Finally, the glass plate was removed by dissolving the photoresist in acetone and the glass wafer was polished by chemical–mechanical polishing (CMP) to obtain a flat surface for photolithography (h). The polishing presented in this work was carried out using a commercially available polishing and lapping system (MA-200, Musashino Electronic. Co., Ltd.). The glass wafer was placed in a holder with the wafer surface in contact with a polishing pad-covered table. The rotation speed was controlled to be 60 r.p.m. The pressure applied at the interface between the wafer and polishing pad was about 0.16 kg/cm^2. Commercially available polishing slurry (Micropolish II, Buehler) was used for the CMP process. The particle size was 0.3 μm in diameter.

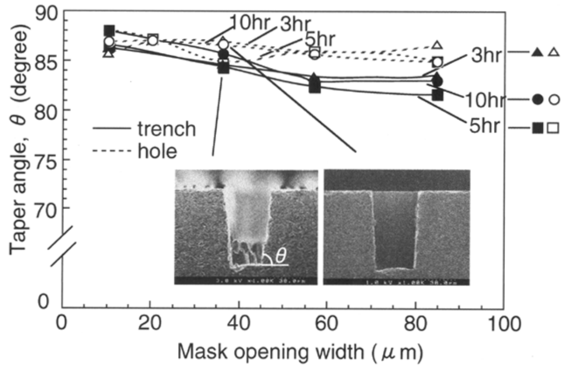

Fig. 3. The tapered angles as a function of mask opening width for trench pattern and for hole pattern. The SEM photographs obtained after 3 h-etching were also shown in the figure.

Passivation of the nickel surface occurred first by the addition of a chemical constituent (H_2O_2, 5.0 vol.%) to the slurry. The passivating film was removed by the mechanical action of the slurry.

All chemicals used in this study were used without further purification. The water used was filtered through a Millipore filter system and had a resistance greater than 18 MΩcm.

III. Results and Discussion

A. Etching Characteristics of Pyrex Glass

The characteristics of the DRIE of Pyrex glass were studied prior to fabrication of the electrical feed-throughs. The dependence of the etching characteristics on pressure, self-bias voltage and temperature in a SF_6 plasma have already been reported in previous papers [6], [7]. In this study, the DRIE was carried out under a pressure of 0.2 Pa, a self-bias voltage of −390 V and a stage temperature of 293 K. Fig. 3 shows the etch profile as a function of the width of the mask opening. The data shown in the figure were obtained for 3, 5 and 10 h of RIE, respectively. The mask patterns had features for trenches and holes. The figure reveals that the slope of the trenches is more tapered compared with that of the holes. The side-wall of hole pattern is twice as large as that of the trench pattern. This result supports our previous study in which the taper of the etched profile depends on the ratio of the area of the side-wall to the volume of the etched glass [6], [7]. Therefore, the amount of nonvolatile products deposited on the side-wall is small in the case of the hole pattern.

Fig. 4 shows the etched depth for holes and trenches as a function of etching time. Interestingly, the etching of hole patterns shows a high etch rate compared with that of trench patterns. However, the etch depth of small hole-patterns became constant (the highest aspect ratio is about 5–7). On the other hands, the effect is small in the case of trench patterns (the highest aspect ratio is about 10). This is due to the deposition of sputtered products from the side-wall. These holes are refilled due to the deposition. The etching is disturbed by nonvolatile products, which are trapped inside the small hole. These effects are small in the case of trench patterns because of the geometry. The decrease of the etched depth for 10 μm trenches at 10 h is due to the mask erosion. This plot also indicates that a 40–80 μm diameter hole is suitable for etching through the glass wafer (150 μm in thickness). To show the difference clearly, the etched depth after 7 hours etching as a function of the width of the mask opening is shown in Fig. 5. As the reference, the etched depth obtained for fused silica glass was also shown in this figure. Interestingly,

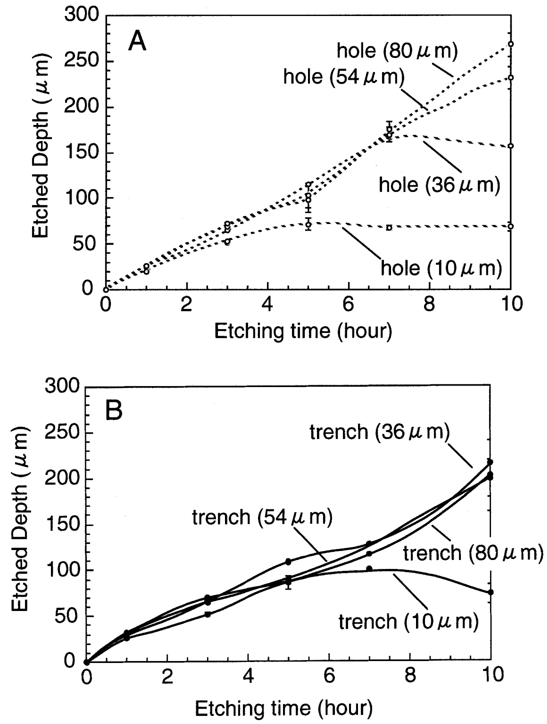

Fig. 4. The etched depth as a function of etching time for (A) hole pattern and for (B) trench pattern.

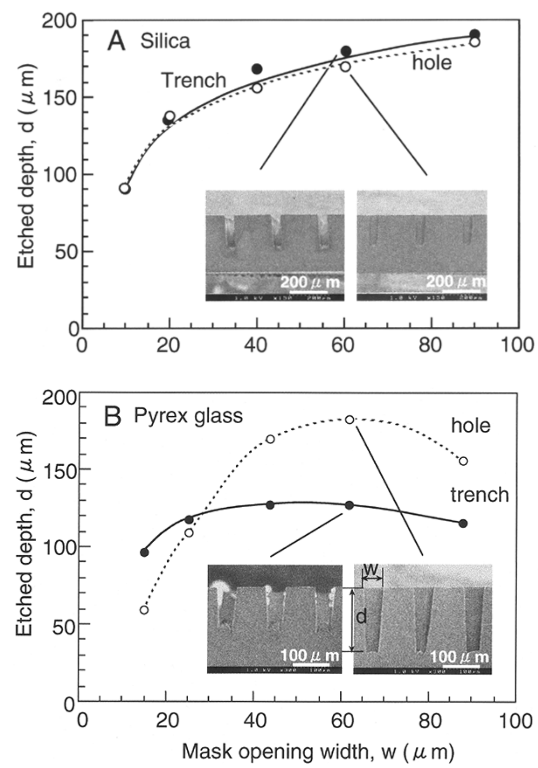

Fig. 5. The etched depth as a function of mask opening width for trench pattern and for hole pattern at 7 h-etching (A: Silica glass, B: Pyrex glass).

there is no clear dependence on the geometry of mask pattern in case of the silica glass etching. It can be concluded that the nonvolatile products such as NaF and AlF_3 cause the unique etching characteristics of Pyrex glass depending on the mask opening width and on the pattern features.

B. Fabrication of Pyrex Glass for High Density Electrical Feed-Throughs

Through the wafer etching was carried out with hole patterns of 50 μm in diameter. The thickness of the Pyrex glass used in this work was 150 μm. The etching time for the wafers was 10 hours. The through-holes before electroplating are shown in Fig. 6(A). We used a pulse reverse plating methods to fill the through-holes with electroplated nickel. The periodic reverse plating method was effective in filling the through-holes with metal especially for the high aspect ratio structures [5]. The fabrication process has already been shown in Fig. 2. The thickness of the nickel within the through-holes was rendered uniform by pulse reverse plating. However, the reproducibility was poor in the case of through-holes with a high aspect ratio. Sometimes no electroplating in the holes occurred. This was due to the formation of air bubbles within the through-holes. In the referenced paper [5], methanol was used to remove air bubbles. This process cannot be applied to our process because we used photoresist as an adhesive between the seed layer and the glass plate as shown in Fig. 2. The methanol would make the photoresist swell and would result in the formation of a mask on the seed layer. In this study, we used an aspirator to remove air bubbles. After aspiration for a few tens of minutes, the glass was immersed in an ultrasonic bath for a few seconds. Bubbles were completely removed by this treatment. Fig. 6(B) shows the glass after the electroplating. We succeeded in filling the through-holes with metal with an aspect ratio of up to 6.

For the next step in the process, we needed to remove nickel that had over-plated on to the top surface of the wafer to obtain a flat surface for photolithography. CMP was applied to selectively remove the overplated nickel. The glass after the CMP process is shown in Fig. 6(C). The estimated polishing rates were 2.0 μm/hour and 8.6 μm/hour for Pyrex glass and electroplated nickel, respectively. The load applied to the interface between the pad and sample was 0.16 kg/cm^2. Here the slurry used for the CMP process was an aqueous solution of alumina (0.3 μm in diameter) and oxygenated water (H_2O_2 5 vol.%). The oxygenated water plays an important roll in passivating the nickel surface and the passivation film can easily be removed by the mechanical action of the alumina particles. We can obtain scratch-free surfaces after polishing using a particle size of 0.3 μm. With the polishing conditions used, the nickel was polished more than the Pyrex glass. If the load is 0.04 kg/cm^2 and no H_2O_2 is added, both the Pyrex glass and the nickel have the same polishing rate (0.8 μm/h). To obtain a flat surface and to remove the passivating film, we polished using these conditions as a final polish. Details on CMP technology and on the polishing mechanism have already been published in the field of semiconductor applications [10].

Fig. 7 shows a cross sectional view of the fabricated Pyrex glass for a high density electrical feed-through. Here the glass was bonded to silicon by anodic bonding as shown in the figure. The anodic bonding was carried out by applying electric field between the metallized through-holes using multipoint electrode.

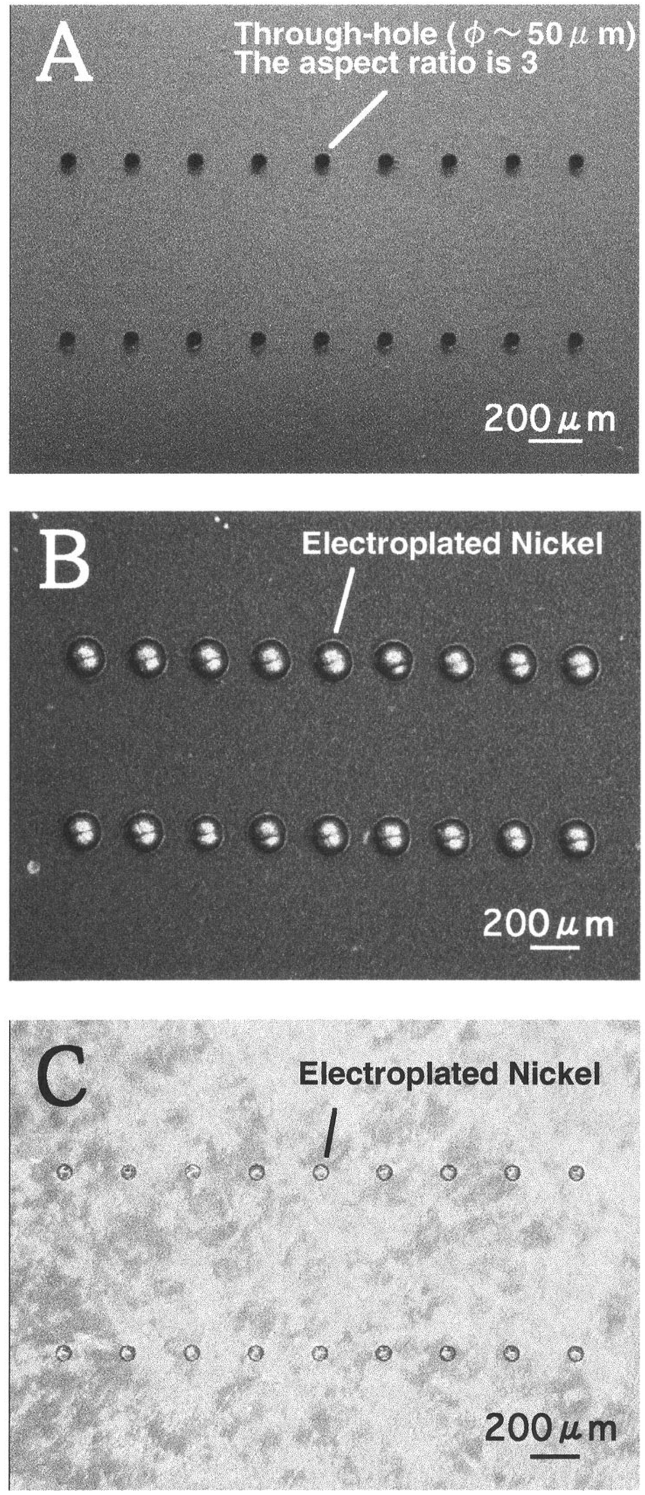

Fig. 6. The top views of the Pyrex glass at various process steps. (A) Through-holes fabricated by DRIE. (B) Through-holes filled with electroplated nickel. (C) Through-holes after chemical mechanical polishing.

The electrical resistance was measured using a four-point wire sensing method. The value of the measured electric resistance was about 40 mΩ per hole. The changes in the resistance were within 2% after a year later. Reliability testing of the

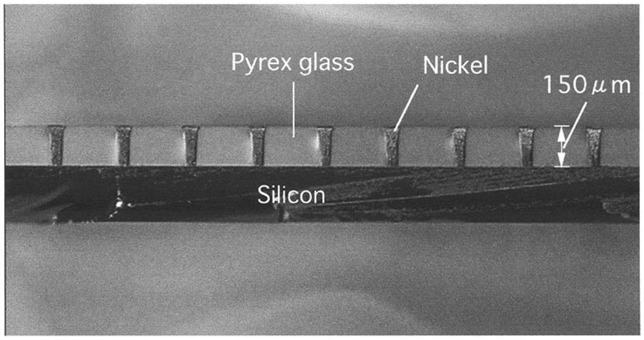

Fig. 7. A cross-sectional view of the fabricated Pyrex glass for high-density electrical feed-throughs. The glass was bonded with silicon by anodic bonding (The temperature is 400 °C and the applied voltage is 1000 V).

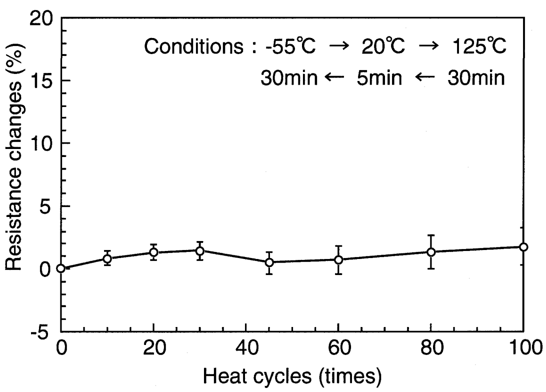

Fig. 8. The heat cycles test from −55 °C to 125 °C.

glass wafers with electrical feed-throughs was also executed, because the device consists of different materials. The mismatch in the coefficient of thermal expansion results in the cracking at the interface of through-holes. Heat cycles test from −55 °C to 125 °C was employed for testing the reliability. The procedure is as follows; The fabricated glass wafers with electrical feed-throughs were placed in a temperature controlled bath at −55 °C for 30 min, then transferred into the other bath controlled at 125 °C with 5-min transition. The sample was placed at the temperature for 30 min, then again transferred into the former bath with five minute transition. The electrical resistance through the test was monitored periodically during the cycle. The result after 100-cycles test was shown in Fig. 8. The resistance changes were within 3%. For the electrical feed-throughs filled with nickel, the resistance changes are very small.

The performance of the electrical feed-through combined with a microprobe array is now under investigation in our laboratory [11], [12].

IV. Conclusion

Pyrex glass for high density electrical feed-throughs were fabricated based on our DRIE techniques. Small through holes (40–80 μm in diameter) in a Pyrex glass wafer have been fabricated using deep-reactive-ion etching in a sulfur hexafluoride (SF_6) plasma. By filling the through holes with an electroplated metal, fine pitch electrical feed-throughs in a Pyrex glass wafer were realized.

Our final goal is to combine the technology with a probe-based technology. In order to meet the demands of researchers engaged in probe-based technologies such as data-storage systems, microswitches, and the other applications, a process for batch-fabrication of high density electrical feed-throughs needs to be established. Most of the microcantilevers were fabricated by silicon micromachining. Pyrex glass can be easily bonded to silicon using anodic bonding. Therefore, a high density of electrical feed-throughs fabricated by the DRIE of Pyrex glass is an attractive method for these applications. Our technology is one of the solutions that will enable us to achieve a high density of electrical feed-throughs.

References

[1] T. Corman, P. Enoksson, and G. J. Stemme, "Deep wet etching of borosilicate glass using an anodically bonded silicon substrate as mask," *J. Micromech. Microeng.*, vol. 8, pp. 84–87, 1998.

[2] P. Dario, M. C. Carrozza, N. Croce, M. C. Montesi, and M. Cocco, "Nontraditional technologies for microfabrication," *J. Micromech. Microeng.*, vol. 5, pp. 64–71, 1995.

[3] T. Diepold and E. Obermeier, "Smoothing of ultrasonically drilled holes in borosilicate glass by wet chemical etching," *J. Micromech. Microeng.*, vol. 6, pp. 29–32, 1996.

[4] H. Wensink, J. W. Berrenschot, H. V. Jansen, and M. C. Elwenspoek, "High resolution powder blast micro-machining," in *Proc. 13th IEEE MEMS 2000 Technical Digest*, vol. 1/23–27/00, Miyazaki, Japan, 2000, pp. 769–774.

[5] T. R. Anthony, "Forming electrical interconnections through semiconductor wafers," *J. Appl. Phys.*, vol. 52, pp. 5340–5349, 1981.

[6] X. H. Li, T. Abe, and M. Esashi, "Deep reactive ion etching of Pyrex glass," in *Proc. 13th IEEE MEMS 2000 Technical Digest*, vol. 1/23–27/00, Miyazaki, Japan, 2000, pp. 271–276.

[7] ——, "Deep reactive ion etching of Pyrex glass using SF_6 plasma," *Sens. Actuators, Phys. A*, vol. 87, pp. 139–145, 2001.

[8] S. Kong, M. Minami, and M. Esashi, "Fabrication of reactive ion etching system for deep silicon machining," *T. IEE Jpn.*, vol. 117-E, pp. 10–13, 1997.

[9] M. Esashi, "Encapsulated micromechanical sensors," *Microsyst. Technol.*, vol. 1, pp. 2–9, 1994.

[10] F. Kaufman, D. B. Thompson, R. E. Broodie, M. A. Jaso, W. L. Guthrie, D. J. Pearson, and B. Small, "Chemical–Mechanical polishing for fabricating patterned W metal features as chip interconnections," *J. Electrochem. Soc.*, vol. 138, pp. 3460–3464, 1991.

[11] D. W. Lee, T. Ono, T. Abe, and M. Esashi, "Fabrication of microprobe array with sub-100nm nano-heater for nanometric thermal imaging and data storage," in *Proc. 14th IEEE MEMS 2001 Technical Digest*, vol. 1/21–25/01, Interlacken, Switzerland, 2001, pp. 204–207.

[12] Y. X. Liu, X. H. Li, T. Abe, Y. Haga, and M. Esashi, "A thermomechanical relay with microspring contact array," in *Proc. 14th IEEE MEMS 2001 Technical Digest*, vol. 1/21–25/01, Interlacken, Switzerland, 2001, pp. 220–223.

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