Bonding properties of metals anodically bonded to glass — **Source link** ⬈

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Bonding properties of metals anodically bonded to glass

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Abstract

Anodically bonded Pyrex-/Metal double stack were investigated for applications in the sensor encapsulation field. The use of metals can increase the robustness of the packaging and eliminate the use of glue. Titanium anodically bonded to glass can lead to biocompatible systems The anodic bonding of metallic sheets of Invar, Kovar, Alloy 42 and titanium to ion-containing glasses, Pyrex and Foturan, was evaluated in terms of samples preparation, bonding parameters, and bonding characteristics. At a bonding temperature below 300 °C, the stress induced to Pyrex was smaller when the Invar was used, however, a weak bonding was obtained at the lowest bonding temperatures investigated. In comparison with Invar and Alloy 42 bonded to Pyrex, Kovar induced a smaller stress for bonding temperatures higher than 350 °C. For bonding temperatures in between 300 and 350 °C, a similar value of stress was obtained for Kovar and Alloy 42 bonded to Pyrex as well as a high bonding strength. A post-annealing step at a temperature of and higher than the bonding temperature was shown to decrease the bonding stress and can be used to improve the bonding strength of samples bonded at low temperature. Kovar and Alloy 42 bonded to Pyrex at temperatures of and higher than 250 °C were tight to liquid at a pressure of 1.5 bars. In the case of titanium, Pyrex and Foturan were successfully bonded to titanium thin films and sheets, respectively. A proper selection of metals and bonding parameters led to levels of residual stress, strength and tightness that make anodic bonding of metals to glass a suitable technique for the assembling and packaging of microsystems, for instance piezoresistive silicon sensors and microfluidic devices.

Keywords: Anodic bonding; Metal; Glass; Silicon; Encapsulation; Packaging; Sensor

1. Introduction

The anodic bonding of various conductive materials to glass has been realised successfully for the first time in 1969 by Wallis and Pomerantz [1]. They demonstrated that it was possible to irreversibly bond different metals and semiconductors to an ion-containing glass by applying a potential between the two samples and heating them at a relatively low temperature. The following papers on metal to glass anodic bonding mostly presented the procedure to realise the bonding and investigations on the mechanisms involved [2–6]. Among the samples, on which the anodic bonding to glass was successfully achieved, were materials having a similar thermal expansion coefficient to the Pyrex–glass, such as Kovar, Alloy 42 and silicon. Since then, with the grow of the microsystems field, this technique has been especially developed for bonding silicon to glass for device assembling and packaging purposes, leaving the use of metals aside for a while [7,8].

However, the anodic bonding of metal to glass can also be of great interest for sensors encapsulation. The use of metals can increase the robustness of the packaging and eliminate the use of glue. The latter could be essential in some applications, as for examples, in the packaging of sensors operating in harsh environments, and of piezoresistive silicon sensors used as pressure or liquid flow sensors [9]. Other applications in microfluidics are the integration of robust interconnects and sealing of devices [10]. Furthermore, the combination of titanium anodically bonded to glass can lead to biocompatible systems.

Few articles on the application of anodic bonding of metal to glass have been published so far, among which, Kovar to glass microfluidic connections [10,11] and a bakable microvalve [12]. Neither in the recent work, nor in the first papers published on the subject, was reported the influence of the bonding parameters on the properties of the bonding itself. Characterisation of the bonding properties would be helpful to define potential applications in the field of microsystems.

In this communication, the characterisation of metal to Pyrex anodic bonding properties is reported. Metals having a coefficient of thermal expansion close to Pyrex, such as

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Invar, Alloy 42 and Kovar, were investigated. The characterisation of the bonding characteristics was concentrated on three main properties, the stress, the strength and the bonding tightness, which are of importance in the field of sensor encapsulation or packaging. The thermal stress induced by bonding the samples together, the bonding strength and the tightness of the bonded interface to liquids depend on the nature of the materials involved, their preparation and the parameters used to anodically bond them, e.g. the bonding temperature and voltage. The emphasis in this study was put on determining the influence of the bonding temperature on the different bonging characteristics mentioned above. The results obtained showed that, with a proper choice of materials and adapted bonding parameters, this bonding technique could be suitable for the encapsulation of sensors.

Moreover, with the growth of the biological micro-electromechanical systems (BioMEMS) involving the field of microfluidics for handling liquid biological samples, experiments were carried on to bond Pyrex and Foturan glasses to sheets and thin films of titanium. The use of titanium anodically bonded to glass could lead to biocompatible systems. In that case, the investigations were mainly concentrated in the determination of bonding parameters resulting in the relatively strong bonding of titanium to the glass materials.

2. Experimental

2.1. Materials

Since the samples are anodically bonded together at a relatively high temperature (300–450 °C), an important parameter to minimise for achieving a successful bond between the metals and glass plates is the resulting thermal stress. The induced stress comes from the difference between the coefficients of thermal expansion (CTE) of the materials used. Two different types of ion-containing glasses were investigated, Pyrex (Corning 7740) and Foturan (Schott) glasses, with respectively a CTE close to Si and close to Ti (Table 1). On one hand, Pyrex was an obvious choice since it is well established in the field of microsystems technology. It is a material used in the fabrication, assembling and packaging of the devices. Since it can be anodically bonded to Si, the realisation of bonded triple stack of metal, Pyrex and Si is conceivable. On the other hand, Foturan, a photostructurable glass, can hardly be anodically bonded to Si (CTE mismatch)

and this for small surfaces. However, it was considered as a good candidate to be bonded to Ti since they have almost the same CTE. The use of Ti can lead to the fabrication of biocompatible microfluidic systems with the integration of robust Ti fluidic interconnects.

Invar (64% Fe, 36% Ni), Kovar (54% Fe, 29% Ni, 17% Co) and Alloy 42 (58% Fe, 42% Ni) are metals that were chosen to be anodically bonded to Pyrex because of their CTE close to the coefficient of Pyrex. In term of stress, Invar and Kovar should provide a minimum stress when bonded at low and at high temperatures, respectively (Table 1). Titanium was the only metal considered to be bonded to Foturan. The CTE of the different materials used are summarised in Table 1.

2.2. Samples preparation and bonding parameters

Pyrex and Foturan square chips (15 mm, 500 µm thick) were anodically bonded on small metal square plates (40 mm diameter, 500 µm thick) with the aim of investigating the influence of the surface roughness and of the bonding conditions on the characteristics of metals bonded to glass. The metal plates were cut using water jet to avoid a mechanical pre-stress. They were then polished with an aluminium oxide slurry (0.3 µm) prior to bonding, which was found to be a necessary step to have the bonding occurring. The surface roughness of the metallic plates was characterised using a Profilometer (Tencor, Alphastep 200) and AFM (DI, Nanoscope 3) prior and after the polishing step.

The bonding of the glass chips to the metallic plates was performed in laboratory air (clean room class 1000) at temperatures varying from 200 to 450 °C and voltages from 500 to 1500 V. The samples were cleaned in acetone and isopropanol before bonding them together.

2.3. Thermal stress

The thermal stress has been studied at room temperature by measuring the bow of bonded metal–Pyrex samples with the tip of a profilometer. The radius of curvature was deduced from the bow and used to calculate the stress of the Pyrex chip bonded on the metallic plate according to the following formula [13]:

$$\sigma_{\mathrm{p}} = \frac{1}{d_{\mathrm{p}}} \cdot \frac{(\alpha_{\mathrm{M}} - \alpha_{\mathrm{P}}) \cdot \Delta T}{1 - \frac{\nu_{\mathrm{P}}}{d_{\mathrm{P}} E_{\mathrm{P}}} + \frac{1 - \nu_{\mathrm{M}}}{d_{\mathrm{M}} E_{\mathrm{M}}}} \tag{1}$$

Table 1
Thermal expansion coefficients of the materials used for the anodic bonding of metals to glass.

Temperature (°C)	Coefficients of thermal expansion (ppm/K)						
	Si	Pyrex	Alloy 42	Kovar	Invar	Foturan	Ti
0–200	3.0	3.1	4.5	5.5	1.7	–	–
0–300	3.3	3.2	4.5	5.1	4.9	8.6	9.5
0–350	3.4	3.2	5.0	–	6.6	–	–
0–400	3.5	3.3	6.0	4.9	7.8	–	–

where $\alpha_{P,M}$ is the coefficient of thermal expansion of Pyrex or metal; ΔT the variation of temperature; $\nu_{P,M}$ the Poisson ratio of Pyrex or metal; $E_{P,M}$ the Young's modulus of Pyrex or metal; $d_{P,M}$ is the thickness of Pyrex or metal.

Moreover, measurements of the residual stress of the bonded samples as a function of the ambient temperature (40–400 °C) were performed in an inert gaseous atmosphere. These experiments were also carried out to investigate the influence of an annealing at a temperature of and higher than the bonding temperature on the residual stress of the metal/Pyrex bonded stacks at room temperature. The samples were placed in the FLX-2900 from Tencor, a wafer curvature measurement apparatus, where they were heated and the bow of the bonded samples measured using a laser.

2.4. Bonding strength

Two methods were used to evaluate the bonding strength of the metallic plates bonded to Pyrex. In the first method, a hole was machined in the metal chips to enable the application of a nitrogen pressure at the bonding interface. The bonding surface energy can be obtained from the measurement of the critical pressure necessary to debond the Pyrex from the silicon chip [14]. The equation is the following:

$$\gamma = \frac{0.088\,p^2 a^4}{E t^3} \qquad (2)$$

where γ is the bonding surface energy; p the critical pressure for debonding; a the radius of the hole; E the Young's modulus of the glass; t is the thickness of the glass chip.

However, the formulae (2) was developed in the case where the Pyrex glass is bonded to silicon. Metals exhibit a plastic deformation, which is not the case of silicon. One should be aware that the values of energy obtained when applying this formulae to metal/glass system have still to be validate. A task not covered in this paper. Therefore, the energy values presented in the results section should be

considered as a qualitative indication of the variation of the bonding strength as a function of the bonding temperature.

The second method used was the blade test [15]. The first results obtained gave qualitative indications on the strength of the bonded interfaces and confirmed the qualitative results obtained from the critical pressure necessary to debond the chips.

2.5. Bonding resistance to liquids

To characterise the bonding tightness to liquid, Kovar–Alloy42/Pyrex bondings performed at different temperatures (1000 V) were immersed for few weeks in a water container, in which a nitrogen pressure of 1.5 bars was maintained. A fluorescent label, fluorescein, with a size of the order of the nanometer, was added to the water. A standard microscope (NIKON) working in the visible optical spectrum and a microscope for the detection of the fluorescent labels (Axios-vert S100, Carl Zeiss) were used to observe the samples after being exposed to the liquid. More details about the set-up used for the detection of fluorescein can be found in reference [16].

3. Results and discussion

3.1. Samples and bonding preparation

The metal sheets had a surface roughness, peak to peak, of ≈4 µm on a 2 mm range before polishing. Kovar and Alloy 42 samples were polished to a surface roughness of ≈150 nm on a 2 mm range. Fig. 1 shows SEM photos of unpolished and polished Kovar plates, similar results were obtained for Alloy 42 and Invar. The local surface roughness of polished Kovar and Alloy 42 evaluated using AFM had a rms value varying from 5 to 10 nm. Polished Invar, Kovar and Alloy 42 were successfully bonded for bonding temperatures between 200 and 450 °C, and for voltages between 500 and 1500 V.
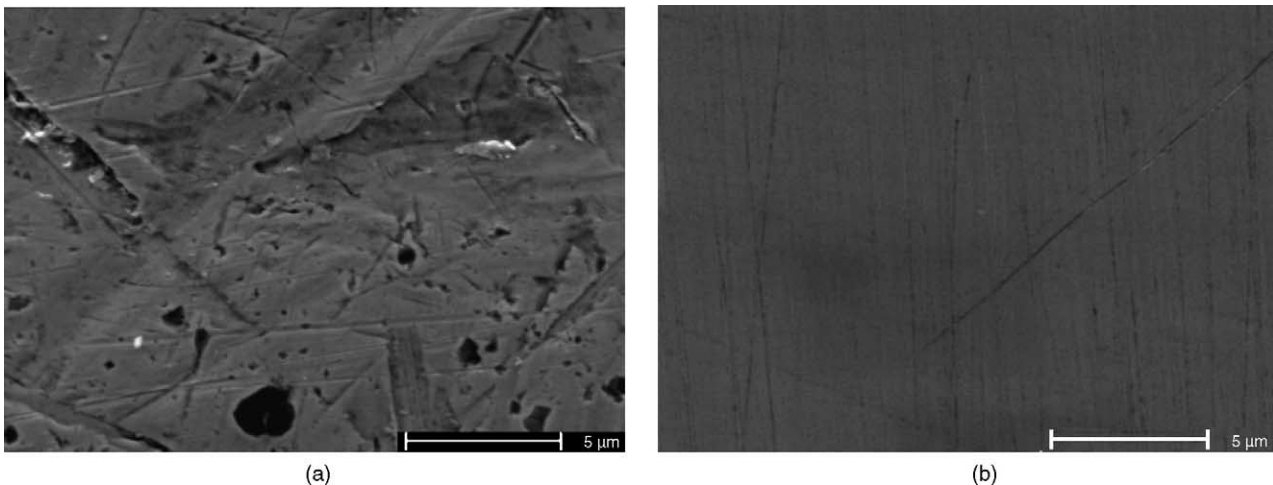

(a)   (b)

Fig. 1. SEM photos of unpolished (a) and polished (b) Kovar plates.

However, bondings performed at temperatures below 250 °C were found to be weak just by manually putting pressure on the samples. The bondings performed to evaluate the bonding characteristics, such as stress, strength and tightness were done at a constant voltage of 1000 V.

In the case of the titanium plates, they were harder to polish than the other metals. The Ti sheets had a peak to peak surface roughness before and after polishing, respectively of ≈6 μm and ≈800 nm on a 2 mm range. Obtaining a successful bonding to Pyrex was harder when using Ti as metal due to the CTE mismatch. The results from the bonding experiments performed to anodically bond Ti to glass (Pyrex and Foturan) are detailed in section 3.5.

3.2. Thermal stress

The residual stress at room temperature of Invar, Kovar and Alloy 42 anodically bonded at different temperatures to Pyrex is presented in Fig. 2. At a bonding temperature below 300 °C, the stress induced to Pyrex was smaller when the Invar was used. However, the very low values of stress obtained for Invar bonded to Pyrex is probably due to the fact that the bonding did not occur well between the two samples, the bonding strength being very weak (see section 3.3). In comparison with Invar and Alloy 42 bonded to Pyrex, Kovar induced a smaller stress for bonding temperatures higher than 350 °C. For bonding temperatures in between 300 and 350 °C, similar values of stress were obtained for Kovar and Alloy 42 bonded to Pyrex, which were smaller than those obtained when Invar was used. Dispersion in the value of the stress obtained for a given bonding temperature was observed and correlated to the curvature of the metallic plate. More pronounced was the bow of the plate, bigger was the residual stress measured, as presented in Fig. 3, showing that a control of the parallelism of the metallic sample is important to have reproducible results.

The residual stress of Kovar and Alloy 42 samples bonded to Pyrex at different temperatures was measured at a function

Fig. 3. Relation between the metal initial curvature and the residual stress obtained when bonded to Pyrex at different temperatures (voltage = 1000 V) for Kovar and Alloy 42 (500 μm thick).

of the ambient temperature. Fig. 4 shows the typical behaviour of the stress variation for Kovar and Alloy 42–Pyrex stacks as a function of the ambient temperature. One factor that could explain the non-linearity observed is the variation of the CTE of the materials investigated as a function of temperature. The samples bonded at different temperatures (250, 350, 450 °C) had a very similar non-linear behaviour, which tend to confirm the suggested explanation. During the

(a)

(b)

Fig. 4. Thermal stress as a function of the ambient temperature for: (a) Kovar/Pyrex; (b) Alloy 42/Pyrex bonded at 350 °C, 1000 V (500 μm thick samples).
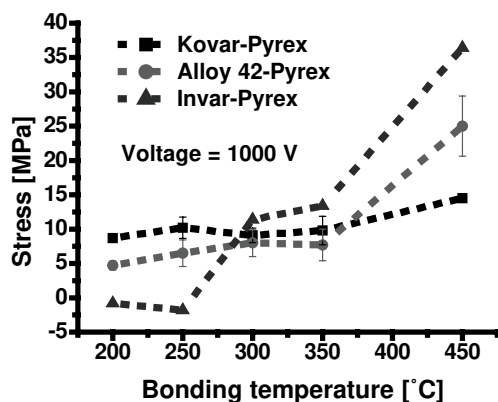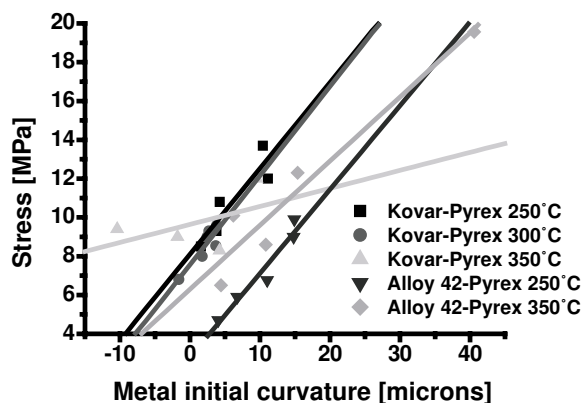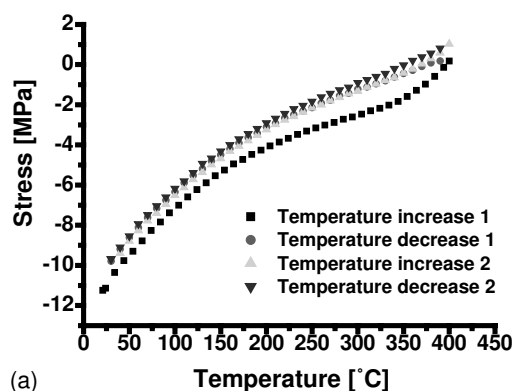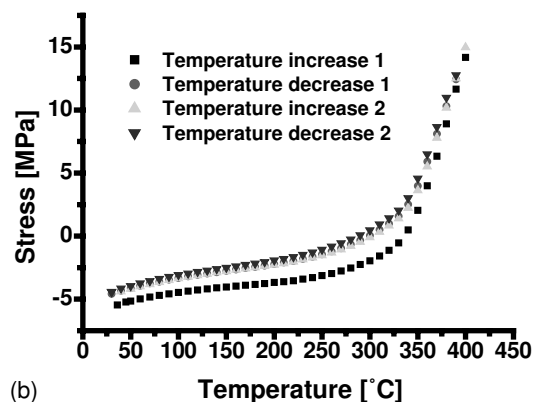

Fig. 2. Residual stress at room temperature for Invar, Kovar and Alloy 42 (500 μm thick) anodically bonded to Pyrex (500 μm thick) at different temperatures, voltage = 1000 V.
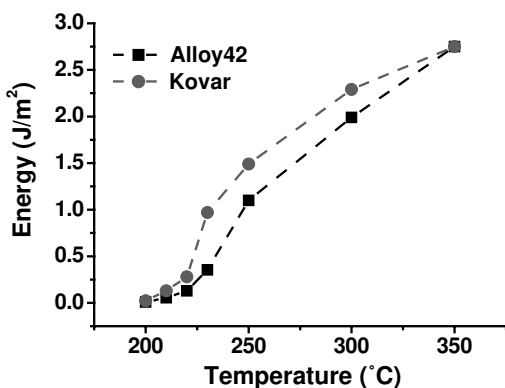
Fig. 6. Relative strength of metals anodically bonded to Pyrex (voltage of 1000 V) as a function of the bonding temperature obtained from the critical pressure for debonding the Pyrex chip (500 μm thick samples).

sufficient. Post-thermal treatments on Invar bonded to Pyrex at low temperature could be a solution to preserve the small residual stress and in the same time increase the bonding strength. Experiments have still to be performed to optimise the processing parameters to obtain satisfactory values for these two characteristics, the stress and the strength, on Invar samples bonded to Pyrex.

3.4. Bonding tightness to liquids

In the test conditions described in section 2.5, the bondings of metals to Pyrex performed using Kovar and Alloy 42 were found to be tight to liquid when bonded at temperatures of and higher than 250 °C. The fluorescein labels did not penetrate at the bonding interface. Invar was not tested due to its small strength when bonded at low temperature, and due to its higher residual stress, compared to Kovar and Alloy 42, when bonded to Pyrex at higher temperature.

3.5. Bonding to titanium

The experiments carried out to anodically bond glass to titanium were performed at temperatures between 300 and 400 °C. The first attempts to bond Foturan to Ti sheets were performed successfully at 400 °C and at a lower voltage of 300–400 V. At higher voltages, electrical breakdown occurred in the Foturan and cracks could propagate in the material and black spots be formed. However, the results were hard to reproduce. Bonding Pyrex to Ti sheets was not performed successfully even if it has been reported to be possible before [1]. Different polishing and cleaning procedures, as well as different bonding conditions were tried out without success. Debonding of the Ti plate occurred during the cooling step performed after the bonding. However, Pyrex was bonded with success to a Ti thin film (500 nm thick) between 300 and 400 °C and 800 V. The Ti thin films were deposited by e-beam evaporation on a 525 μm thick Si wafers on which a 80 nm thermally dry silicon oxide was grown.

Pyrex to Pyrex plates could be anodically bonded together (300–400 °C, 800 V) using an intermediate Ti thin film deposited on one Pyrex plate. The parameters leading to successful bondings using Ti and glasses, especially Foturan, still need more investigations and the bonding properties to be characterised systematically.

4. Application to microsystems packaging

From the characterisation results obtained in this study, Kovar and Alloy 42, when bonded to Pyrex at a temperature between 300 and 375 °C, exhibit levels of residual stress, strength and tightness that are suitable for applications in the field of sensor assembling, encapsulation and packaging. An interesting reduction of the residual stress can be obtained by annealing the bonded samples at a temperature of and higher than bonding temperature. This annealing could also be performed on devices bonded at lower temperature to increase their bonding strength and still maintain a small residual stress.

Different applications of metal to glass anodic bondings have been reported recently in the field of microsystems technologies [10–12]. In our case, metal to glass anodic bonding was used to integrate robust fluidic interconnects to a piezoresistive silicon/Pyrex liquid flow sensor [9]. Metallic plates of Kovar and Alloy 42, on which a Pyrex PCB was anodically bonded and tubes fixed, were used for this purpose. Moreover, concerning silicon piezoresistive sensors, metal to glass anodic bonding is foreseen to have also applications in the packaging field of pressure sensors operating in harsh environments. In the case of titanium, its biocompatible property combined with its assembling to Pyrex and Foturan, materials in which microchannels can be structured, make it a good candidate to be used in biocompatible microfluidic systems. Bonding Foturan and Pyrex to titanium plates are investigated to seal microchannels and integrate robust biocompatible titanium fluidic interconnects. Anodic bonding of Pyrex can also be successfully performed on thin metallic films [1]. An application of this technology is the encapsulation under vacuum of quartz resonators. Pyrex caps are bonded on thin metallic films forming the electrodes of the resonators [17].

5. Conclusions

This communication presented the characterisation of anodically bonded Pyrex–Foturan/metal double stack for applications in the field of sensors encapsulation. Invar, Kovar, and Alloy 42 were bonded successfully to Pyrex at temperatures of 250–450 °C and voltages of 500–1500 V. The stress, strength and tightness to liquid of these bonded samples were characterised as a function of the bonding temperature. Selected metals anodically bonded with proper process parameters to glass exhibited levels of residual
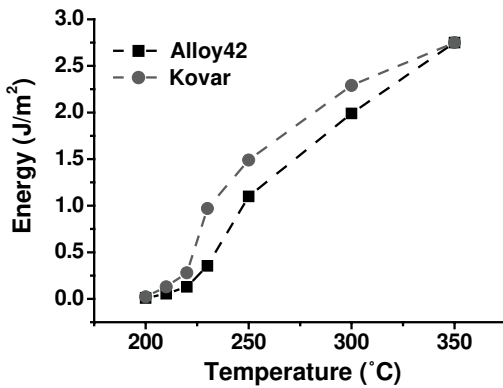
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Pyrex to Pyrex plates could be anodically bonded together (300–400 °C, 800 V) using an intermediate Ti thin film deposited on one Pyrex plate. The parameters leading to successful bondings using Ti and glasses, especially Foturan, still need more investigations and the bonding properties to be characterised systematically.

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stress, strength and tightness that make this technique suitable for packaging microsystems.

The application of this technique to microsystems packaging was demonstrated through the integration of robust fluidic interconnects to a piezoresistive silicon/Pyrex liquid flow sensor. Titanium was also considered for applications in the field of microfluidics where biocompatibility can be of great interest. Pyrex was successfully bonded to thin titanium films on silicon and to Pyrex using an intermediate Ti thin film. Preliminary results showed also the bondability of Foturan to thicker titanium sheets. Bonding Foturan to titanium plates and Pyrex to Pyrex using an intermediate Ti thin film are investigated to seal microchannels and integrate robust biocompatible titanium fluidic interconnects.

Acknowledgements

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Biographies

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Patrick Weber obtained his diploma in physics in 2001 from the University of Neuchâtel, Switzerland. He joined the Sensors, Actuators and Microtechnology Laboratory, Neuchâtel, Switzerland to work on a European project in the field of wafer bonding. He is now a PhD student in the particle physics group from the University of Neuchâtel, Switzerland, working on the development of detectors for neutrinos detection.

Nicolaas F. de Rooij received a PhD degree from Twente University of Technology, The Netherlands, in 1978. From 1978 to 1982, he worked at the Research and Development Department of Cordis Europa N.V., The Netherlands. In 1982, he joined the Institute of Microtechnology of the University of Neuchâtel, Switzerland (IMT UNI-NE), as professor and head of the Sensors, Actuators and Microsystems Laboratory. Since October 1990 till October 1996, he was acting as director of the IMT UNI-NE. Since 1987, he has been a lecturer at the Swiss Federal Institute of Technology, Zurich (ETHZ), and since 1989, he has also been a professor at the Swiss Federal Institute of Technology, Lausanne (EPFL). His research activities include microfabricated sensors, actuators and microsystems.