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Conference

Steady-state measurement of wafer bonding cracking resistance

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Abstract

A steady-state wedge-opening test has been developed in order to measure the fracture toughness of bonded silicon wafers. Comparison between non-steady-state and steady-state tests is performed. The importance of allowing the rotation of the testing stage is discussed and appears to be essential in order to have the wedge perfectly aligned with the sample. Significant influence of (1) surface treatment; (2) thermal annealing; and (3) crack velocity on the toughness is observed for Si/Si wafer bonding and related to the interface chemistry.

Keywords: Wafer bonding; Wedge-opening test; Computational method

1. Introduction

Wafer bonding is a key microfabrication technique for the assembling of MEMS components and the packaging of microsystems. Reliability of MEMS thus requires the development of characterization and modelling tools that allow the assessment of the integrity of bonded interfaces against fracture and delamination. Simple uniform tensile or shear tests provide a value of the critical stress required for wafer separation. However, such critical stresses heavily depend on interface defects and do thus not constitute an intrinsic characteristic of the bond [1]. Indeed, the probability of finding defects will depend on the specimen size. The relevant approach for characterizing interface toughness is to work within the framework of fracture mechanics and to measure the critical energy release rate G_c, i.e. the "bond fracture toughness" [2]. The choice was made to employ *fracture toughness* concept rather than *surface energy*, because it relates to the true capacity of the interface to resist debonding which is the necessary ingredients for the design of a fail-safe structure. Indeed, G_c is equal to twice the surface energy γ only in the case of brittle materials or bonds in which no other mechanism than atomic planes separation takes place. In fact, even in very brittle materials, "atomic trapping" and reconstruction effects lead to $G_c > 2\gamma$ [2]. When, for instance, friction occurs between crack faces or dislocations are emitted at the crack tip, G_c becomes significantly larger than 2γ (e.g. G_c is typically 10^5 larger than 2γ in metal alloys), although very much dependent on slight changes of γ. In order to measure G_c and evaluate various bonding techniques and rate effects, a steady-state wedge-opening test has been developed extending earlier works in the literature on static wedge-opening method [1,3]. The first aim of the paper is to present this method and show the importance of conducting steady-state tests with respect to the accuracy of the measurement and to strain rate/environmental effects. The second objective is to discuss different wafers bond preparation routes regarding the interface toughness.

The samples preparation is presented in a first section. Then, the measurement technique is described followed by a presentation and discussion of the main results. Finally, a short introduction to the modelling tools used for the simulation of the interface behaviour is provided.

2. Experimental procedures

2.1. Samples preparation

The procedure for the bonding of Si wafer is presented in Fig. 1. After standard cleaning, the wafers undergo a surface treatment, which is either oxygen plasma or thermal oxidation or pressure enhanced chemical vapour deposition

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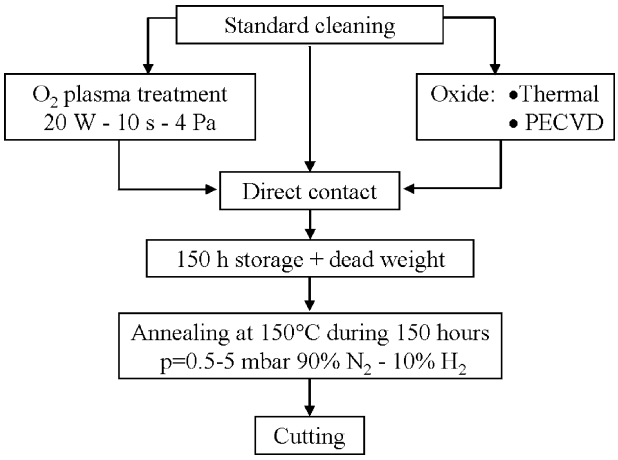

Fig. 1. Samples preparation procedure.

(PECVD) oxidation. In the oxygen plasma treatment, the wafers are exposed for 10 s to a 20 W, 25 cm^3/min oxygen plasma in a reactive ion etcher with a working pressure of 30 mTorr. Thermal oxide is obtained by the transformation of Si in SiO_2 at high temperature (950 °C) in the presence of vapour water. The thickness of the thermal oxide is around 4000 Å. The PECVD oxide is obtained by the continuous circulation of gas (SiH_4) on top of the wafers. This gas react with O_2 to form SiO_2 on top of the wafers. The thickness of the PECVD oxide is around 2500 Å.

The wafers are then brought in contact under constant pressure and stored for 150 h with a dead weight on the centre of the samples in order to avoid air bubbles. As explained in [1], this long storage time also allows the diffusion of water molecules present at the interface to the rim of the samples. The samples are then annealed at 150 °C for 150 h. Long rectangular specimens of 10 mm width are finally diced from the bonded wafers. The chemical reaction occurring during the annealing is.

$$\text{Si–OH} + \text{Si–OH} \leftrightarrow \text{Si–O–Si} + \text{H}_2\text{O} \quad (1)$$

First, when the wafers are in contact, the link between the wafers is made only of hydrogen bonds. Then, during the annealing, the formation of covalent bonds Si–O–Si (see Eq. (1)) between the two wafers induces the cohesion between these wafers. Reaction (1) is reversible until 450 °C [1]. It is essential to avoid the back reaction (1) that favours the dissociation of the covalent bonds. The elimination of water molecules is thus a key parameter for promoting strong bond. Elimination of water occurs following two different ways: (i) water molecules diffuse outside the sample; (ii) water molecules diffuse through the oxide layer and react with silicon. This last effect is easier in a native oxide layer which is thinner than the two oxides used in this research. In order to favour the evacuation of the water molecules, annealing was performed for a very long time (150 h) and under vacuum (from 0.5 to 5 mbar).

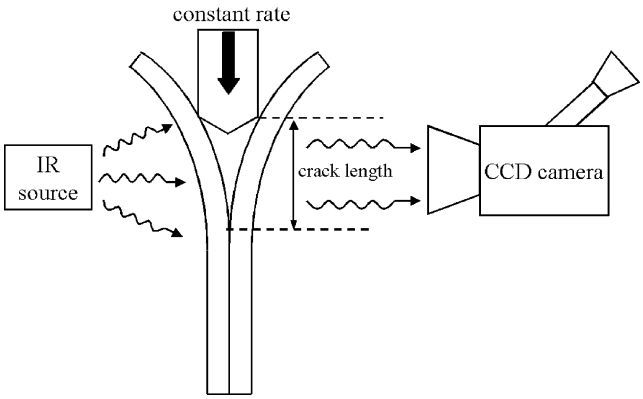

Fig. 2. Principe of the wedge-opening test.

2.2. Measurement technique

The measurement set-up is represented in Fig. 2. A razor blade of thickness u is inserted between the two wafers of thickness h at a constant rate using a home made set-up mounted on an universal mechanical testing machine. The crack length is continuously measured owing to the IR transparency of silicon and the contrast resulting from the presence of air between the wafers [4] (Fig. 3). The image acquisition software VISILOG 5.0® is used in order to record the images and to evaluate the crack length.

Simple beam theory leads to the following relationship between the crack length and the toughness for the configuration presented in Fig. 2 [2]:

$$G_c = \frac{3Eu^2h^3}{16a^4} \quad (2)$$

where E is the Young's modulus of the silicon wafers (taken to be equal to 200 GPa in our analysis), a the crack length, u the wedge thickness and h is the thickness of the wafers.

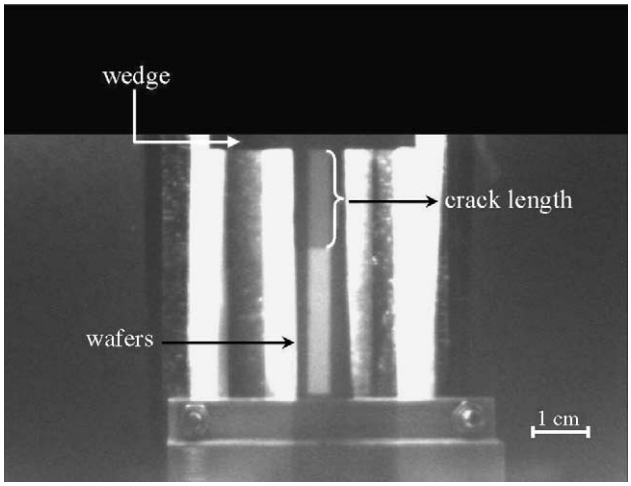

Fig. 3. Wedge-opening set-up. Note that the two outer white stripes are used in order to distinguish the edges of the wedge while the two inner stripes are not made on purpose but come from slight aperture between the wafers and the background mask.

All the tests were performed at room temperature (≅21 °C), under atmospheric pressure and relative humidity of approximately 50%.

3. Results

3.1. Typical result—importance of the steady-state condition

Fig. 4 shows the variation of the crack length as a function of the displacement of the wedge for two annealed samples. Three zones can be distinguished. The size of the first zone varies from one measurement to another due to transient effects related to small initial misalignment of the razor blade and uncontrolled initial velocity of blade insertion. The second zone corresponds to the true steady-state regime giving relevant crack length values. Only crack length values belonging to this plateau will be considered in the determination of the bond toughness. In the third zone, the crack tip stress field begins to interact with the specimen end which invalidates the use of Eq. (2).

3.2. Influence of the wedge thickness

In order to assess the validity of the test method and of the use of simple beam theory to derive Eq. (2), two different wedge thicknesses have been employed for the measurement of the toughness of samples coming from the same bonded wafers. The results are presented in Table 1 and demonstrate that the bond toughness obtained with the steady-state wedge-opening method is independent of the wedge thickness.

Table 1
Toughness of similar samples extracted using the steady-state wedge-opening test using two different wedge thicknesses

	Wedge's thickness = 100 μm	Wedge's thickness = 230 μm
G_c (J/m^2)	1.567 ± 0.3	1.629 ± 0.1

3.3. Comparison steady-state versus non-steady-state measurements

As shown in Fig. 5, when the wedge with an initial velocity of 25 mm/min is stopped within zone II (Fig. 4), i.e. when the wedge is perfectly aligned with the interface, the crack continues to propagate at a decreasing cracking velocity until no further crack propagation can be observed.

3.4. Influence of the wedge velocity

Fig. 6 shows the variation of the fracture toughness as a function of the crack velocity for annealed samples (no specific surface treatment except the cleaning and the 150 h annealing at 150 °C). A significant increase of the bond fracture toughness with increasing crack velocity is observed. At high crack velocity, the toughness reaches a plateau value. Another plateau is also expected to appear at very low velocity [2,5].

3.5. Influence of the surface treatment

The fracture toughness measured for different specimen preparation procedures for a low (0.25 mm/min) and a high (25 mm/min) value of the crack velocity are gathered in Fig. 7. The PECVD oxide treatment does not appear because

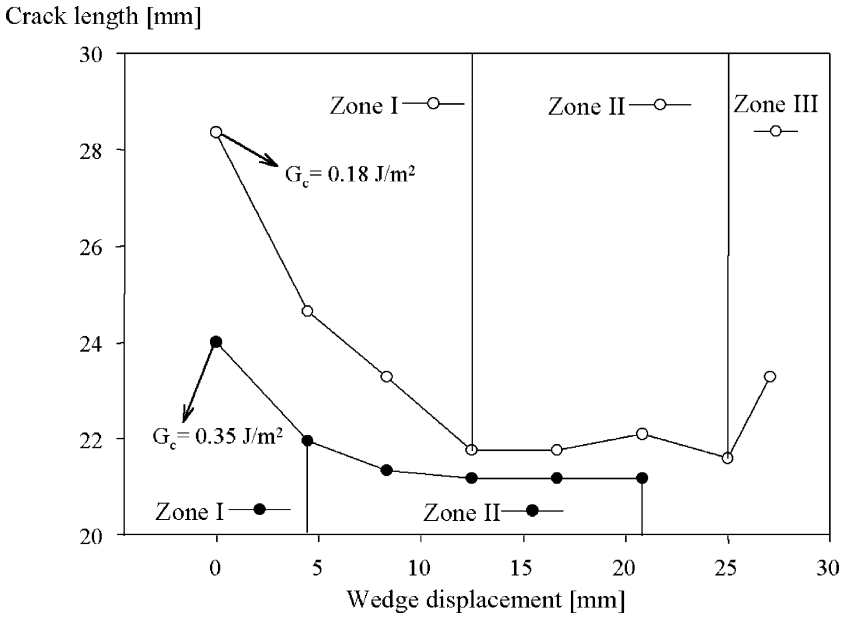

Fig. 4. Evolution of the crack length during wedge displacement. Crack length value from the second zone must be used to determine the toughness of bonding.

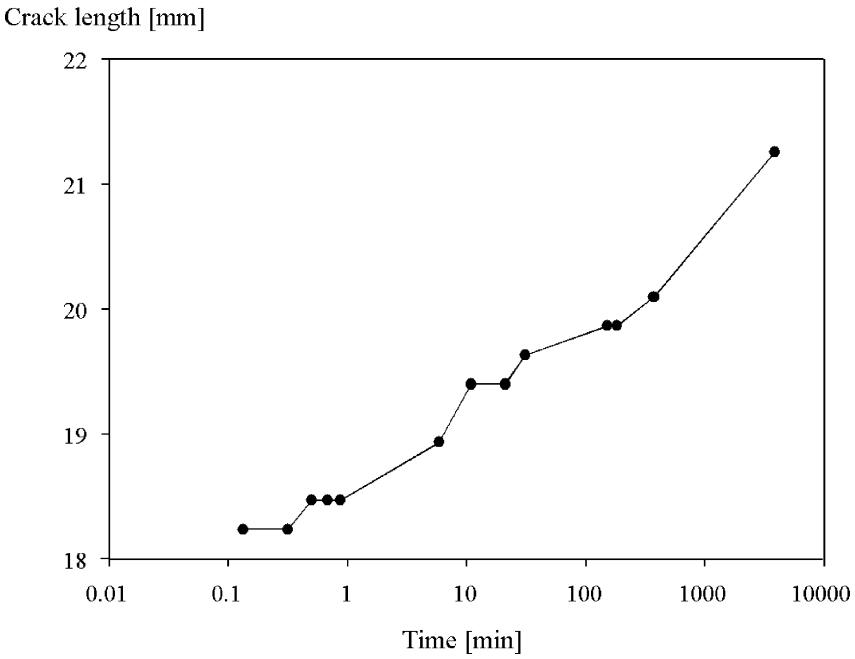

Fig. 5. Evolution of the crack length after stopping the wedge. The wedge is stopped at time $t = 0$ min.

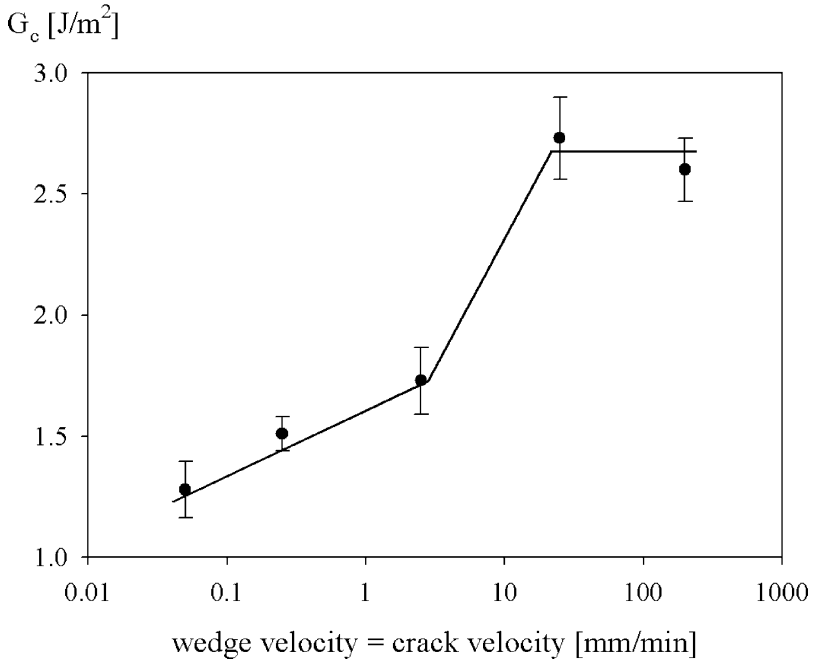

Fig. 6. Variation of the fracture toughness as a function of crack velocity. Measurements are performed on samples prepared by the same procedure (no specific surface treatment: 150 h annealing at 150 °C).

wafers did not bond during the contact due to a roughness (RMS 1.5 nm) larger than the maximum value of roughness required for pre-bonding [1]. Plasma treated wafers give the highest fracture toughness. The toughness is then so large that some specimens failed by the cracking of the silicon substrates.

4. Discussion

4.1. Measurement method

Regarding the test procedures, Fig. 4 shows that care should be taken when interpreting non-steady-state tests in

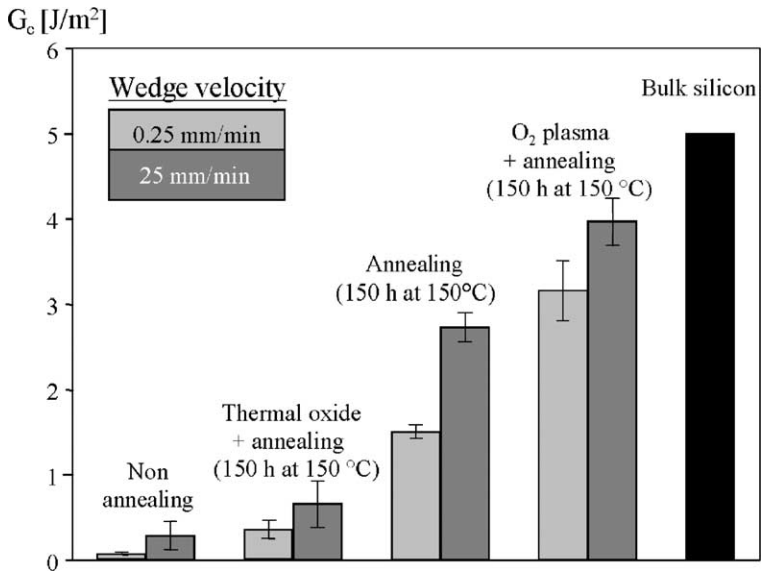

Fig. 7. Fracture toughness at two different crack velocities for various surface treatments.

which, for instance, the wedge is inserted manually between the wafers and the crack length is measured directly after. Indeed, small misalignment of the razor blade with respect to the crack plane leads to an effective opening that can be significantly larger than the blade thickness. The error can be very important as G_c is proportional to the square of the effective opening (see Eq. (2)). The specimen must be mounted on a free rotation grip to allow perfect alignment of the blade during testing. It is also possible that remaining water molecules close to the rim have dissociated the covalent bonds, weakening the bond strength and leading to longer crack length close to the edges of the samples. Note that our annealing conditions have been adapted in order to minimize this effect (see Section 2.1). A continuous measurement of the crack length also contributes to the evaluation of a representative average bond toughness as it allows several measurements at different location along the bonded wafers. As shown in Fig. 4, the values of the crack length in the second zone are almost the same for two samples even if they are quite different at the beginning of the test. Moreover, as shown in Table 1 the wedge test is independent of the wedge thickness. This means that the change of stress state in the wafers due to the change of wedge thickness is properly taken into account by the simple beam theory used in the derivation of relation (2) (mainly neglecting shear stresses close to the crack tips as well as anticlastic effects [6]). Note that in a study dealing with adherents that deform plastically during bending, Ferracin et al. [6] have shown significant effect of the wedge thickness on the stress state at the crack tip that was not accounted for by the simple beam theory. Finally, as shown in Fig. 5, great care must be taken when performing "static" test in laboratory environment. Indeed, even if the wedge is stopped, the crack continues to propagate until it attains a plateau value. Such type of "static" measurement are also less accurate as they only probe a small area of the interface leading to a local measurement.

4.2. Influence of the surface treatment

The toughness obtained for the different surface treatments were expected from the literature when considering the chemistry of the interface [1]. In non-annealed samples, only hydrogen bonds contribute to the adhesion of the wafers leading to very small bond toughness. The poor results obtained for the oxidized wafers come from the large roughness. The annealed samples show relatively good bond toughness due to the formation, during the annealing, of Si–O–Si bond between the wafers [1]. The higher value of bond toughness obtained for O_2 plasma treated samples is due to the higher density of these Si–O–Si bridges. These bond fracture toughness values almost reach the fracture toughness of bulk silicon. There is thus no interest to look for procedures leading to larger toughness. Indeed, if the toughness of the interface is too high, the crack will propagate into the substrate.

4.3. Influence of the wedge velocity

In Fig. 6, the evolution of the bond fracture toughness with wedge velocity can be attributed to the interaction with the environment, essentially the moisture. At high velocity, air water molecules cannot be adsorbed at the silicon surface and react with the Si–O–Si bond to dissociate it into two Si–OH groups as explained in [1,5]. When the crack velocity decreases, bond rupture by chemical reaction and healing events takes place. Toughness becomes much smaller at low crack velocity. More details on the mechanisms affecting the bond resistance to chemical cracking and responsible for the shape presented in Fig. 6 can be

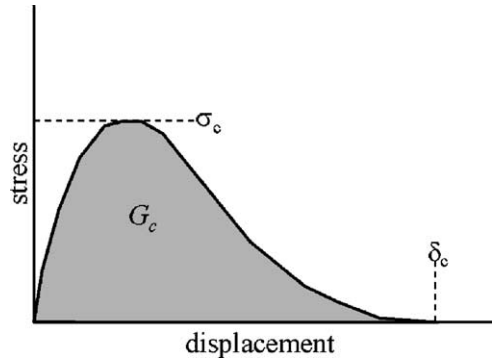

Fig. 8. Cohesive zone model.

found in [2,5]. Similar environmental effects leading to rate dependent fracture toughness were recently observed by Xu et al. [7] for TiN/SiO_2 interfaces.

4.4. Modelling

Knowing the toughness G_c is not sufficient for assessing the integrity of bonded elements in real microsystems with complex architecture. Modelling tools are required for the transfer of the toughness index measured in the laboratory on ideal specimens to structures involving complex stress and strain states. A methodology based on a physical failure criterion for the interface behaviour and implemented using the finite element method is currently used to describe the fracture behaviour of real MEMS. The interface is modelled using a "cohesive zone" relationship similar to the one drawn in Fig. 8 [8,9]. This stress displacement relationship represents the interaction between the two wafers. The area under the curve is the toughness of the bond G_c that is measured experimentally. A second parameter is required in order to fully characterize the fracture process: the maximum stress that the interface can sustain. It corresponds to the local strength of the interface and it plays a role to predict cracking when the initial defects is not very sharp (e.g. corner) [10]. The peak stress can be inferred from microtensile tests [1] or directly evaluated using atomistic calculations. This type of model has been successfully used for 5–10 years in the solid mechanics community in several applications such as adhesive bonding (e.g. [8,10]). An extension to rate dependent materials has also been proposed [11]. Accounting for rate effects (i.e. slow versus high loading rates) is an important ingredient of the modelling framework.

5. Conclusions and perspectives

The fracture toughness of bonded wafers for different surface treatments has been measured using a steady-state wedge-opening test. The importance of choosing the steady-state crack length is emphasized. High bond fracture toughness values were obtained for O_2 plasma treatment before annealing. The variation of the toughness with crack velocity is also observed and results from the interaction of moisture with interfacial atomic groups. Moreover, subcritical crack propagation due to the interaction between the external environment and the specific chemical species present at the interface is observed. Care should thus be taken when transferring these measurements to real MEMS depending on the environment of the interface and the nominal loading rates.

This type of test does not allow measuring toughness values greater than the substrate toughness. Indeed, if the toughness of the interface is greater than the toughness of the substrate, the crack will propagate into the substrate and preventing to the measurement the cracking resistance of the interface. Nevertheless, it is possible to solve this problem by decreasing the adhesive surface of the samples [12]. These types of measurements are currently under investigation.

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Biographies

Yannick Bertholet was born on 17 September 1977 in Dinant, Belgium. He received a materials science engineering degree from the Université Catholique de Louvain, Louvain-la-Neuve, Belgium, in 2001. He currently prepares a PhD in the Department of Materials and Processes Sciences (Division of Physical-Chemistry and Engineering of Materials) of the Université Catholique de Louvain, dealing with the characterisation of thin films (adhesion, mechanical properties, internal stresses) used in the microelectronics fields and particularly in MEMS.

François Iker was born on 13 July 1979 in Ottignies, Belgium. He received the electrical engineering degree from the Université Catholique de Louvain, Louvain-la-Neuve, Belgium, in 2002. He wrote his undergraduate thesis on low temperature wafer bonding techniques for MEMS and IC. He currently prepares a PhD at the Microwave Laboratory, UCL.

Jean-Pierre Raskin (M'97) was born in Aye, Belgium, in 1971. He received the industrial engineer degree from the Institut Supérieur Industriel d'Arlon, Belgium, in 1993, and the BS and PhD degrees in applied sciences from the Université Catholique de Louvain, Louvain-la-Neuve, Belgium, in 1994 and 1997, respectively. From 1994 to 1997, he was a research engineer at the Microwave Laboratory, Université Catholique de Louvain, Belgium. He worked on the modelling, characterization and realization of MMICs in silicon-on-insulator (SOI) technology for low-power, low-voltage applications. In 1998, he joined the EECS Department of The University of Michigan, Ann Arbor, USA. He has been involved in the development and characterization of micromachining fabrication techniques for microwave and millimeter-wave circuits and microelectromechanical transducers/amplifiers working in hard environments. Since January 2000, he is associate professor at the Microwave Laboratory of the Université Catholique de Louvain, Louvain-la-Neuve, Belgium. His research interests are the modelling, characterization and fabrication of: SOI MOSFETs for rf and microwave applications; planar circuits at millimeter and sub-millimeter waves frequencies; rf microelectromechanical systems (MEMS) and micromachined sensors. He is a IEEE Member and Member of the Research Center in Micro and Nanoscopic Materials and Electronic Devices of the Université Catholique de Louvain. He is author or co-author of more than 100 scientific articles.

Thomas Pardoen is Belgian, born in the US in 1971. He graduated from the Université Catholique de Louvain, Louvain-la-Neuve, Belgium, in 1994 as a materials science engineer, in 1996 with a degree in philosophy, and was awarded a PhD from the same university in 1998 for a the thesis on the micromechanics of ductile fracture in copper. During a 2 years post-doctoral visit at Harvard University in the solids mechanics group of the Division of Engineering and Applied Sciences, his research activities diversified to include plasticity and fracture of multiphase materials, adhesive bonding, mechanics of thin films, and fracture in rate dependent materials. Since September 2000, T. Pardoen has joined the faculty of the Université Catholique de Louvain in the Department of Materials and Processes Sciences, supervising researches and teaching in the field of the mechanics and micromechanics of materials and microsystems from both an experimental and modelling point of view. T. Pardoen has been elected FEMS lecturer for 2002–2003 by the Federation of European Materials Societies. He is a member of the Research Center in Micro and Nanoscopic Materials and Electronic Devices of the Université Catholique de Louvain.