

Université catholique de Louvain Faculté des Sciences Appliquées

## MEASUREMENT, OPTIMIZATION AND MULTISCALE MODELING OF SILICON WAFER BONDING INTERFACE FRACTURE RESISTANCE

Thèse présentée par **Yannick Bertholet** en vue de l'obtention du grade de Docteur en Sciences Appliquées

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## Abstract

Wafer bonding is a process by which two or more mirror-polished flat surfaces are joined together. This process is increasingly used in microelectronics and microsystems industries as a key fabrication technique for various applications: production of SOI wafers, pressure sensors, accelerometers and all sorts of advanced MEMS.

Unfortunately, the lack of reliability of these systems does not allow them to enter the production market. This lack of reliability is often related to the lack of understanding and control of the thermo-mechanical properties of materials used for the fabrication of MEMS (indeed, at this small scale, properties of materials are sometimes quite different than at large scale) but it is also due to the limited knowledge of the different phenomena occurring during the working of these devices, the most detrimental of them being fracture. Among all of these fracture processes, the integrity of the interfaces and, particularly, the interfaces created by wafer bonding is a generic problem with significant technological relevance.

In order to understand the bonding behavior of silicon wafers, the interface chemistry occurring during the different steps of the bonding process has been detailed. The formation of strong covalent bonds across the two surfaces is responsible of the high fracture resistance of "wafer bonding" interfaces after appropriate surface treatments and annealing. The bonding process (surface treatments and annealing step) has been optimized toward reaching the best combination of interface toughness and bonding uniformity.

The fracture resistance of "wafer bonding" interfaces or interface toughness has been determined using a steady-state method developed in the framework of this thesis.

The high sensitivity to geometrical and environmental factors of "wafer bonding"

interfaces has been quantified and related to the interface chemistry.

A new technique involving the insertion of a dissipative ductile interlayer between the silicon substrate and the top silicon oxide has been proposed in order to increase the overall fracture resistance. A multiscale modeling strategy which involves the description of the interface fracture at the atomic scale, of the plasticity in the thin interlayer at the microscopic scale, and of the macroscopic structure of specimen has been used to guide the optimization of this technique. Numerical simulations have shown the influence of the ductile interlayer parameters (yield strength, workhardening exponent and thickness) and the critical strength of the interface on the overall toughness of such assemblies.

A first set of experimental data has allowed increasing the interface toughness by 70%.

The critical strength of the interface is finally determined by inverse identification and turns out to be in the expected range of theoretical strength.

The knowledge of the strength and the fracture toughness of "wafer bonding" interfaces is of practical importance because these two values can be used in a simple fracture model (e.g. cohesive-zone model) in order to observe the behavior of such interfaces under complex loading using finite element simulations.

## Remerciements

Un définition trouvée sur le web mentionne que :

"Le promoteur de thèse, sollicité en raison d'une maîtrise reconnue du champ de recherche concerné, doit aider le doctorant à dégager le caractère novateur dans le contexte scientifique et s'assurer de son actualité ; il doit également s'assurer que le doctorant fait preuve d'esprit d'innovation"

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Chapter 1

## Introduction

Molecular bonding is a process by which two mirror-polished wafers adhere to each other at room temperature without the application of any macroscopic gluing layer [1]. Silicon wafer bonding is the main application of molecular bonding in modern technology, specifically in microelectronics and microsystems technology. Although wafer bonding is extensively used nowadays, the basic phenomenon has been known and used for many centuries. The history of present-day wafer bonding has been very well reviewed in Refs. [2, 3] and is summarized in the next section.

## Molecular bonding history

Around 1230-1240, the Franciscan friar Bartholomaeus Anglicus, one of the medieval encyclopaedists digesting and compiling other sources, stressed the importance of cleanliness when joining silver and gold [4] : "When a plate of gold is to be merged with or joined to a plate of silver, one needs to keep in mind three things: dust, air and moisture. If any dust, air or moisture comes between the two plates, they cannot be joined together, the one to the other. Therefore it is necessary to join these two metals together in a completely clean and still place. And when they are joined together in such a manner, they become so inseparable that they cannot later be taken apart."

Around 1630, Galilei argued that two completely plane, smooth and polished plates of marble, metal or glass, one placed on top of the other would adhere to each other if one tries to lift the upper whereas two plates with rough surfaces would not [5].

In  $18^{th}$  century, Desagulier showed that two spheres of lead, when pressed together,

strongly adhere to each other [6]. In that case, plastic deformation allows the two bodies to be in intimate contact and thus to enable strong metallic bonding. This type of bonding called cold welding is not a desirable option for brittle materials. However, the observation, reported by Desagulier in 1734, that friction between sliding surfaces decreased with decreasing surface roughness until the surfaces became so smoothly polished that the adhesion between two bodies dramatically increased the friction [7], indicated that a better polishing technology would make brittle materials also bondable.

In the last century, the phenomenon that optically polished bulk pieces of metals used for precision length measurements stick to each other was observed by German craftsmen. The analogous phenomenon was also found for optically polished glass such as precision prisms.

In 1930, Obreimoff [8] started his report on the surface energy of mica with a reference to glass brought in optical contact, stated that the analogue can be observed when two freshly split foils of mica are brought in contact.

In 1936, Lord Rayleigh [9] reported probably the first thorough scientific study of room temperature adherence between glass slides. He found that the interaction energy per unit area was on the order of  $100 \text{ mJ/m}^2$ .

For a long time, this adhesion phenomenon did not have any noticeable technological impact except in the well-known traditional area of optics and for some isolated specific applications : He-Ne gas lasers were fabricated with mirrors bonded to the body of fused silica [2], direct bonding was utilized as a tool for the transfer of epitaxial layer used for the fabrication of transmission photocathode [10].

### Technological relevance:

## On the use of wafer bonding in microelectronics and microsystems

In spite of long history of the direct bonding observations, the modern development of science and technology has only been stirred by reports on silicon-silicon wafer bonding in the mid-eighties [11, 12, 13]. These reports concern the use of wafer bonding as substitute process for the growth of thick epitaxial layers of singlecrystalline silicon on silicon for potential application in power devices and use of wafer bonding for the preparation of Silicon-On-Insulator (SOI) structures. These two important processes are illustrated in Figure 1.1.



Figure 1.1 : Schematic of bonded and thinned silicon wafers (a) for replacement of an epitaxial layer and (b) for Silicon-On-Insulator (SOI) structure.

Parallel to the electronics-related work, wafer bonding has been also used to produce micro-electromechanical systems (MEMS). The primary goal of a MEMS is to integrate onto the same wafer the active parts of the system (actuators, sensors, ...) and the associated electronics, hence reducing the size and the cost of these systems. Pressure sensors are one type of MEMS using silicon wafer bonding as a step of their fabrication process, as sketched in Figure 1.2.



Figure 1.2 : Schematic of the fabrication steps of a pressure sensor involving wafer bonding.

Nowadays, wafer bonding is used for fabricating more and more complex structures by bonding several plain or patterned wafers. Multi-wafers bonding allows the fabrication of 3D structures like the miniature high pressure bipropellant rocket engine shown in Figure 1.3 [14].



Figure 1.3 : Fabrication of a high pressure bipropellant rocket engine (from [14]).

Moreover, wafer bonding is also used in a many other domains like high-voltage and high power devices, micromechanics (pressure sensors, accelerometers, microvalve, ...), optoelectronics, packaging, ...

These many examples show that wafer bonding is becoming a prominent technique in microfabrication process of microsystems.

## Importance of reliability of interfaces in MEMS

A 2000 to 2005 years market analysis of microsystems made by the Network of Excellence in Multifunctional Microsystems (NEXUS) task force [15, 16] shows that the Microsytem Technologies (MST) market is forecast to rise by 20% annually as shown in Figure 1.4.



Figure 1.4 : Evolution of the market size for MST products (from [15]).

The graph shows that the total world market for microsystems was expected to grow from \$ 30 billion in 2000 to \$ 68 billion by 2005. Prediction forecast tends to show that the market will grow to \$ 200 billion by 2010 [17].

Unfortunately, most MEMS fail to enter the production market due to the lack of mechanical reliability. Indeed, in the particular field of MEMS, parts of the structures undergo various kind of loading such as thermal stress, friction, impact, fatigue, ... and fracture of the moving parts often takes place during the process or in the early stages of service. This lack of reliability is often related to the lack of understanding and control of the thermo-mechanical properties of materials used for the fabrication of MEMS (indeed, at this small scale, properties of materials are sometimes quite different than at large scale) but it is also due to the limited knowledge of the different phenomena occurring during the working of these devices, the most detrimental of these phenomena being fracture.

Fracture processes occurring in MEMS, as shown in Figure 1.5, can originate from sources such as bad handling, creep, fatigue, wear, environmental attack, ... and can occur in different parts of the MEMS (fracture of thin films due to internal or external stress, fracture of beam anchor point, delamination of thin films due to environmental attack, ...).



Figure 1.5 : Example of fracture in MEMS (from [18]).

Among all of these fracture processes, the integrity of the interfaces and, particularly, the interfaces created by wafer bonding is a generic problem with significant technological relevance.

### Object and outline of the thesis

The goal of this thesis is to contribute to a better understanding and control of the fracture resistance of the "wafer bonding" interfaces in order to guide the bonding process and to assess the integrity of the structures.

The focus will be on :

- 1. the development of a robust experimental method to test interface fracture resistance,
- 2. the optimization of the bonding process,
- 3. the development of a better understanding of the chemical, physical and mechanical factors affecting the interface toughness,
- 4. the development of a method to improve the interface fracture toughness of the bonding by the insertion of a ductile interlayer near the interface,
- 5. the use of modeling and simulation tools to quantify this improvement of interface fracture toughness.

In order to achieve these objectives, interface fracture mechanics was intensively used in conjunction with microfabrication techniques. In the second chapter of the thesis, materials and microfabrication techniques are presented. A brief review of the mechanical properties of the materials and their fabrication processes are shown in the first part of this chapter. The second part of this chapter deals with the chemistry of the bonding process that is essential to understand and explain the different fracture behaviors of "wafer bonding" interfaces.

At the center of our methodology lies the theory of interface fracture mechanics which will support both the testing and modeling. So, the first section of the third chapter will provide a short overview of that theory. The next section of this chapter deals with the mechanical test procedure developed to measure fracture resistance properties of interfaces created by the wafer bonding process. Particularly, the wedge test and the tensile test are presented and detailed. Two different versions of the wedge test were setup, depending on the method used to evaluate crack length.

The fourth chapter is devoted to the optimization of the wafer bonding process. In this chapter, the experimental conditions used to obtain the best interfaces are established.

In the fifth chapter, the influence of external parameters (environment, geometry, ...) on the bonding resistance are presented.

Finally, in Chapter 6, the influence of a ductile interlayer inserted near the interface is studied owing to coupling experimental results with simulation tools.

Chapter	2
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# Materials and microfabrication techniques

Wafer bonding generally refers to the direct bonding of two silicon wafers, even though other type of materials are currently assembled in the microelectronic industry (glass on silicon, GaAs, ...). The first part of this chapter is dedicated to the presentation of the different materials addressed in this thesis. The fabrication processes and mechanical properties are detailed in a first section. The different steps of the samples fabrication are reviewed in the second section of this chapter. For each step of the bonding process, the chemical reactions occurring between two silicon wafers and responsible for the bonding are described.

### 2.1 Materials

#### 2.1.1 The silicon substrate

Solid state electronics began in 1947 as an applied research project at what was the Bell laboratories [19]. The assignment was to develop a solid state device to replace traditional vacuum tubes as switching and amplifying devices. The material selected for this study project, polycrystalline germanium, was chosen based on its position in the periodic table, specifically its electron configuration. In addition to germanium, another element in the same group IV family, silicon, was also under investigation as a potential material for semiconducting devices. By the early 1950's, the success achieved with single-crystal silicon had allowed it to win out over germanium as the material of choice, for several reasons. Its bandgap, the electron energy level separation between a conducting state and a non-conducting state is larger than the bandgap of germanium, making feasible to operate devices at a higher temperature. Oxidation of silicon produces a water-insoluble oxide that is suitable as both an impermeable diffusion mask and a high-quality insulator material between conducting layers; germanium oxide, on the other hand, is watersoluble. Finally, sources of high-purity silicon are readily available: the starting material for semiconductor-grade silicon is beach sand.

#### Fabrication of single-crystal silicon : from sand to silicon

Silicon used for microelectronics manufacturing comes from quartz. To achieve electronically acceptable grades of silicon, quartz undergoes a series of purification processes. Mineral quartz is first reacted with carbon, obtained from coke or coal, to make metallurgical grade silicon:

$$SiO_2 + 2C \xrightarrow{2000^\circ C} Si + 2CO$$
 (2.1)

A small portion of this metallurgical silicon is further purified by reaction with hydrochloric acid, yielding a mixture of chlorosilanes:

$$Si + xHCl \xrightarrow{approx 1250^{\circ}C} SiH_{x-2}Cl_x + H_2$$
 (2.2)

Trichlorosilane is separated from this mixture and is reduced with hydrogen to form ultrapure silicon:

$$SiHCl_3 + H_2 \xrightarrow{1100^{\circ}C} Si + 3HCl$$
 (2.3)

Silicon produced with this sequence of reactions has only trace impurities, typically below the parts per trillion range, and it becomes the source material for growing single-crystal silicon (by using either Czochralski (CZ) process or the float zone (FZ) process).

Czochralski silicon is manufactured by melting the silicon (produced according to Eq. (2.3)) in a resistant or radio frequency heated quartz crucible. A seed of desired crystallographic orientation ( $\langle 100 \rangle$  or  $\langle 111 \rangle$ ) is dipped into the molten silicon and slowly withdrawn with a rotational motion as sketched in Figure 2.1.



Figure 2.1 : Liquid Encapsulated Czochrazski crystal pulling chamber.

The float zone (FZ) process is another method for growing single-crystal silicon. This process is presented in Figure 2.2.



Figure 2.2 : The float zone layout for the needle-eye technique.

It involves the passing of a molten zone through a polysilicon rod that approximately has the same dimensions as the final ingot. The purity of an ingot produced by the FZ process is higher than that of an ingot produced by the CZ process. As such, devices that require ultrapure starting silicon substrates should use wafers produced using the FZ method. However, float zone crystal growing cannot produce the large diameters capable with the CZ process and the CZ process is also more economical and takes less time than the float zone process does. The result of theses processes is a long rod of single crystal silicon as shown in Figure 2.3.



(a) Rods of single silicon crystal (b) Rod with slices of single silicon crystal

Figure 2.3 : Single crystal silicon rods.

At the conclusion of these processes, the single-crystal silicon rod is cooled, and high-speed mechanical saws slice individual wafers of pure silicon from the rod. Further mechanical and chemical processes prepare the product wafers for semiconductor fabrication. These include grinding, lapping, chemical etching for surface impurities removal, polishing and final cleaning to achieve a wafer with exact diameter, flatness, co-parallel front and back surfaces, and surface smoothness.

#### Mechanical properties of silicon

As this work focuses on the mechanics of "wafer bonding" interfaces, only the mechanical properties of silicon will be presented.

Silicon crystallizes in the Diamond structure like diamond and germanium. This



crystallographic structure is presented in Figure 2.4.

Figure 2.4 : Crystallographic structure of silicon

Silicon is a highly anisotropic material and its mechanical properties depend on the crystalline orientation.

The general relationship between stress and strain is:

$$\sigma_{ij} = C_{ijkl} \epsilon_{kl}^{el} \tag{2.4}$$

where  $C_{ijkl}$  is the second order stiffness tensor,  $\epsilon_{kl}$  is strain and  $\sigma_{ij}$  is stress.

For convenience short hand matrix notation can be used. The notation takes use of symmetry relationship ( $\epsilon_{12} = \epsilon_{21}, \epsilon_{13} = \epsilon_{31}$  and  $\epsilon_{23} = \epsilon_{32}$ ) between shear stress to write  $\epsilon_{11} \rightarrow \epsilon_1, \epsilon_{22} \rightarrow \epsilon_2, \epsilon_{33} \rightarrow \epsilon_3, \epsilon_{32} = \epsilon_{23} \rightarrow \epsilon_4/2, \epsilon_{31} = \epsilon_{13} \rightarrow \epsilon_5/2, \epsilon_{21} = \epsilon_{12} \rightarrow \epsilon_6/2$ . With this notation, Eq. (2.4) can be rewritten as

$$\sigma_i = C_{ij} \epsilon_j^{el} \tag{2.5}$$

with

$$C_{ij} = \begin{pmatrix} C_{11} & C_{12} & C_{13} & C_{14} & C_{15} & C_{16} \\ C_{21} & C_{22} & C_{23} & C_{24} & C_{25} & C_{26} \\ C_{31} & C_{32} & C_{33} & C_{34} & C_{35} & C_{36} \\ C_{41} & C_{42} & C_{43} & C_{44} & C_{45} & C_{46} \\ C_{51} & C_{52} & C_{53} & C_{54} & C_{55} & C_{56} \\ C_{61} & C_{62} & C_{63} & C_{64} & C_{65} & C_{66} \end{pmatrix}.$$

$$(2.6)$$

For cubic systems, the stiffness matrix  $C_{ij}$  in [100]-crystal axes (i.e the axes defined in Figure 2.4) can be reduced, taking into account for the crystalline symmetry, to

$$C_{ij} = \begin{pmatrix} C_{11} & C_{12} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{12} & C_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{44} \end{pmatrix}$$
(2.7)

The relationship (2.7) allows direct calculations of the effective Young's modulus associated to a specific direction [hkl] from the three known elastic constants,  $C_{11}$ ,  $C_{12}$  and  $C_{44}$ .

We can write that [20]:

$$\frac{1}{E_{[hkl]}} = \frac{\epsilon_{hkl}^{el}}{\sigma_{hkl}} = S_{11} + \frac{\left(2S_{12} - 2S_{11} + S_{44}\right)\left(k^2l^2 + h^2l^2 + k^2h^2\right)}{\left(h^2 + k^2 + l^2\right)^2}$$
(2.8)

where  $S_{ij}$  are the components of the compliance tensor and are equal to :

$$S_{11} = \frac{C_{11} + C_{12}}{(C_{11} - C_{12})(C_{11} + 2C_{12})}$$
(2.9)

$$S_{12} = \frac{-C_{12}}{(C_{11} - C_{12})(C_{11} + 2C_{12})}$$
(2.10)

$$S_{44} = \frac{1}{C_{44}} \tag{2.11}$$

The values of the stiffness tensor components for silicon are respectively equal to  $C_{11}=166$  GPa,  $C_{12}=64$  GPa and  $C_{44}=80$  GPa. The Young's modulus in the [100] direction is thus equal to  $E_{[100]} = \sigma_{[100]}/\epsilon_{[100]}^{el}$ . This gives:

$$E_{[100]} = \frac{1}{S_{11}} = \frac{(C_{11} - C_{12})}{(C_{11} + 2C_{12})C_{11} + C_{12}} = 130 \text{ GPa.}$$
(2.12)

The values of Young's modulus to [110]- and [111]-direction are  $E_{[110]}=168$  GPa and  $E_{[111]}=187$  GPa respectively. The Poisson ratios can similarly be obtained as:

$$\nu_{[100]} = \frac{C_{12}}{C_{11} + C_{12}} = 0.28.$$
(2.13)

More details about the calculation of the Young's modulus and Poisson ratio of anisotropic silicon can be found in [20, 21].
The silicon wafers used in this thesis are [100] silicon wafers. Each silicon wafer has a flat located in the circumference of the wafer. The flat of the wafer indicates the type and the orientation of crystal. The crystallographic directions associated with this type of silicon wafers are represented in Figure 2.5.



Figure 2.5 : (a) Crystallographic directions of the [100] silicon wafers used in this thesis, (b) corresponding Young's modulus.

Unfortunately, dealing with anisotropic properties makes the interpretation of mechanical test much more difficult. It is thus convenient to consider silicon as an isotropic material, at least in the plane of the wafer. In most cubic crystal, the isotropic modulus, noted E in the following, is usually close to the value of  $E_{[110]}$ that is equal to 168 GPa for silicon [20]. A value of E=165 GPa is generally reported for isotropic silicon [22]. This latest value was adopted and is then used in the present work. The isotropic value of the Poisson ratio of silicon used in the simulations (see Chapter 6) was arbitrarily chosen equal to 0.3.

## 2.1.2 The thin layers

In the framework of this thesis, two categories of thin layers have been used. The first category consists of the two majors types of silicon oxide encountered in microelectronics i.e. thermal oxide and PECVD oxide. The second category of thin layers consists of aluminum thin layers. They are used to increase the global toughness of the assembly as explained in more details in Chapter 6.

## 2.1.2.a SILICON OXIDE

In order to mimic layers that are present in MEMS, silicon wafers are often covered with silicon oxide. The silicon oxide layer is sometimes used as an insulator barrier in thin film device or in the MOSFET transistor as grid oxide. In our particular case of wafer bonding process, silicon oxide can also be used to generate chemical reactive species that favor bonding between the two silicon wafers as detailed in the third part of this chapter.

Silicon oxide is deposited on top of the silicon wafer by chemical vapor deposition (CVD). In a typical CVD process the substrate is exposed to one or more volatile precursors, which react and/or decompose on the substrate surface to produce the desired material. Frequently, volatile byproducts are also produced, which are removed by gas flow through the reaction chamber. Two types of silicon oxide can be made by CVD. The first type is thermal oxide and the second one is PECVD (Plasma Enhanced Chemical Vapor Deposition) oxide. These two techniques are presented in the two next sections.

## The thermal silicon oxide

Thermal oxide is obtained by the transformation of Si in  $SiO_2$  at high temperature in presence of  $O_2$  or  $H_2O$  vapor:

$$Si(s) + O_2(g) \xrightarrow{700-1200^{\circ}C} SiO2(s),$$
 (2.14)

$$Si(s) + 2H_2O(g) \xrightarrow{700-1200^{\circ}C} SiO2(s) + 2H_2(g).$$
 (2.15)

Overall factors affecting wafer oxidation include temperature, pressure, availability of the oxidizing species in proper chemical stoichiometry to insure complete oxidation, and surface energy of the wafer where oxidation occurs. Reactant oxygen atoms diffuse through just-grown  $SiO_2$  film to further oxidize underlying silicon atoms. The interface between oxidized silicon and underlying unoxidized silicon progresses down into the wafer as oxidation proceeds, 44% of the grown oxide thickness layer taking the place of silicon substrate. More informations about the formation of thermal oxide can be found in Ref. [23].

## Properties of thermal silicon oxide

The thermal silicon oxide used in this thesis is obtained by wet oxidation at 1000°C. The thickness of this type of oxide depends on the oxidation time. Different thickness have been used in this work and they will be systematically detailed. The RMS roughness of this type of oxide is about 2.5 Å [24]. This low roughness allows good prebonding as explained in Section 5.2.1. The mechanical properties of thermal oxide have been measured by nanoindentation (see Appendix B). The Young's modulus of a thermal oxide is about 80 GPa and the hardness is about 10 GPa. As many deposited materials, thermal silicon oxide contains residual stress. The residual stress of 400 nm thermal oxide layer is equal to about -331 MPa [25]. As in the case of strained native oxide where the internal stresses increase the chemical reactivity [1], the internal stresses present in the thermal oxide layer are expected to increase the reactivity of these oxides.

The growth of thermal silicon oxide requires high temperature and this is not compatible with CMOS technology. Moreover, it is not possible to deposit this type of oxide on an another substrate than silicon. In order to solve this problem, PECVD oxide has also been investigated.

## The PECVD silicon oxide

The Plasma-Enhanced CVD (PECVD) process utilizes a plasma to enhance chemical reaction rates of the precursors and then allows deposition at lower temperatures.

Inside a PECVD reactor, radio frequency discharges occur between two opposite electrodes. As a result of plasma-chemical decaying of gases, various radicals are formed and a thin film is deposited on the substrate. The PECVD silicon oxide deposited on top of silicon substrate is obtained by the continuous circulation of a gas  $(SiH_4)$  on top of the silicon wafers. Gases react to form a  $SiO_2$  layer on the silicon wafer. The reaction between gases can be written as

$$SiH_4(g) + 2O_2(g) \xrightarrow{300-500^{\circ}C} SiO_2(s) + 2H_2O(g).$$
 (2.16)

## Properties of the PECVD oxide

The PECVD silicon oxide used in this thesis is produced at 450°C. The thickness of this type of oxide depends on the oxidation time and will be systematically mentioned. The roughness of this type of oxide is very high (about 15 Å [24]) and the prebonding step cannot occur. In order to circumvent this problem, wafers covered by PECVD silicon oxide must be subjected to a Chemico-Mechanical Polishing (CMP) treatment. The polishing decreases the roughness down to about 3 Å. This level of roughness allows proper prebonding between the two wafers. The mechanical properties of thermal oxide have been measured by nanoindentation (see Appendix B). The Young's modulus is about 90 GPa and the hardness about 10 GPa. As for the thermal silicon oxide, the PECVD oxide undergoes residual stresses which are expected to increase the reactivity of these oxides. For information, the residual stress of 300 nm PECVD silicon oxide layer is about -395 MPa [25].

A second type of layer is also deposited on the silicon substrate. Indeed, a thin ductile layer deposited between the silicon substrate and the silicon oxide can increase the fracture resistance of the assembly as explained in more details in Chapter 6. The chosen ductile material was aluminum.

## 2.1.2.b ALUMINUM

Unlike silicon oxide, aluminum is deposited using PVD (Physical Vapor Deposition) technique. Physical vapor deposition is a technique whereby physical process such as evaporation, sublimation or ionic impingement on a target facilitates the transfer of atoms from a solid or molten source onto a substrate.

Evaporation and sputtering are the two PVD techniques used at UCL to deposit thin aluminum layers. They are presented in the two next sections.

#### Deposition of a thin aluminum layer by evaporation

Figure 2.6 schematically illustrates the basic features of evaporative deposition. In this process, thermal energy is supplied to a aluminum source from which atoms are evaporated for deposition onto a silicon substrate. Heating of the source material can be accomplished by several methods. The simplest method is resistance heating of wire or stripe of refractory metal to which the material to be evaporated is attached. The evaporated atoms travel through reduced background pressure ( $< 10^{-6}$  Pa) in the evaporation chamber and condense on the growing surface.



Figure 2.6 : Schematic of the basic features of an evaporative deposition system.

## Properties of evaporated aluminum

The thickness of aluminum layers deposited by the evaporation process can range from 100 nm to 2  $\mu$ m. The mechanical properties of this type of aluminum were determined by nanoindentation (see Appendix B) leading to a Young's modulus about 80 GPa and a hardness equal to 0.3 GPa. A common simple rule allows the determination of the yield stress from the hardness [26]:

$$\sigma_y \approx \frac{H}{3}.\tag{2.17}$$

So, the yield stress of this aluminum is about 100 MPa and in good agreement with values from literature [23, 24]. The roughness is about 120 Å.

## Deposition of a thin aluminum layer by sputtering

In sputter deposition, ions of a sputtering gas, typically argon, are accelerated at high speed toward the target by an imposed electric field. Gaseous ions striking the target or the source material from which the film is made dislodge surface atoms which form the vapor in the chamber. These atoms transit through the discharge and condense onto the substrate, thus providing film growth. Figure 2.7 schematically shows the basic elements of a sputter deposition system. Several sputtering methods are widely used for the deposition of thin films in different applications:(i)



Figure 2.7 : Schematic of the basic features of a DC sputter deposition chamber.

DC sputtering, (ii) radio frequency sputtering with frequencies typically in the 5-30 MHz range, (iii) magnetron sputtering, where a magnetic field is applied in a superposition with a parallel or perpendicularly oriented electric field between the substrate and the target source and (iv) bias sputtering, where either a negative DC or RF (radio frequency) bias voltage is applied to the substrate so as to vary the energy and flux of the incident charged species.

## Properties of sputtered aluminum

The source used in the sputtering process is made of 99% of aluminum and 1% of silicon. The thickness of aluminum layers deposited by the sputtering process can range from 100 nm to 2  $\mu$ m, depending on the exposure time. The mechanical properties of these sputtered aluminum layers were determined by nanoindentation (see Appendix B). The value of the hardness using the constant Young's modulus assumption (see Appendix B), is equal to 600 MPa leading to a yield stress (according to Eq. (2.17)) of 200 MPa. The highest hardness and thus yield stress of sputtered aluminum layers is due to the presence of silicon atoms in the aluminum crystallographic network that decrease the mobility of dislocations into the aluminum network. The roughness of the sputtered aluminum layer is lower than the evaporated aluminum and about 5 Å.

## 2.2 Bonding Process

The first step in the wafer direct bonding process is the bonding (contacting) at room temperature. Since there is no adhesive involved in the wafer bonding process, surface forces between the wafers determine the adhesion. Therefore, a knowledge of surface forces and the resulting interaction between mating wafers is necessary to understand the direct bonding process. Numerous interactions occur between two surfaces. Depending on the environmental conditions (e.g. vacuum, vapors, liquids), different forces can be dominating. There are mainly three types of surface forces acting between two solids in sufficient proximity:

- van der Waals attraction forces that originate from atomic or molecular electric dipole (polarized or polarizable atoms or molecules) whose orientations are correlated in such a way that they attract each other,
- electrostatic Coulombic forces occurring when the surfaces become macroscopically charged by either adsorbing or desorbing electrons or ions,
- capillarity forces acting when two surfaces are in a vapor which can condense on the surface of the two wafers. The narrow gap between the two wafers is filled by a capillarity-condensed liquid, resulting in an additional capillarity attractive force.

Figure 2.8 shows schematically these three types of interactions.



Figure 2.8 : Schematic of three types of interactions between two solids.

Depending on the nature of the cleaning prior bonding, the silicon surface can be either hydrophilic or hydrophobic. Hydrophilic bonding occurs when the surface are covered with an oxide (native or "artificial") that reacts with water to form silanol (Si - OH) groups following relationship (2.18):

$$Si - O - Si + H_2O \longleftrightarrow 2SiOH$$
 (2.18)

The silanol groups are the precursor of bonding between the two wafers as explained in more details in the next section. Hydrophobic bonding occurs between wafers that are not covered with any oxide. Room temperature bonding usually occurs due to the presence of Si - H bonds on top of the silicon wafers. Figure 2.9 compares hydrophilic and hydrophobic bonding as a function of annealing temperature.



Figure 2.9 : Surface energy of direct bonded wafers as a function of annealing temperature for hydrophilic (HL) and hydrophobic (HB) wafers (from [1]).

In order to allow compatibility with CMOS technology used for the electronic parts of MEMS (maximum temperature of 450°C), it appears that it is preferable to work with hydrophilic bonding (silicon wafers covered by an oxide layer) which allows to reach good adhesion at moderately low annealing temperatures. Consequently, we focused only on hydrophilic bonding.

The bonding process between two hydrophilic wafers can be divided into three steps which will be detailed next:

- activation of the silicon oxide top layer
- contact and storage of the wafers
- annealing of the assembly

## 2.2.1 Cleaning of the wafers

Wafer bonding requires wafers with clean surfaces which are free of contaminants. These contaminants can be of different types as sketched in Figure 2.10.



Figure 2.10 : Possible surface contamination of silicon wafers.

The removal of these contaminants is an essential step because the surface cleanliness has a direct effect on both the electrical and structural properties of the bonded interface as well as on the resulting electrical properties of the bonded materials. The cleaning techniques employed prior to room-temperature wafer bonding must be able to remove all contamination on the surfaces without degrading surface smoothness. Hydrogen-peroxide based RCA wet cleans are most commonly used in the semiconductor industry and specifically in wafer bonding. The basic cleaning step involves two solutions:

- RCA 1 ( $NH_4OH:H_2O_2:H_2O=1:1:5$  to 1:2:7)
- RCA 2 (HCl: $H_2O_2$ : $H_2O=1:1:6$  to 1:2:8)

which are used sequentially [27]. The *RCA* 1 solution is used to remove particles, organics and some metals whereas alkali and heavy metals are removed by the *RCA* 2 solution.

The procedure used in the clean rooms of UCL is slightly different and it gives good results. This home-made cleaning procedure was used for most all of our samples preparation with a few exceptions that will be clearly notified.

The wafers are first dipped in a sulfuric solution  $(H_2SO_4(96\%) : H_2O_2(30\%) = 5 :$ 2) for 10 minutes at 100°C. Next, the wafers are rinsed in two separate DI (deionized) water containers for 5 minutes in each container. The wafers are dipped again in a second sulfuric solution. The native oxide which can contain impurities in the bulk or on the surface is then removed by dipping the wafers in fluorhydric acid (HF 2%) for about 15 seconds. In order to recover the hydrophilicity of the wafers, the wafers are dipped again in the second sulfuric solution. A clean "native" oxide of a few angströms is created. The wafers are finally rinsed and dried.

Wafers covered by thermal or PECVD oxide have been subjected to the same cleaning sequence prior the oxidation step. After the oxidation step, the wafers have been cleaned again following the same procedure except for the dipping in HF which remove the silicon oxide.

Wafers covered with an aluminum thin layer and a silicon oxide thin layer (see Chapter 6) are cleaned before aluminum deposition and after silicon oxide deposition except for the HF dipping.

This cleaning step is also used to promote the activation of the oxide top layer as explained in the next section.

## Influence of the cleaning step on the interface chemistry

If hydrogen bonding (between an hydrogen atom and a negative polarized atom, e.g, an oxygen atom) can be achieved across two mating surfaces, a strong dipole-dipole van der Waals attraction force will be exerted between these surfaces. Moreover when the mating surfaces are hydrophilic and water molecules are present, the linkage of two or three water molecules can bridge the gap between the two mating surfaces as sketched in Figure 2.11. This long-range hydrogen bonding will ease the



Figure 2.11 : Schematic of a linkage of three water molecules between two hydrophilic mating surfaces to bridge the wafers at RT (from [1]).

smoothness requirements of the mating surfaces for successful room temperature bonding.

The purpose of surface activation is thus to achieve a reactive surface with practically available flatness and smoothness for room temperature bonding. Since hydrogen bonding is a strong form of dipole-dipole attraction, a surface suitable for hydrogen bonding is highly desirable.

## Wet chemical activation of $SiO_2$ surface

Native oxide-covered or artificially oxidized silicon surface can be activated by wet chemical treatment based on the reaction between the silica network and  $H^+$  or  $OH^-$  groups [1]:

$$Si - O - Si + OH^{-} \longleftrightarrow Si - OH + Si - O^{-}$$
 (2.19)

$$Si - O^- + H_2 O \longleftrightarrow Si - OH + OH^-$$
 (2.20)

and

$$Si - O - Si + H_2O + H^+ \longleftrightarrow 2Si - OH + H^+.$$

$$(2.21)$$

The  $H^+$  and  $OH^-$  ions determine the surface hydrophilization rate. These two equations can be simplified as

$$Si - O - Si + H_2O \longleftrightarrow 2Si - OH.$$
 (2.22)

However, this equation is valid only when the surface siloxane bonds (Si - O - Si)are strained [1]. The native oxide formed by wet chemical cleaning has been found to be a strained oxide with a mean (Si - O - Si) angle of around 130°, deviating from its equilibrium value of 144° in bulk vitreous silica [1]. The bond angle deformation significantly increases the chemical reactivity of the siloxane bond. Therefore, the chemical reaction (2.22) readily occurs on the surface native oxide of silicon wafers. Thermal and PECVD oxides are also strained oxide. It is thus also expected that reaction (2.22) occurs readily on the surface of such oxides.

Two main types of silanol (Si - OH) groups can form on the surface: (a) isolated (or free) groups and (b) hydrogen-bonded (associated or vicinal) groups (see Figure 2.12).



Figure 2.12 : Two types of silanol groups on silicon surface covered with silicon oxide. (a) Isolated groups and (b) hydrogen-bonded groups (from[1]).

The latter occurs because adjacent silanol groups are close enough and are suitably oriented toward one another that they form hydrogen bonds. The surface hydroxyl (-OH) groups are polarized and are therefore reactive. They are the most important sites for the surface adsorption of water via hydrogen bonding.

A third type of silanol group, geminal silanol exists. It is represented in Figure 2.13. This type of silanol groups is less reactive because of steric hindrance.



Figure 2.13 : Geminal silanol group on silicon surface covered with silicon oxide.

#### Surface activation by plasma treatment

Plasma treatment can also activate the silicon surface [28]. The silicon wafers covered with native, thermal or PECVD oxide are put in a plasma produced by lowpressure gas electric discharges. The plasma-treated surface shows a significantly enhanced chemical reactivity with water to form silanol groups (Si - OH) which are the precursors of the bonding. It is believed that, in addition to cleaning the surface, plasma induces bond defects on the surface that are most likely responsible for the increased reactivity. Indeed, oxygen radicals generated from the  $O_2$  plasma atmosphere can be efficient enough to break any Si - OR bonds, where R is for hydrocarbon contamination. Oxygen radicals are also efficient enough to be adsorbed, leading to form Si - O - dangling bonds, and then to generate Si - OHbonds with adsorbed water. Moreover, several authors recommend to use water dipping after plasma treatments to enhance these effects [29, 30].

## 2.2.2 Contacting of the wafers

At the beginning of the thesis, samples were bonded manually. The lower wafer was placed on a special tool. This tool had three pins that cover the lower wafer. The upper wafer was contacted manually to the lower wafer, the three pins preventing the contact at the outer border of the wafers. As shown in Figure 2.14, the contact thus starts at the center of the wafers and propagates to the surface of the wafers when the three pins are removed. Then, the samples (made of two wafers) are stored during a given period of time with a dead weight. This storage step allows the reorganization of the chemical species present at the interface as extensively studied by Tong and Gosële [1]. The storage step was not performed in all cases. The manual contacting step did not allow a perfect alignment of the two wafers which should match as closely as possible (it is preferable to have perfectly aligned wafers because the fabrication of the specimens requires to cut rectangular



Figure 2.14 : Propagation of the contact wave starting at the center of the sample and propagating to the border (from [1]). Dark area is non-bonded and white area is bonded.

specimens from the entire bonded wafers). In order to circumvent this problem, the second type of samples were made using a mask aligner machine (the machine was purchased in the course of this work). Contacting was initiated at the center of the wafers as in the first type of samples but the alignment of the wafers was better than when manual bonding was performed.

## Influence of the contacting step on the interface chemistry

As shown in Figure 2.11, the bonding between the two wafers is due to the reaction of water with silanol groups present on the top surface. The interface chemical structures of hydrophilic Si/Si pairs immediately after contact in room temperature can be seen in Figure 2.15.



Figure 2.15 : Postulated interface chemical structures of hydrophilic Si/Si pairs immediately after RT contacting: (a) at an isolated silanol site and (b) at an associated silanol site (from [1]).

After contacting, three main processes take place during room-temperature storage:

- interface molecular water is continuously reacting with the bonding surface to generate more silanol groups (slow fracture) (see chemical reaction (2.22)),
- interface water molecules can rearrange themselves to reach an energetically more favorable state,
- some of the interface water molecules can migrate out of the bonding interface and/or can diffuse into the surrounding oxide.

These three effects bring the two mating surfaces closer and, finally, the two wafers are bridged together with hydrogen bonds without the presence of water as shown on Figure 2.16.



Figure 2.16 : Hydrogen bonds at interface of hydrophilic silicon bonding pairs after removal of molecular water: (a) at an isolated silanol site and at an (b) associated silanol site (from [1]).

Moreover, the last effect would allow silanol-silanol interaction (see Figure 2.16), that can promote the polymerization of the two silanol groups following:

$$Si - OH + Si - OH \longleftrightarrow Si - O - Si + H_2O$$
 (2.23)

This reaction can occur at room temperature if the local separation of the surfaces is lower than 4 Å. The polymerization of two silanol groups (each belonging to one silicon wafer) into a strong covalent bond is responsible of the high bonding energy between two silicon wafers. As shown in Figure 2.9, this polymerization is enhanced by temperature and an annealing step will thus greatly increase the fracture resistance of "wafer bonding" interfaces.

## 2.2.3 Annealing of the samples

After the first prebonding contact stage, samples have been annealed in order to increase the interface toughness. Low temperature annealing (until 150°C) has been performed in an oven whereas high temperature annealing has been performed in a furnace, both under inert gas atmosphere. Time and temperature annealing were different for each samples and are thus mentioned when necessary.

## Influence of the annealing step on the interface chemistry

As shown in Figure 2.9, the samples are annealed in order to form covalent bonds between the two mating surfaces (see Eq.(2.23)). The range of annealing temperature can be divided into three intervals.

Firstly, for temperatures below 110°C, the chemical reactions on the interface are the same as those proposed for RT bonding, i.e. formation of stable hydrogen bonding between molecular water across the bonding interface. The surface coverage of molecular water on the silica  $(SiO_2)$  surface depends on the relative humidity (RH). The bonding energy of room-temperature bonded vitreous silica wafers was found to decrease when bonding was performed at RH < 15% owing to a reduction in water coverage [1]. On the other hand, if RH is greater than 15%, the molecular water at the interface of hydrophilic Si/Si pairs does not change significantly during annealing in the temperature range from room temperature to 110°C since a mobile water film exists at this stage.

The main interface reactions in this temperature range are:

- the slow fracture effect of Si O Si bonds (reaction 2.23) on both bonding surfaces via attack by the interface water which leads to an increased number of -OH groups extending from the surface into the interface region;
- rearrangement of the interface molecular water to form more stable hydrogenbonded structures.

Secondly, between 110 and 150°C, interface toughness is rising and this can be explained by the fact that most of the molecular water in silica is removed upon heating to 120°C leading to polymerization of silanol groups across the interface. This tentative explanation is supported by the fact that the interface energy of bonded silicon pairs with hydrophobic wafers surfaces, which does not involve molecular water on their surfaces, does not change appreciably during the interval from RT to  $150^{\circ}$ C (see Figure 2.9).

Thirdly, from 150 to 450°C (maximum temperature in low temperature wafer bonding), the interface toughness exhibits a constant value (see Figure 2.9). In this area, the bonding energy is limited by the contacted area. It is believed that almost all silanol groups in the real contacted areas have been converted into siloxane bonds at 150°C. Since the bonding surfaces are never perfectly smooth, the area over which bonding really occurs (termed contacted area) is limited. The value of the contacted area can be improved by working in low vacuum.

During the second and the third interval, covalent bonds form and bridge the two wafers following Eq. (2.23).

During and after annealing, we can expect interface chemistry similar to the one presented in Figure 2.17.



Figure 2.17 : Chemical structure of hydrophilic pairs during annealing (from [1]).

If the annealing time is sufficiently long, the concentration of covalent bonds increases and an interface like the one depicted in Figure 2.18 is obtained.



Figure 2.18 : Chemical structure of hydrophilic pairs after annealing for a sufficient time (from [1]).

The three previous steps (contacting, storage and annealing) were imposed for all samples. However, optional treatments of the wafers were also applied to study special cases occurring in wafer bonding. These treatments are presented hereafter.

## 2.2.4 Optional treatments

## Plasma treatment of the wafers

In order to increase the reactivity of the oxide, wafers can be subjected to a plasma treatment. The plasma used to prepare our samples was an  $O_2$  plasma made in an Oxford (PlasmaLab System 100) machine. The characteristics (power, flux, pressure, time) of the plasma were modified in order to achieve the best bonding and are mentioned when necessary.

## Etching of the wafers

Samples with channels were processed in order to analyze the effect of the presence of patterns, and thus of unbonded areas, on the toughness of the final assembly. After a classical cleaning (described here above), the wafers were covered with a photoresine sensible to UV. A mask is then deposited on the wafers. The wafers are then subjected to UV and the photoresine exposed to UV is dissolved. The channels are then dry etched by the action of sulfur hexafluoride  $SF_6$  on the silicon and a vertical profile is obtained.

A lateral view of this type of samples is shown in Figure 2.19.



Figure 2.19 : Lateral view of samples with channels.

The size of the channels and the spacing between them were systematically varied. These geometrical characteristics will be detailed in Chapter 5 where the geometrical effects on the toughness will be addressed.

## **Chemico-Mechanical Polishing**

As already mentioned, PECVD silicon oxide is too rough for allowing enough prebonding adhesion. In order to decrease this roughness and to allow prebonding of wafers covered by PECVD silicon dioxide, a Chemico-Mechanical Process (CMP) process was performed by the company Kemesys from which no specific information about the process could be obtained. This treatment decreased the roughness of the PECVD silicon oxide from 15 Å to around 3 Å, an acceptable value for prebonding adhesion.

## 2.3 Preparation of the specimens

After the complete bonding process, the bonded wafers are cut into rectangular shape specimens in order to be tested using the steady-state wedge test presented in Chapter 3. The samples are cut in a parallel direction to the [010] direction defined in Figure 2.5.a. as depicted in Figure 2.20.



Figure 2.20: (a) Cutting of the four available specimens from the bonded wafers. (b) Front and lateral view of one specimen after cutting.

The cutting of the specimens was first performed by cleavage of the silicon wafers and then, using a diamond web saw (without and with water). No significant difference on the interface toughness of bonded wafers was observed between these different techniques.

Chapter	3
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## Interface fracture mechanics Theory and testing

One of the main goal of this thesis was to properly determine the fracture properties of bonded wafers interfaces. Fracture mechanics is the relevant framework for addressing cracking resistance.

The first section of this chapter is thus dedicated to a brief presentation of the fracture mechanics theory and its application to interfaces.

In a second section, a brief description of the parameters describing the fracture resistance of an interface is presented.

In a third section, the different tests used to determine the interface fracture resistance quantities are presented. The specific wedge-opening test and the tensile test setup will be described in more details. Results showing the advantages and drawbacks of these two mechanical tests are also presented in order to critically assess their relevance. The profilometer test that is an alternative to the wedge-opening test is also presented in this section. Validation of this test as an alternative to the wedge-opening test is then reviewed.

## **3.1** Fracture mechanics : a short review

This section is mainly based on references [20, 31, 32].

The first report on fracture mechanics originates from the stress analysis of Inglis (1913) [33] of an elliptical cavity in an uniformly stressed plate (see Figure 3.1).



Figure 3.1 : Plate containing an elliptical cavity with semi-axes a and b, subjected to uniform applied tension  $\sigma_{\infty}$ .

His analysis showed that the local stress at the tip of a sharp notch or corner rises to a level larger than the applied stress by an amount given by:

$$\sigma_c = \sigma_\infty \left( 1 + 2 \left( \frac{a}{b} \right) \right) \tag{3.1}$$

$$=\sigma_{\infty}\left(1+2\sqrt{\frac{a}{\rho}}\right) \tag{3.2}$$

with  $\rho = b^2/a$  is the radius of curvature. In the Inglis configuration, the limiting case of an infinitesimally narrow ellipse might be considered to represent a crack. However, in the case of an ideally sharp crack with  $\rho = 0$ , the stress,  $\sigma_c$ , becomes infinite and the presence of a crack produces a stress singularity in linear elastic solids. Of course, in real materials, the stress at any location cannot exceed a limit imposed by the occurrence of non-linear mechanisms.

Ignoring the existence of a non-linear zone, it is possible to determine the stress field at some distance from the crack tip. Considering the orientations defined in Figure 3.2, stress and displacement fields in region close to the crack tip (i.e. r < a/10) express as

$$\sigma_{ij} = \frac{K}{\sqrt{2\pi r}} f_{ij}(\theta), \qquad (3.3)$$

$$u_i = \frac{K}{2E} \sqrt{\left(\frac{r}{2\pi}\right) f_i(\theta)} \tag{3.4}$$

where  $\sigma_{ij}$  are the stresses,  $u_i$  the displacements,  $(r, \theta)$  the position from the crack tip,  $f_i$  and  $f_{ij}$  are geometrical functions that can be found in e.g. [31] and K is the stress intensity factor.



Figure 3.2 : Stress field around a crack tip showing rectangular and polar coordinates.

The K factors depend only on the outer boundary conditions, i.e. on the applied loading and specimen geometry, and consequently determine the intensity of the local field. Formulations for K factors can be found in [20, 31].

A major problem facing early workers in fracture mechanics was how to accommodate the essential elements of non-linearity and irreversibility (crack-tip singularity) in the linear elastic fracture mechanics framework. A significant advance was made by Irwin and Barenblatt who proposed to divide the crack system into two zones as in Figure 3.3 : the outer zone, linear elastic, transmits the applied loadings to the inner zone, where all energy absorption processes (including bond rupture) operate. The major assumption is that the size of the inner zone must be relatively small relative to the outer zone. The outer zone is then insensitive to events within the inner zone and linear elastic fracture mechanics may be retained.



Figure 3.3 : Irwin-Orowan small-scale zone model. Surface-separation processes are confined to a frontal zone (shaded) small compared to crack dimensions.

One of the first model representative of brittle fracture process occurring in the inner zone is the one of Barenblatt (1962) [31]. It recognizes the underlying atomic nature of the fracture process by specifying the resistance in terms of a non-linear traction-separation law of the type shown in Figure 3.4.



Figure 3.4 : Cohesive traction stress-separation function for two atom planes in brittle solids.

The continuum basis of the linear fracture mechanics is preserved by assuming the forces be distributed over a sufficiently long zone along the crack plane. Moreover, this cohesive-zone approach leads to removal of the singularity.

Until now, the cracked body has been analyzed under the scope of solid mechanics by examining the crack tip stress field. Cracking can also be addressed from an energetic point of view by looking at the energy fluxes taking place within the body during crack propagation. This approach of fracture was proposed the first time by Griffith. Griffith's idea [31] was to model a static crack as a reversible thermodynamic system. He simply sought the configuration that minimized the total free energy of the system; the crack would then be in a state of equilibrium, and thus on the verge of extension. The first step in the treatment is to write down an expression for the total energy U of the system. To do this, we consider the individual energy terms that are subjected to change as the crack is allowed to undergo virtual extension. Generally, the system energy associated with crack formation may be partitioned into mechanical and surface terms. The mechanical energy consists of two terms,  $U_M = U_E + U_A$  where  $U_E$  is the strain potential energy stored in the elastic medium;  $U_A$  is the potential energy of the outer applied loading system, expressible as the negative of the work associated with any displacement of the loading points. The surface term  $U_S$  is the free energy expended in creating the new crack surfaces. We may therefore write

$$U = U_M + U_S. \tag{3.5}$$

Thermodynamic equilibrium is then attained by balancing the mechanical and surface energy terms over a virtual crack extension, da. The mechanical energy will generally decrease as the crack extends  $(dU_M/da < 0)$  because the restraining tractions across the incremental crack boundary are suddenly relaxed, accelerating the crack until a new configuration of lower energy is reached. On the other hand, the surface energy term will generally increase with crack extension since cohesive forces of molecular attraction must be overcome during the creation of new surfaces  $(dU_S/da > 0)$ . Thus the first terms of Eq. (3.5) favors crack extension while the second opposites it. This is the *Griffith energy-balance concept*, a formal statement of which is given by the equilibrium requirement

$$\frac{dU}{da} = 0. \tag{3.6}$$

A crack would extend or retract reversibly for small displacements from the equilibrium length, according to whether the left-hand side of Eq. (3.6) is negative or positive

### The energy release rate

It is accordingly convenient to define a quantity called the mechanical-energy-release rate  $^1$  ,

$$G = -\frac{dU_M}{dA},\tag{3.7}$$

with A the crack interfacial area. Observe that G has the dimension of energy per unit area. For the special case of a straight crack, where length a is sufficient to define crack area, Eq. (3.7) may be reduced to an alternative, more common (but more restrictive) form

$$G = -\frac{dU_M}{da} \tag{3.8}$$

per unit width of crack front. When linear elasticity applies, the stress intensity factor K and the energy release rate G are univocally related. Both K and G characterize the crack tip stress concentrations. The relation between K and G is demonstrated in [20] and is:

$$G = \frac{K^2}{\overline{E}} \tag{3.9}$$

where

$$\overline{E} = \frac{E}{(1-\nu^2)}$$
 in plane strain, (3.10)

$$= E \quad \text{in plane stress.} \tag{3.11}$$

As previously mentioned, a crack will extend if

$$\frac{dU}{da} = \frac{dU_M}{da} + \frac{dU_S}{da} < 0. \tag{3.12}$$

Using the definition of G (Eq. (3.8)) in Eq. (3.6), we see that a crack extends if

$$G > \frac{dU_S}{da}.\tag{3.13}$$

We can now define a material property,  $G_c$ , as the critical energy release rate of the material. If  $G \ge G_c$ , crack will extend.

<sup>&</sup>lt;sup>1</sup>Rate relative to spatial crack coordinate, area or length, not time

 $G_c$  represents the fracture toughness of the material i.e. its resistance against crack extension. Note that, in principle,  $G_c$  is equal to twice the surface energy if fracture is purely brittle i.e when no dissipation mechanisms occur, i.e.

$$G_c = 2\gamma. \tag{3.14}$$

As a matter of fact, even in very brittle materials, surface reconstructions always lead to some discrepancies between  $G_c$  and  $2\gamma$  [31].

Physically, the fracture toughness,  $G_c$ , can be seen as the energy per unit area required to separate the material within the process zone. Because G and K are related through Eq. (3.9), we can also define the criterion for crack initiation in terms of K. If  $K \ge K_c$ , cracking will occur in the material. It is important to remember that, in spite of the notations,  $G_c$  and  $K_c$  are very different from G and K.  $G_c$  and  $K_c$  represent the toughness of the material and represent a property of a material or an interface. They are related to the micromechanisms occurring during crack propagation. On the other hand, G and K are mechanical concepts which depend on the loading, the geometry of the cracked specimen and on the details of the loading conditions.

## Application to interfaces

The concepts developed in the previous section can be applied when a crack opens and propagate along an interface between two elastic materials. In most of real cases, a complex distribution of load that can both open and shear the crack will provoke crack propagation as sketched in Figure 3.5. In that case, the stress field near the crack tip has the form:

$$\sigma_{ij}(r,\theta) = \frac{K_I}{\sqrt{2\pi r}} \Sigma_{ij}^I(\theta) + \frac{K_{II}}{\sqrt{2\pi r}} \Sigma_{ij}^{II}(\theta).$$
(3.15)

The index I and II are respectively associated to open and shear loads. By extension, a mode I crack propagation is due to opening loads acting on the crack whereas a mode II crack propagation is caused by shear loads. The  $\theta$ -dependent functions  $\Sigma_{ij}^{I}(\theta)$  and  $\Sigma_{ij}^{II}(\theta)$  can be found e.g in [31]. The stress intensity factors,  $K_{I}$  and  $K_{II}$ , scale the amplitudes of opening and shearing loads.

The mixed mode conditions are characterized by the two loading parameters  $K_I$ and  $K_{II}$  that transmit the remote loading conditions to the crack tip process. The relative amount of mode II to mode I is specified by the mode angle  $\psi$ , defined



Figure 3.5 : Crack lying along a bimaterial interface.

by:

$$\tan(\psi) = \frac{K_{II}}{K_I}.\tag{3.16}$$

A pure mode I crack corresponds to  $\psi=0$  and a pure mode II to  $\psi=\pm\pi/2$ . Using the relationship (3.9), it is possible to write the strain energy release rate of a mixed mode cracking configuration as :

$$G = \frac{1}{\overline{E}} (K_I^2 + K_{II}^2).$$
(3.17)

In our particular case of interfaces resulting from wafer bonding, it is possible to write the stresses on the plane  $\theta=0$  at a distance r ahead the crack tip :

$$\sigma_{22}(r,0) = \frac{K_I}{\sqrt{2\pi r}},$$
(3.18)

$$\sigma_{12}(r,0) = \frac{K_{II}}{\sqrt{2\pi r}}.$$
(3.19)

This stress field is similar to the one presented in Eq. (3.4) when pure mode I is considered.

In this thesis, only pure mode I will be encountered. Mixed mode cracking was extensively reviewed in a paper by Suo and Hutchinson [34].

# 3.2 Fracture mechanics : an atomistic point of view

If a crack propagates into a solid or along an interface as sketched in Figure 3.6(a), the evolution of the force needed to separate two atomic planes can be represented by the curve of Figure 3.6(b).



Figure 3.6 : Atomistic point of view of fracture mechanics.

First, the force increases linearly, reaches a maximum value and then drops to zero when the atomic bond is broken. This simple representation of crack propagation confines the bond rupture mechanisms to two interatomic layers that bound the crack plane. Atomic layers further from the crack plane deform following the same traction-separation law, but only up to a stress smaller than the maximum stress, and (not withstanding certain relaxation processes that occur when atomic structures on newly created surfaces rearrange themselves) reversibly, restoring to their intact configuration when the crack moves ahead. The apparent simplicity of this fracture representation can be directly applied to our wafer bonded interfaces because fracture along this interfaces is mostly brittle.

The cracking resistance of interfaces is then essentially characterized by two parameters [31, 35, 36]:

• the first one is the energy needed to propagate the crack. This is the area under the curve of Figure 3.6(b). It is expressed in  $J/m^2$  and it corresponds to the energy used to create 1 m<sup>2</sup> of new surface. When no dissipation mechanisms are present, the cracking energy will indistinctly be noted  $G_c$  or  $\Gamma_0$  in the rest of the document except when it specifically mentioned.

• the second one is the critical stress or interface strength. This is the maximum value of force/stress of Figure 3.6(b). It is expressed in Pa and it is noted  $\sigma_c$  in the rest of the document.

A lot of experimental techniques allow the determination of  $G_c$ . For reasons that will be explained later, we have chosen the wedge-opening test, presented in details hereafter. The direct measurement of the interface strength is much more controversial and only a test used to measure an overall strength of the interface, the tensile test, will be presented and critically discussed.

## Factors affecting the interface toughness

The overall or macroscopic fracture toughness,  $G_c$ , is influenced by several parameters which are presented briefly here below and detailed in the next chapters. The overall toughness of an interface can be written as:

$$G_c^{macro} = \Gamma_0 + \Gamma_p - G_{curvature} - G_{roughness} \tag{3.20}$$

 $\Gamma_0$  corresponds to the intrinsic interface toughness. It depends mainly on the chemical bonds linking the two wafers and the reaction of these chemical bonds with environmental species (water, ...) (see Chapter 5).  $\Gamma_p$  represents the plastic dissipation that can occur in an ductile layer lying near the interface when the interface is submitted to external loads.  $\Gamma_p$  depends mainly on geometric factors and mechanical properties of the ductile layer. The influence of  $\Gamma_p$  on the toughness will be presented in details in Chapter 6.  $G_{curvature}$  and  $G_{roughness}$  are terms that will decrease the global toughness. They are respectively due to the curvature and roughness of the wafers. Influence of the roughness and the curvature of the wafers on the toughness is presented in Chapter 5.

## 3.3 The wedge-opening test

Note: the description of the test and the justification of its relevance for the present problem have been given in [37].

The wedge opening test is used to determine the interface toughness,  $G_c$ , of the bonded wafers. The test consists of inserting a thin wedge between the two wafers and of promoting crack propagation at the interface.

First, mechanical concepts associated to the wedge test and leading to a formulation for  $G_c$  are presented. Then, a brief description of the measurement setup and the main advantages resulting from the use of this technique are reviewed. Finally, an alternative to the previous technique is presented.

## 3.3.1 Mechanics of the wedge-opening test

An important experiment undertaken by Obreimoff [8] in 1930 on the cleavage of mica can be considered as the ancestor of the wedge test. The basic arrangement used by Obreimoff is shown in Figure 3.7.



Figure 3.7 : Obreimoff's experiment on mica.

A wedge of thickness d is inserted beneath a thin flake of mica attached to a parent block, and is made to drive a crack along the cleavage plane. In this case, we may determine the energy of a cracked system by treating the cleavage lamina as a freely loaded cantilever of thickness h and width w, built-in at the crack front distant a from the point of application of the wedge. We note that allowing the crack to form under constant wedging conditions, the bending (line) force F suffers no displacement, so the net work done by this force is zero, i.e.

$$U_A = 0.$$
 (3.21)

At the same time, we have, from simple beam theory (see Appendix C for details), the elastic strain energy in the cantilever arm,

$$U_E = \frac{Ewh^3 d^2}{32a^3}.$$
 (3.22)

The mechanical energy-release rate G is defined by (see Eq. (3.8))

$$G = -\frac{dU_M}{dA} = -\frac{d(U_A + U_E)}{dA}$$
(3.23)

which reduces using Eq. (3.21) to

$$G = -\frac{dU_E}{dA} \tag{3.24}$$

where A the crack interfacial area.

For the special case of a straigth crack, the crack interfacial area A is then equal to

$$A = aw \tag{3.25}$$

with w, the width of the specimen. The mechanical energy release-rate can be rewritten as

$$G = -\frac{dU_E}{wda}.$$
(3.26)

Inserting Obreimoff's energy terms (Eq. (3.22)) into the definition of G (Eq. (3.26)), we obtain

$$G = \frac{3Eh^3d^2}{32a^4}$$
(3.27)

Let us now consider the wedge-opening test also called the double-cantilever beam test (DCB). A versatile system is that of the double cantilever, obtained by symmetrically pre-cracking a beam specimen as shown in Figure 3.8.



Figure 3.8 : Double cantilever beam (DCB) test specimen.

This specimen may be seen as an elaboration of the Obreimoff arrangement (see Figure 3.7). The elastic strain energy,  $U_E$ , stored in each arm of the specimen is then:

$$U_{E,i} = \frac{Ewh_i^3 d^2}{32a^3} \tag{3.28}$$

with i=1, 2 the index of the arm. The total elastic strain energy stored in the specimen is then:

$$U_E = 2U_{E,i} = \frac{Ewh^3d^2}{16a^3} \tag{3.29}$$

and the mechanical energy release rate is equal to:

$$G = \frac{3Eh^3d^2}{16a^4} \tag{3.30}$$

for the double cantilever beam (DCB) test specimen. An interesting aspect of this test is that G is a decreasing function of a. The system is thus stable regarding crack propagation: the crack advances at the same rate as that of the wedge. So,

the measurement of the propagating crack length allows determining  $G_c$  because during crack propagation,  $G=G_c$ . Moreover, the only unknown in the relationship (3.30) is the crack length, a, the other parameters being geometrical parameters (d, h) or a material parameter (E).

The wedge-opening test or double cantilever beam (DCB) test was first proposed by Maszara [38] for the determination of the toughness of bonded silicon wafers. A typical result coming from [38] is shown in Figure 3.9.



Figure 3.9 : Typical wedge test result coming from [38].

Unfortunately, the geometry shown in Figure (3.9) is quite different from the beam bending theoretical geometry used to derive Eq. (3.30), leading to errors in the obtained cracking resistance values. In order to circumvent the drawbacks of the method proposed by Maszara and to offer other options such as the testing at different crack velocity, a new test geometry was developed to measure the toughness of bonded silicon wafers. This test is presented in the next section.
#### 3.3.2 Description of the measurement set-up

Before showing the testing method, we provide some details about the measurement of the crack length, a, which is the key for an accurate estimation of  $G_c$ . Indeed, the crack length enters the expression of  $G_c$  (see Eq. (3.30)) to the fourth power.

#### Determination of the crack length

As previously mentioned, the only parameter to determine for characterizing interface toughness with the wedge-opening test is the crack length (see Eq. (3.30)). In order to measure the crack length, we make use of the optical properties of silicon. As shown in Figure 3.10, the absorption coefficient of undoped silicon is very low in the infrared (IR) region of the light spectrum allowing IR radiation to easily propagate into silicon wafers.



Figure 3.10 : Absorption coefficient of undoped silicon (from [39]).

It is thus interesting to analyze the behavior of light when it meets various interfaces occurring in wafer bonding [40]. Part of the IR flux is reflected at the different interfaces present in the system. The reflectivity for the normal direction of incident radiation is given in the form:

$$R_0 = \frac{(n_1 - n_2)^2}{(n_1 + n_2)^2} \tag{3.31}$$

where  $n_1$  is the refractive index of the first medium and  $n_2$  is the refractive index of the second medium. On the basis of relationship (3.31), the reflectivity associated to a particular interface can be calculated for:

- air-silicon,
- silicon dioxide-silicon,
- air-silicon dioxide.

Assuming the following values of the refractive index:  $n_{SiO_2} = 1.42$ ,  $n_{Si} = 3.42$  and  $n_{air} = 1$  leads to :  $R_{air-Si} = 0.3$ ,  $R_{Si-SiO_2} = 0.17$  and  $R_{SiO_2-air} = 0.03$ . Due to the weak absorption coefficient of silicon and silicon dioxide toward IR wave, the ratio of the radiation intensity flux measured at the spot of a bonding defect to the radiation intensity flux associated to a bonded interface is only determined by the losses resulting from reflections on the bonding surfaces.

Let us consider the three most probable cases occurring in bonding technology.

1. The infrared radiation of intensity  $I_0$  falls onto the bonded silicon wafers at point A (see Figure 3.11) not containing any defect and is recorded after passing through two wafers of thickness h taking into account the reflections at two interfaces (air/silicon, silicon/air). The reflection at the bonded interface can be neglected. According to the notation of Figure 3.11,

$$I_A = (1 - R_{air-Si})^2 I_0 = 0.49 I_0.$$
(3.32)

For a radiation flux incident on the sample at point B and passing through the non-bonded region, we get

$$I_B = (1 - R_{air-Si})^4 I_0 = 0.24 I_0.$$
(3.33)

The relative transmission is given in the form

$$\frac{I_A}{I_B} = \frac{(1 - R_{air-Si})^2 I_0}{(1 - R_{air-Si})^4 I_0} = \frac{1}{(1 - R_{air-Si})^2} = 2.04.$$
(3.34)



Figure 3.11 : Direct bonding defects, air niche between silicon wafers.

2. The infrared radiation of intensity  $I_0$  enters the bonded silicon wafers at point A (see Figure 3.12). Consider that one of the wafers is covered by a silicon dioxide layer. The radiation is recorded after passing through two wafers of thickness h, the oxide layer and after reflections on the interfaces between four different media. According to the notation of Figure 3.12 and following a procedure similar to case 1, we obtain

$$I_A = (1 - R_{air-Si})^2 (1 - R_{Si-SiO_2})^2 I_0 = 0.34 I_0.$$
(3.35)

For radiation flux incident on the sample at point B and passing through a region containing a defect, we get

$$I_B = (1 - R_{air-Si})^3 (1 - R_{Si-SiO_2})(1 - R_{SiO_2-air})I_0 = 0.28I_0.$$
(3.36)

The relative transmission is given as

$$\frac{I_A}{I_B} = \frac{0.34I_0}{0.28I_0} = 1.21. \tag{3.37}$$

3. The infrared radiation of intensity  $I_0$  enters the bonded silicon wafers at point A (see Figure 3.13). Consider that both wafers are covered by a silicon dioxide layer. The radiation is recorded after passing through two wafers of



Figure 3.12 : Indirect bonding defect, air niche between  $SiO_2 - Si$ .

thickness h, two oxide layers and after reflection on the interfaces between five different media.



Figure 3.13 : Indirect bonding defect, air niche between  $SiO_2$  layers.

According to the notation of Figure 3.13 and following a procedure similar to case 1, we obtain

$$I_A = (1 - R_{air-Si})^2 (1 - R_{Si-SiO_2})^2 I_0 = 0.34 I_0.$$
(3.38)

For radiation flux incident on the sample at point B and passing through defect region, we get

$$I_B = (1 - R_{air-Si})^2 (1 - R_{Si-SiO_2})^2 (1 - R_{SiO_2-air})^2 I_0 = 0.32I_0.$$
(3.39)

The relative transmission is given as

$$\frac{I_A}{I_B} = \frac{0.34I_0}{0.32I_0} = 1.063. \tag{3.40}$$

The presence of defects, and especially of air, modifies the intensity of the radiation detected by the camera, forming a contrast between bonded area and unbonded area. This contrast will be used to determine the boundary between bonded and unbonded area, i.e the crack front. The more difficult case is the  $Si/SiO_2//SiO_2/Si$  system for which the decrease of transmission intensity due to the presence of a defect is relatively limited.

Moreover, the IR transparency of silicon decreases when the level of silicon wafer doping increased. So, in the case of highly doped silicon wafers, it is not possible to determine the crack length.

#### Error due to the use of IR radiation

The minimum height of air trapped in between the two wafers affecting the transmission is equal to a quarter of the wavelength,  $\lambda$ , of the incident radiation [1]. The wavelength of the infrared radiation corresponding to the minimum absorption of silicon is equal to around 1.1  $\mu$ m (see Figure 3.10). So, the minimum detectable height of air trapped is 0.275  $\mu$ m. It results that an error of 4 % on the crack length is made leading to an error approximately equal to 19 % on the cracking resistance (see Eq. 3.30) if a wedge of 100  $\mu$ m is used. If a wedge of 230  $\mu$ m is used these errors become equal to 2.8 % for the crack length and 12 % for the toughness. The details of the calculations can be found in Appendix D.

However, all the values presented in this thesis do not take into account this measurement error in order to compare the toughness values we obtained with values coming from the literature.

#### The loading setup

The only measurable parameter needed for the determination of the toughness of bonded interface is the crack length, a.

The experimental setup used for measuring the interface toughness is schematically represented in Figure 3.14.



Figure 3.14 : Measurement set-up used to determine crack length.

A wedge is mounted on an universal mechanical testing machine (Instron 556, Load cell of 10kN). The tested samples are illumanited using a polychromatic source of light (conventional slides projector) and the transmitted light is recorded by a CCD camera. The signal is then treated and analyzed by an image acquisition software (VISILOG 5 from NOESIS). A typical result obtained with this measurement setup is presented in Figure 3.15. The presence of air decreasing the intensity of radiation received by the camera, the unbonded zone is darker (see Section 3.3.2) and the crack length can easily be measured as indicated in Figure 3.15. The



Figure 3.15 : (a) Lateral view of the wedge test with d is the thickness of the wedge and a is the crack length. (b) Typical result obtained with the wedge test (front view of the wedge test).

originality of this method is that the tests are performed continuously, meaning that the crack opening method is performed at a controlled blade insertion rate. Most authors [1, 38] seem to perform their tests statically: they insert a blade to a certain depth and measure the resulting crack length. The results proposed later will show the differences between those two methods in terms of toughness values. Another particularity of our set-up is the geometry of the samples: tests proposed in the literature [1, 38, 41] are performed on entire wafer which is more difficult to interpret mechanically. To perform efficient tests, it is preferable to work with samples having a width smaller than the width of the inserted blade in order to avoid edges effect. Regarding test procedures, great care should be taken when interpreting static tests with respect to rate effects, spatial heterogeneity in the bond resistance, and misalignment during initial wedge insertion. Performing fracture test under steady-state conditions is also important for properly addressing environmental effects such as moisture effects [31, 42].

#### $"Pixelisation"\ error$

The determination of the exact crack front location on the picture taken by the camera can be sometimes difficult. A small error on the position of the crack front will lead to a large error on the toughness as explained above.

If we consider an error of 1 pixel on the position of the crack front and a crack length measured on the computer ranging from 50 to 80 pixels (this is a typical value obtained with our measurement set-up on many different samples), the maximum error made on the crack length is equal to 2% leading to an error of 8% on the interface toughness. However, this inaccuracy can be circumvented by multiplying the number of measurements of the crack length. The crack length could be underestimated in one measurement and overestimated in the next one leading to a correction by averaging.

#### **3.3.3** Data reduction scheme

The first important result appears when looking at the evolution of the crack length with the wedge displacement in Figure 3.16. This results comes from the testing of annealed samples (150°C during 150 hours) covered with a native oxide. Three zones can be usually distinguished. The first zone, whose size can vary from one measurement to another, corresponds to transient effects related to small initial misalignment of the razor blade and uncontrolled initial velocity of blade insertion. The value of the crack length in this zone can vary from one sample to another (Figure 3.16) even if they undergo the same treatment due to bonding heterogeneity at the scale of the wafer (this is the case for results presented in Figure 3.16). When tests are performed "statically" i.e. without a continuous insertion of the wedge as presented in e.g. [38], crack length values associated to the first zone are often determined and this can lead to an important error on the determination of the interface toughness. The second zone corresponds to the true steady-state regime giving relevant crack length values. So, only crack length values belonging to this plateau can be considered in Eq. (3.30) to determine the toughness of the bonding. In the third zone, the crack tip stress field starts interacting with the specimen end and therefore the value of the crack length is not representative anymore of the intrinsic interface toughness of the bonded wafers. From now, all the interface fracture toughness results presented are calculated using the relationship (3.30)based on the value of the crack length measured in the second zone.



Figure 3.16 : Evolution of the crack length for two samples coming from same wafers during wedge displacement. Crack length value from the  $2^{nd}$  zone must be used to determine the interface toughness. Note : a wedge displacement equal to 0 means that, after inserting the wedge manually and correctly aligning the specimen, a crack length can thus be measured at "wedge displacement=0" because the manually inserted wedge has promoted an initial debonding of the specimen.

#### 3.3.4 Validation of the testing method

#### Influence of the wedge thickness

In order to verify the validity of the methodology, two different wedge thicknesses have been used in order to measure the interface toughness of samples coming from the same bonded wafers. The averaging is made on 4 samples for each wedge thickness and around 10 crack length measurements for each sample. The results are presented in Table 3.1 and provide a first demonstration that the interface toughness of bonded wafers obtained with the steady-state wedge-opening characterization technique is independent of the wedge thickness.

	Wedge thickness = $100 \ \mu m$	Wedge thickness = 230 $\mu m$
$G_c  [\mathrm{J/m^2}]$	$1.6 \pm 0.3$	$1.6 \pm 0.1$

 Table 3.1 : Interface toughness of similar samples determined using the steady-state wedge-opening test with two different wedge thicknesses.

#### Influence of the sample width

The influence of the width of the samples was addressed by M. Legros in the framework of his undergraduate thesis [43]. Samples with three different widths (5, 10, 15 mm) were tested using the wedge-opening test. The results presented in Table 3.2 show that the determination of the interface toughness is also independent of the sample width. This also shows that the so-called anticlastic effect<sup>2</sup> does not affect the results.

	Width = $5 \text{ mm}$	Width = $10 \text{ mm}$	Width = $15 \text{ mm}$
$G_c \; [\mathrm{J/m^2}]$	$1.9 \pm 0.2$	$1.8 \pm 0.2$	$2.1 \pm 0.2$

 Table 3.2 : Interface toughness of similar samples determined using the steady-state wedge-opening test with three different sample widths.

# 3.3.5 Indirect method for the measurement of the crack length

The measurement of the crack length using IR radiations is only possible if the sample is transparent to IR. In some applications and particularly when a ductile (non-IR transparent) layer is inserted between the substrate and a silicon dioxide (as described in Chapter 6), the use of the steady-state wedge-opening test methodology described in Section 3.3 does not allow determining the interface toughness of bonded wafers. For this type of samples, an indirect method based on the measurement of the out-of-plane displacement profile is used.

<sup>&</sup>lt;sup>2</sup>The anticlastic effect is the transverse bending of an elastic beam occurring when bending is imposed in the length of the beam. It can lead to crack tunnelling (curvature of the crack front with crack advancing faster in the middle of the specimen) and perturb the measurement.

#### The profilometer method - Theory

Let us consider a beam subjected to a vertical displacement d/2 as sketched in Figure 3.17.



Figure 3.17 : Bending of a beam subjected to a vertical displacement.

From linear elastic beam theory, there exists a unique relationship between the bending of the beam (x and y coordinates), the vertical displacement, d/2 and the distance between the imposition of the vertical displacement and the fixed end of the beam, noted a. This relationship is :

$$y = f(x) = \frac{d}{4} \left( 2 - 3\frac{x}{a} + \frac{x^3}{a^3} \right)$$
(3.41)

Hence, if a wedge of known thickness is inserted between two bonded wafers, the measurement of the profile of one of the bended wafer can be used to indirectly measure the crack length, a, through Eq. (3.41). Moreover, using Eq. (3.41) allows the determination of the true crack length (no error from direct measurement due to limiting thickness of the air gap) because the silicon wafers remain purely elastic when subjected to bending. However, this method is more time consuming than the "classical" wedge-opening method and cannot, up to now, be performed in a "steady-state" regime.

#### The profilometer method - Practical use

After inserting a wedge of known thickness, the specimen is deposited on the support plate of a DEKTAK 3030 profilometer. A schematic representation of the specimen profile is sketched in Figure 3.18.



Figure 3.18 : Typical profile of specimen when laying on the profilometer support plate.

As shown in Figure 3.18, a change in reference axis must be made before using Eq. (3.41) which requires:

- a rotation of the profile in order to make the linear part in front of the crack tip following the crack tip coincident with X-axis.
- a translation of the points so that the y-coordinate at x=0 must be equal to half the wedge thickness (see Figure 3.17).

#### The profilometer method - Validation

In order to validate the profilometer method, samples subjected to exactly the same surface treatment are tested with the steady-state wedge-opening test and the profilometer test. These samples are covered with a PECVD oxide, CMP-polished by KEMESYS, and then annealed at 120°C during 10 h. Results are presented in Table 3.3.

	Direct method	Profilometer method
$a \ [mm]$	9.3 +/- 0.4	9.4 + -0.22

Table 3.3 : Comparison of crack lengths measured by the direct method (on 4 samples) and the profilometer method (on 7 samples). Silicon wafers are covered with a PECVD silicon oxide, CMP-polished, activated by O<sub>2</sub> plasma (5s) and then annealed at 120°C during 120 h. The thickness of the wedge is 100 μm.

The wedge test and the profilometer method give, considering the error margins, the same crack length. From the reason explained above in this chapter, it is expected that the direct method always underestimates the crack length (by theoretically 4%) in agreement with the result of Table 3.3. The profilometer technique is thus adequate for determining accurately the crack length of samples involving a ductile interlayer (see Chapter 6). Unfortunately, the profilometer method cannot be used with the loading setup described in Section 3.3.2 but requires a manual insertion of the wedge. However, great care must be taken when inserting the wedge in order to avoid errors such as presented in Figure 3.16.

### **3.4** The tensile test

The tensile test is used to determine an overall strength of the interface. A schematic of this test is presented in Figure 3.19



Figure 3.19 : Schematic of the tensile test.

An universal tensile testing machine (ZWICK 1478) is used to pull apart the bonded interface. The overall bond strength  $\langle \sigma_c \rangle$  is obtained by dividing the force recorded at fracture by the area of the sample:

$$\langle \sigma_c \rangle = \frac{F_{max}}{A} \tag{3.42}$$

#### 3.4.1 Details of the tensile test methodology

After surface and bonding treatments, samples are cut from the bonded wafers in a square shape (from different size ranging from 10 to  $100 \ mm^2$ ). After IR inspection (no visible bubbles are allowed), the samples are stuck to the clamp of the tensile testing machine using a high strength glue (Loctite<sup>®</sup>480). After the drying time of the glue, the samples are tested. The cracking path is characterized after fracture. Indeed, if the crack propagates along the wafer/wafer interface as sketched in Figure 3.20(a), (b), the measurement is correctly performed and a overall strength could be extracted from this type of measurement. On the contrary, if the crack propagates along the wafer/wafer in the silicon substrate or in the glue as depicted in Figure 3.20(c), (d), the measurement is rejected.



Figure 3.20 : Typical cracking path occurring during tensile tests.(a) Schematic of a crack propagating along the wafer/wafer interface (crack path in red, glue in blue), (b) upside view of a half of sample with cracking occurring at the interface, (c) schematic of a crack propagating along the wafer/wafer interface then kinking in the silicon substrate and finally propagating at the clamp/wafer interface, (d) upside view of half of the sample showing this type of fracture.

#### 3.4.2 Tensile tests results

Only one campaign of tests was performed and the results of these tests are presented hereafter. The fracture stress of a large set of samples coming from the same wafers as a function of the cross-sectional area of the sample are shown in Figure 3.21.



Figure 3.21 : Overall strength  $\langle \sigma_c \rangle$  of bonded wafers interfaces as a function of the size of the samples. Wafers have been subjected to a plasma treatment (5s) and annealed during 120 hours at 400° C.

The scatter of the results is extremely large and the interface strength cannot be determined using this test. The tensile test does thus not provide a useful and reliable characteristic mechanical quality factor of the strength of wafers bonded interfaces.

#### **3.4.3** Validity of the tensile test

Theoretical values (calculated from interatomic potentials or ab-initio calculations) for the fracture strength,  $\sigma_c$  are expected to be in the range 1-30 GPa for bonds involving silicon and oxygens [44, 45]. There is no reason, for the present system, which does not involve any relaxation mechanisms to expect values lower than those. Hence the measured  $\langle \sigma_c \rangle$  are probably three orders of magnitude smaller than  $\sigma_c$ .

In fact, the use of the tensile test allows only the determination of an overall strength which is strongly related to defects present at the interfaces. By looking at Eq. (3.43) known as the Griffith's relationship,

$$\sigma_c = \sqrt{\frac{E'G_c}{\pi a}} \tag{3.43}$$

where  $\sigma_c$  is the strength of the material or the interface, E' is the Young's modulus,  $G_c$  is the fracture energy and a is the size of a flaw in the solid or at the interface, one can easily quantify the presence of a defect of length a along the interface. By considering minimal and maximal values of the  $\langle \sigma_c \rangle$  of Figure 3.21 (1 MPa and 10 MPa), it is possible to determine the size of the critical flaw,  $a_c$ , using relationship (3.43). The critical flaw size range from 1 mm (an acceptable value considering the size of the samples) to 0.1 m. This latest value is obviously too large considering the size of the samples. However, crack initiation for low mean strength samples probably occurred at the outer edges of the samples. When performing such tensile test, the only parameter measured is the maximum flaw size which has promoted debonding.

One of the other drawbacks of this type of tests is that the glue used to fix the sample to the clamp must have a strength higher than the overall strength of the interface.

Note finally that a plot such as the one of Figure 3.21 is an indicator of the homogeneity of the bonding.

A original methodology used to determine the true interface strength,  $\sigma_c$  by coupling experiment and simulation will be presented in Chapter 6.

Chapter	4
CHAPTER	4

# Optimization of wafer bonding process

The control of the processing of "clean", exempt of bubbles, interfaces with the desired and often, as high as possible, bond toughness is necessary to make wafer bonding a reliable tool for microelectronic industry.

In this chapter, the experimental conditions used to obtain the best bonding quality between silicon wafers are presented. By "best bonding", we mean that the bonding must be uniform, i.e. no interface bubbles should be present at the interface at the end of the process, and that the toughness of the interface must be as high as possible to support external loading and then to avoid delamination.

Note that the problem of bubbles can be independent of the local quality of the interface toughness: very local high interface toughness,  $\Gamma_0$ , is possible between large bubbles whereas the global mean interface,  $\langle \Gamma_0 \rangle$ , will not be good as sketched in Figure 4.1.

The first part of this chapter deals with the formation of the interface bubbles and with the different ways to avoid or eliminate them.

In the second part of this chapter, the different surface treatments aiming at optimizing the fracture resistance of interfaces are presented.

Finally, the influence of an "artificial" (thermal or PECVD) silicon oxide covering the silicon substrate is discussed.



Figure 4.1 : Representation of the local toughness,  $\Gamma_0$ , between large bubbles at the silicon/silicon interface.

# 4.1 Quality of the wafer bonding process

One of the most common problem associated with wafer bonding is the occurrence of unbonded interface areas which are frequently termed "interface bubbles" or "voids". Interface bubbles form either immediately during the room-temperature bonding process, during storage, or during a heat treatment. The cause of the bubbles formed during room temperature bonding includes:

- particles on the bonding surface;
- localized surface protrusions;
- localized absence of sufficient density of bonding species;
- trapped air pockets.

Interface bubbles developed during storage or thermal annealing usually originate from:

• reaction of the interface bonding species with the bonding materials (e.g. water molecules react with silicon to generate hydrogen in bonded hydrophilic Si/Si pairs);

- dissociation of bonded groups on the bonding surfaces (e.g. dissociation of siloxane (Si O Si) bonds into silanol Si OH groups);
- desorption or outgassing from contaminants on or inside the bonding materials.

#### 4.1.1 Room-temperature bubbles

For the first case where particles or contaminants are responsible for the presence of bubbles, it is possible to predict the size of the bubbles as a function of the size of the particles. During wafer bonding at room temperature, wafers are deformed around particles on bonding surfaces, leaving circularly unbonded interface areas. Using the simple theory of small deflections of a thin plate, it is possible to determine the radius, R, of the unbonded area as sketched in Figure 4.2.



Figure 4.2 : Schematic of particle leading to an unbonded area with radius R larger than wafers thickness, h.

The expression for debonding radius R is [1]:

$$R = \sqrt[4]{\left(\frac{4\overline{E}h^3}{3G_c}\right)}\sqrt{d} \tag{4.1}$$

where  $\overline{E} = E/(1 - \nu^2)$  with E and  $\nu$  are the Young's modulus and Poisson ratio of the wafers, h is the thickness of the wafers,  $G_c$  is the toughness of the interface and d is the radius of the particle. When the size of the particle decreases to reach the condition R < 2h, i.e. using Eq. (4.1), that  $d < \sqrt{\left(\frac{12hG_c}{E}\right)} = d_{crit}$ , it has been suggested that an elastomechanical instability occurs leading to an unbonded area with a much smaller radius than predicted by Eq. (4.1) as sketched in Figure 4.3 [46].



Figure 4.3 : Schematic of sufficiently small particle leading to an unbonded area with radius R much smaller than wafers thickness, h.

In that case, the size of the unbonded area is :

$$R \approx kd \text{ with } k \approx 1$$
 (4.2)

It is then extremely important to eliminate large particles, those of a size larger than  $d_{crit}$ . Indeed, at this particle size, the radius of the unbonded area would change from about  $2h \ (\approx 1000 \ \mu m)$  to about  $d_{crit} \ (\approx 0.1 \ \mu m)$  for room-temperature bonding), corresponding to a decrease of  $10^4$  in R and to a decrease by a factor  $10^8$  in the surface of the unbonded area.

#### 4.1.2 Temperature-dependant interface bubbles

In addition to interfaces bubbles in room-temperature bonded pairs which are caused by particles and surfaces irregularities, bubbles frequently appear during annealing at elevated temperature [47]. In the case of hydrophilic bonded wafers, it is assumed that bubbles formation occurs at a temperature over 170°C and disappears at a temperature over 1000°C [1]. In a lot of applications (MEMS, CMOS), it is not possible to impose a post-annealing at a temperature in the range of 1000°C without degrading electrical or mechanical properties.

Figure 4.4 shows the presence of bubbles on the bonded interface after thermal annealing.



Figure 4.4 : Infrared transmission images before (a) and after (b) annealing for Si/Si bonded wafers. Annealing conditions are: 120°C for 70h followed by a subsequent annealing at 260°C for 90h.

The source of these bubbles is the presence of water molecules along the interface. These water molecules can come either from the two or three monolayers present at the bonding interfaces of hydrophilic silicon wafers after room-temperature contacting or from the polymerization of silanol groups. This polymerization reaction leads to the formation of strong covalent siloxane bonds that bridge the two wafers but water is generated as a byproduct of the reaction:

$$Si - OH + Si - OH \longleftrightarrow Si - O - Si + H_2O.$$
 (4.3)

However, it appears that the gas forming bubbles is mainly hydrogen [1]. Indeed, molecular water diffuse inside the silicon oxide (native, thermal or PECVD) covering the silicon wafers and react with the surrounding silicon to form hydrogen according to the following reaction:

$$Si + 2H_2O \longrightarrow SiO_2 + 2H_2.$$
 (4.4)

The thickness of the silicon oxide increases and the produced hydrogen migrate to the interface where it promotes the formation of bubbles. This reaction is possible if water molecules can reach the silicon wafers i.e. if the silicon oxide layer is not too thick. Moreover, native oxide absorb released gases at the bonding interface less effectively than dry thermal oxide films. So, thermally induced bonding voids are more prevalent in hydrophilic bonded wafers with native oxides than thermally oxidized wafers [48].

In order to form interface bubbles, hydrogen molecules have to diffuse into the silicon oxide and along the interface until they find a suitable nucleus (hydrocarbons, particles, ...). Indeed, experiments on the nucleation of interfaces bubbles clearly show that the presence of hydrogen alone is generally not sufficient to nucleate interface bubbles, but that thermally desorbed hydrocarbon molecules appear to play a major role [47]. Methods to prevent bubbles formation at the interface of hydrophilic bonded silicon wafers can therefore be based on:

- removal of interface hydrocarbons;
- removal of interface molecular water;
- removal of either desorbed hydrocarbon and hydrogen molecules by adsorbing them in a adapted material.

Some particular surface treatment (cleaning) can be used to remove thermally unstable hydrocarbons from the bonding interfaces [47, 49, 50]. One attractive surface treatment consists of an  $O_2$  plasma treatment. Indeed, oxygen radicals coming from the  $O_2$  plasma atmosphere can be efficient enough to break any Si - O - R bonds, where R stands for hydrocarbons contaminations [51]. Moreover, as seen in the next section, this type of surface treatment will also increase the toughness of the bonding.

In order to remove desorbed hydrocarbons and water molecules, it is also possible to trap them in cavities avoiding the formation of bubbles. The two pictures shown in Figure 4.5 exhibit the surface of bonded silicon wafers with channels of 200  $\mu$ m width, 1  $\mu$ m deep and with distance of 600  $\mu$ m between them before and after an annealing at 120°C followed by a post-annealing at 260°C for 90 hours. No bubbles appear because the precursors of the bubbles (water, hydrocarbons,...) have moved into these channels. Thermal silicon oxide will also prevent the generation of bubbles at the interface. Thermal oxides, with only 43% of the lattice space occupied [1] involve a very open structure allowing hydrogen, gaseous hydrocarbons, and other impurities to diffuse, resulting in a significant reduction of the gas pressure at the bonding interface. Therefore, a bonding pair consisting of, at least, one thermally oxidized silicon wafer is much less prone to the generation of temperature-dependent bubbles.



Figure 4.5 : Infrared images for Si/Si wafers with trenches before (a) and after(b) annealing. Annealing conditions are: 120° C for 70h followed by a subsequent annealing at 260° C for 90h. These pictures must be compared with pictures of Figure 4.4 corresponding to non-patterned wafers and where bubbles are generated after thermal annealing.

# 4.2 Obtaining the highest interface toughness for bonded silicon wafers

Silicon wafers pairs are thermally treated in order to enhance the interface toughness of the bonding after room-temperfature contacting. It is important to remind that we are dealing with hydrophilic silicon wafers i.e. that the surface of the silicon wafer is covered by a silicon oxide (native oxide, thermally grown oxide or PECVD oxide). For hydrophilic Si/Si pairs, the chemical processes leading to a significant increase of the toughness are:

1. dissociation of Si - O - Si bonds from the silicon oxide layer via attack by interface molecular water:

$$Si - O - Si + H_2O \longleftrightarrow Si - OH + Si - OH$$
 (4.5)

which leads to an increased number of -OH groups;

2. rearrangement of the interface molecular water to form more stable hydrogen

bonded structures and removal of the excess interface molecular water by migration or diffusion (see Figures 2.15, 2.16 and 2.17)

3. polymerization of the hydrogen-bonded silanol groups into strong covalent siloxane bonds between the two wafers (see Figures 2.17 and 2.18) following reaction:

$$Si - OH + Si - OH \longleftrightarrow Si - O - Si + H_2O.$$
 (4.6)

These reactions are expected to be both time and temperature dependent. One should be aware that, at a given temperature, a stable state of the bonding interface is not achieved after a short time annealing. In order to ensure saturated values of bonding energy, sufficiently long annealing time should be used [1].

In order to obtain a higher bonding toughness than with a simple annealing, the wafers can also be subjected to a plasma treatment before bonding and annealing [1, 52]. Plasma surface treatments before bonding have been shown to induce strong bonding energies even if a low temperature annealing is applied to strengthen the direct bonding. The plasma medium consists of electrons, ions, radicals and UV radiations. The plasma treatment can be considered as a combination of chemical reactions (e.g. between surface bonds and radicals formed in the discharge volume) and sputtering effects by ion bombardment. Moreover, plasma treatment effects can include the transformation of surface liquid films (e.g. water for hydrophilic surfaces) or surface contaminants (e.g. hydrocarbons) into volatile species, which are evacuated by the pumping system. Then, the plasma treatment cleans the wafers by eliminating hydrocarbons responsible of bubbles formation. Moreover, oxygen radicals generated from the  $O_2$  plasma atmosphere are also efficient enough to be absorbed, leading to form Si - O - dangling bonds, and finally to generate Si - O - H bonds with adsorbed water. These Si - O - H bonds are then used as the precursors of the strong covalent Si - O - Si bonds connecting the two wafers. Figure 4.6 shows the interface toughness measured for three different surface treatments using the wedge opening method described in Chapter 3.

The first set of samples were subjected to a standard cleaning as described in Section 2.2.1 and were then put in contact just before testing. The second set of samples were cleaned, put in contact and annealed at 150°C for 150 hours. Finally, the third set of samples were subjected to an  $O_2$  plasma treatment before contacting and annealing <sup>1</sup>. The different samples tested are then:

<sup>&</sup>lt;sup>1</sup>These results were reported in Bertholet *et al.* [37]

- non-annealed samples;
- annealed samples at 150°C for 150 hours;
- $O_2$  plasma treatment (10s, 20W, 25 cm<sup>3</sup>/min  $O_2$  plasma, 4Pa ) before annealing at 150°C during 150 hours.



Figure 4.6 : Fracture toughness for various surface treatments. The wedge velocity is fixed to 25 mm/min.

The toughness of bulk silicon is also shown in Figure 4.6 for the sake of comparison. The toughness obtained for the different surface treatments are expected based on the values reported in 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 annealed samples show relatively good bond toughness due to the formation, during the annealing, of Si - O - Si bond between the wafers [1]. Plasma treated wafers provide the highest fracture toughness. The toughness is then so large that some specimens failed by the crack kinking-off in the silicon substrate. The high value of bond toughness obtained for  $O_2$  plasma treated samples is due to the higher density of

the Si - O - Si bridges. 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. However, if the time of the plasma is too high, the surface of the silicon wafers will be damaged and bubbles will occur at the interface as seen in Figure 4.7.



Figure 4.7 : Infrared images of Si/Si bonded wafers (a), (c) before and (b), (d) after annealing at 400° C for 120 h and for O<sub>2</sub> plasma pretreatment exposure time of (a), (b) 20 s and (c), (d) 3 s prior to bonding (from [53]).

More time has been spent to analyze in details the effect of the  $O_2$  plasma treatment.

## 4.2.1 Optimization of the $O_2$ plasma treatment

This study was initiated by Dr. Zhang during his postdoctoral visit at UCL and continued by B. Olbrechts (EMIC-UCL) in close collaboration with myself.

As previously explained, excessive plasma exposure raises the density of annealing voids. The increase of the roughness of the wafers can explain partly this phenomenon. Moreover extra bonding reaction products cannot be released from the bonding interface [54], promoting the formation of bubbles during annealing.

The annealing voids density in  $Si/SiO_2$  or  $SiO_2/SiO_2$  wafer bonding can be reduced due to the absorption of byproducts by an  $SiO_2$  layer. However, many annealing voids were still found at the  $SiO_2/SiO_2$  bonding interface when a plasma exposure time of 20 seconds was performed as surface pretreatment. When the thickness of the  $SiO_2$  layer in a sandwich structure is lower than a critical value [52, 54], the reaction byproducts cannot be absorbed completely in the dioxide layer leading to the formation of annealing voids. Therefore, as in Si/Si wafer bonding,  $O_2$  plasma exposure time has an important impact on  $SiO_2/SiO_2$  wafer bonding quality.

In order to prevent annealing voids and meanwhile to increase the bonding surface energy, an optimal  $O_2$  plasma exposure time has to be determined for Si/Siand  $SiO_2/SiO_2$  bonding interfaces. Toughness of the bonding as a function of the plasma pretreatment duration is shown in Figure 4.8 for Si/Si and  $SiO_2/SiO_2$ bonding wafers annealed at 400°C for 120 hours.

Looking at Figure 4.8, we clearly see that the toughness first increases, reaches a maximum and then decreases with the plasma exposure time. For shorter plasma exposure time, the relatively low value of the interface toughness can be explained by incomplete cleaning process. The presence of remaining hydrocarbons contaminants can then be responsible for this low value. After reaching a maximum, the interface toughness of the bonding decreases whereas the scatter increases. These variations are caused mainly by the presence of annealing voids which affect all along the wedge test the measurement of the crack length. An optimal value for the bonding energy associated to an uniform distribution of the bonding energy is reached for 3 seconds  $O_2$  plasma pretreatment prior to bonding for Si/Si bonding and 5 seconds for  $SiO_2/SiO_2$  bonding. Moreover, the bonding energy variations for  $SiO_2/SiO_2$  bonded wafers are always smaller than for Si/Si bonded pairs due to byproducts absorption by dioxide layers at the interface.



Figure 4.8 : Interface toughness as a function of plasma exposure time for Si/Si and  $SiO_2(100nm)/SiO_2(100nm)$  wafer bonding (from [54]).

# 4.3 Influence of the oxide layer type

This study was performed by B. Olbrechts (EMIC-UCL) in close collaboration with myself and are presented in [55].

The presence of a silicon oxide layer can, in principle, lead to better bonding quality by absorbing reaction byproducts during the bonding process.

Three main types of oxide layers can be used to bond two hydrophilic silicon wafers:

- native oxide present on top of silicon wafers or formed during wet chemical cleaning;
- dry or wet thermally grown silicon oxide;
- PECVD oxide.

Native oxide formed by wet chemical cleaning has a high chemical reactivity (fast formation of Si - OH groups on top of the silicon wafers surface) but the thickness of this type of oxide is too small to absorb all the byproducts of the bonding reaction and bubbles can occur during the annealing step.

It is not possible to use as-it PECVD oxide for bonding because of its high roughness. Room-temperature pre-bonding is less effective than with the other types of oxide leading to a poor bond toughness after the annealing step. Using a Chemico-Mechanical Polishing (CMP) treatment reduces the roughness of PECVD oxide and making the bonding possible. The toughness of  $SiO_2(PECVD)/SiO_2(PECVD)$  interface have been measured for samples subjected to CMP and are presented in Chapter 6.

In order to obtain thick oxide layers, dry or wet thermal oxides are grown on the top of silicon wafers. A study of the influence of the oxide thickness coupled to the plasma exposure time on the interface toughness of the bonding has been conducted in order to optimize this process.

#### 4.3.1 Silicon oxide thickness and plasma exposure time

The evolution of the bond toughness with plasma exposure time for two silicon oxide thicknesses is shown in Figure 4.9.



Figure 4.9 : Variation of the interface toughness of the bonding as a function of  $O_2$  plasma exposure time for two different silicon oxide thicknesses.(a)  $SiO_2(100nm)/SiO_2(100nm)$ , annealed at  $400^\circ$ C during 120 hours, (b)  $SiO_2(400nm)/SiO_2(400nm)$ , annealed at  $400^\circ$ C during 70 hours (from [53, 55]).

Even if the annealing time is different between the two results, a shift of the maximum of the curve is clearly seen, moving from 5 seconds for silicon oxide thickness of 100 nm to about 60 seconds for thickness of 400 nm. The increase of the plasma exposure time required for reaching the maximum toughness is principally due to the absorption of oxygen radicals (created during the plasma treatment and responsible for the formation of reactive Si - OH groups) by the silicon oxide layer reducing the plasma surface activation efficiency [55]. Thicker silicon oxide layer can absorb more oxygen radicals and the plasma exposure time needed to achieve high interface toughness should thus increased.

In order to observe the influence of the total silicon oxide thickness (i.e. the sum of the two silicon oxide thicknesses, one per wafer), two set of samples were made. In the first set, each wafer is covered with silicon oxide  $(Si/SiO_2//SiO_2/Si)$  whereas in the second set, only one silicon wafer is covered by a silicon oxide layer  $(Si//SiO_2/Si)$  such that the total silicon oxide thickness is the same for both sets as sketched in Figure 4.10.



Figure 4.10 : Schematic illustration of the sandwich-like structures: (a)  $Si/SiO_2$  or (b)  $SiO_2/SiO_2$  wafer bonding.



The results are presented in Figure 4.11

Figure 4.11 : Variation of bonding energy as a function of the interfacial oxide thickness for Si/SiO<sub>2</sub> and SiO<sub>2</sub>/SiO<sub>2</sub> wafer bonding with O<sub>2</sub> plasma surface activation (from [54]).

When plotting the variation of the bond toughness as a function of the interfacial oxide thickness (see Figure 4.10), the two bonding configurations  $(Si/SiO_2 \text{ and } SiO_2/SiO_2)$  show very similar trends. Following these results, the vertical position of the bonded interface in the sandwich-like structure does not seem to play a role on the resulting bonding energy.

## 4.4 Conclusions

The wafer bonding process requires to have an interface with a uniform bond quality (no voids allowed) and with the desired interface fracture toughness, often as high as possible.

The uniformity of the bonding depends on the different ways the byproducts of the polymerization reaction are evacuated. These byproducts, mainly water or hydrogen molecules, can be trapped in the silicon oxide or in cavities made at the interface or can diffused to the outer boundary of the samples. The thickness of the silicon oxide on the top of the silicon wafers must be thick enough to absorb all the byproducts. It is thus preferable to use an "artificial" (thermal or PECVD) silicon oxide whose thickness can be controlled by the deposition process. However, the roughness of these silicon oxide must be as low as possible in order to promote a good pre-bonding step. Hence, polishing techniques (CMP, ...) must be used to decrease the roughness of the silicon oxide layer if necessary.

After pre-bonding, an annealing step is necessary to increase the interface toughness. This annealing favors the formation of strong covalent bonds (Si - O - Si)across the two silicon wafers. Temperature and time of the annealing step have to be optimized to reach the desired toughness and bonding uniformity. However, it is important to note that the annealing conditions must be compatible with the surrounding materials and is often limited to 450°C in CMOS and MEMS technologies.

Finally, plasma treatment of the silicon wafers before contacting is used to obtain the higher interface fracture toughness. This treatment increases the reactivity and the quantity of the -Si - OH bonds responsible of the pre-bonding between the two wafers. The different parameters controlling the plasma treatment (power,  $O_2$  flux, ...) and the exposition time must be optimized in order to reach the higher interface toughness coupled with a perfect bonding uniformity. Moreover, the optimized exposition time seems to be dependent of the silicon oxide thickness, indicating that an optimization procedure must be performed for each type of assembly.
Chapter	5
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# Environmental and geometry effects

Most of the microsystems are working under environmental conditions which can affect their reliability. Particularly, the presence of water can influence the interface between bonded wafers and affect the bond toughness. This effect is an important issue for microsystems intended to work in relatively wet atmosphere or inside/or with the presence of water (e.g. microfluidic devices).

In addition to these environmental effects, geometrical effects can also influence the bonding of silicon wafers. These geometrical effects are mainly due to the design of the microsystems consisting in the existence of bonded and unbonded areas of various sizes. Moreover, geometrical irregularities of the silicon wafers (flatness, roughness) can affect the bonding process occurring between two silicon wafers. These geometrical effects are treated in the same chapter as the environmental effects because they might be sometimes related.

### 5.1 Influence of environment on the fracture process

### 5.1.1 Theoretical background Chemical process in crack propagation

This section is inspired by Chapter 5 of Ref. [31] which deals with subcritical chemically affected crack propagation in brittle solids.

Most fracture processes take place in a chemically interactive environment. The

effects of the environment on crack propagation can be strongly detrimental. One of the most distinctive manifestation is a rate-dependent growth, even at a sustained applied stress well below the "theoretical" strength. The first reported evidence of "slow" crack growth was made on mica by the Russian school, following Obreimoff's experiment. In the late 1960's, a series of studies on silicate glasses in aqueous environments was initiated by glass scientists, notably Wiederhorn [56, 57]. Wiederhorn's studies were to lay the groundwork for subsequent adoption of fracture mechanics technique in the construction of v - G curves (with v is the crack velocity and G is the mechanical energy release rate). Most of the results determined on different types of ceramics (typically glass, sapphire, mica, ...) fall into a typical v - G curve presented in Figure 5.1.



**Figure 5.1 :** Schematic representation of the typical v - G curve (from [31]).

This typical curve can be divided into four regions implying transitions in the

rate-controlling process as sketched in Figure 5.1.

- Region 0 is the threshold region and is difficult to establish from forward velocity measurements alone, because of increasingly excessive time intervals needed to measure velocities as the crack approaches equilibrium.
- Region I crack-growth behavior is characterized by a dependence of crack velocity on mechanical energy-release rate, G, and on the partial pressure of water in the environment [56]. If one of these two quantities is increased, the crack velocity is observed to increase. In region I, the crack velocity is reaction rate limited, and, therefore, is controlled by the partial pressure of water in the environment at any level of applied stress. The extreme sensitivity of the velocity to applied load (see Figure 5.1) suggests exponential or power law velocity functions with large coefficients or exponents in G. Therefore, an expression to the velocity in region I can be either

$$v_I = v_0 exp(\frac{\alpha G}{kT}) \tag{5.1}$$

where  $v_I$  is the velocity in region I,  $v_0$  is a parameter depending on the partial pressure of water, the adsorption-desorption activation energy, the lattice atoms spacing, the fundamental lattice frequency and the temperature,  $\alpha$  is an activation area, G is the mechanical energy release rate, k is the Boltzmann's constant and T is the temperature or

$$v_I = K_I G^m \tag{5.2}$$

where the constant K and the crack-growth exponent m depend on the material and the environmental conditions.

• Region II crack-growth behavior is also characterized by a dependence of crack velocity on the amount of water in the environment. In contrast to region I behavior, however, the crack velocity in region II does not depend strongly on the applied load. Theoretical interpretations of region II crack-growth behavior lead to the conclusion that the crack velocity is controlled by the rate of transport of water from the environment to the crack tip [56]. The velocity in region II can be written as:

$$v_{II} = K_{II}f(G) \tag{5.3}$$

where  $K_{II}$  is a constant that depends on the partial pressure of water, the lattice atoms spacing, the temperature, the Boltzmann's constant and the molecular mass. The function f(G) is equal to CG where C is a very low constant. Thus, the velocity in region II varies relatively slowly with G.

• Region III crack-growth behavior is characterized by a strong dependence of crack-growth rate on the applied load, and by a complete absence of any dependence of crack-growth rate on water in the environment. Region III crack growth is controlled by fracture mechanisms that do not depend on environment.

### 5.1.2 Application to wafer bonding

In wafer bonding, where the link between the two silicon wafers is mainly due to strong Si - O - Si covalent bonds (similar to the ones in glass), similar effects can be observed. By varying the cracking velocity, v (which is equal to the wedge velocity in the steady-state wedge opening test), it is possible to observe the dependence of  $G_c$  on v.

Figure 5.2 shows the variation of the fracture toughness as a function of crack velocity for annealed samples (no specific surface treatment except cleaning followed by a 150 h annealing at 150  $^{\circ}$ C) that we measured using the steady-state wedgeopening test. A significant increase of the bond fracture toughness with increasing



Figure 5.2 : Variation of the crack velocity as a function of the interface toughness. The measurements were performed on samples prepared by the same procedure (no specific surface treatment - 150 h annealing at 150° C).

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 [31, 42] (see Figure 5.1). Models have been developed in order to describe this dependence of  $G_c$  on v and they have been found to accurately predict the relationship between

moisture content and crack velocity for each region [31, 42, 58]. The basis for these models is the reaction of a water molecule with the strained Si - O - Si bonds at the crack tip. This reaction can be divided in three stages as depicted in Figure 5.3.



Figure 5.3 : Three-stages interaction between a water molecule and strained crack-tip siloxane bond in glass: (a) adsorption, (b) reaction and (c) separation (from [59]).

- (a) Adsorption. At small strains the water molecule attaches itself physically to the bridging Si O Si bond. The electron orbitals in the water molecule are tetrahedrally coordinated ( $sp^3$  hybrids) about the oxygen, two forming bonds with the hydrogen atoms (excess positive charge) and two forming lone pairs (excess negative charge). Since the bond has also some polar character (silicon excess positive charge), the water molecule aligns itself as shown in Figure 5.3.
- (b) Reaction. At higher applied load, such that the bond is stretched, the water molecule donates an electron to one of the silicon and a proton to the linking oxygen. Two new Si OH bonds are thereby formed.
- (c) Separation. After electron redistribution, the polar terminal bonds mutually repel, thereby completing the bond fracture process.

Knowing how water molecules dissociate the covalent bond linking the two wafers, it is possible to explain the three different regions of Figure 5.2 from a molecular standpoint.

Region III is found to be independent of environmental factors and dominated by critical fracture events. At high crack velocities, water molecules do not have the possibility to reach the crack tip, adsorb and react with the covalent bond. The crack propagation behavior is then the same as if the test was performed in a vacuum chamber.

In region II, the crack velocity is found to depend strongly on the environment and only slightly on the crack driving energy. Crack growth rates are such that moisture must diffuse to the crack tip before the  $SiO_2/H_2O$  complex can form. Once the complex forms, crack extensions occurs. In this region, the crack velocity is linearly dependent on the concentration of water vapor allowed by the diffusional process.

In region I, when the crack velocity drops,  $H_2O$  diffusion is no longer the rate limiting step for crack advance. Rather, the crack velocity is now controlled by bond rupture  $(Si - O - Si \rightarrow 2SiOH)$  and healing events  $(2SiOH \rightarrow Si - O - Si)$ .

A threshold region (region 0 in Figure 5.1) is sometimes observed in very low crack driving energies. In that case, the opening behind the debond may become too narrow for a  $H_2O$  molecule to reach the crack tip. This leads to a so-called "steric hindrance threshold" [60].

The different models used to explain the v - G curves (firstly observed in ceramics and particularly in glass) and the detailed expressions for the crack velocity in the three regions can be found in [31, 56, 58, 61]. Because either loading and environmental species influence crack growth rate in region I, let us focus on crack growth in this region.

### **Region I crack propagation**

Another manifestation of the influence of water molecules on crack propagation at the interface of bonded wafers can be observed by performing static wedge-opening test, i.e by inserting the wedge up to a given location, by leaving it fixed and by recording the evolution of the crack length with time. The evolution of the crack length with time for an annealed samples (150 hours at  $150^{\circ}$ C) is shown in Figure 5.4.



Figure 5.4 : Increase of the crack length after stopping the wedge.

Extensive debonding of the Si/Si interface is typically found to extend by more than 3 mm after stopping the wedge in 50% R.H. (relative humidity) laboratory air at 22°C. Knowing the evolution of the debond length with time, it is possible to determine the corresponding debond-growth rates as sketched in Figure 5.5. The evolution of the crack velocity in region I is characterized by an exponential or a power-law dependence of the applied load. Fitting the experimental data of Figure 5.5 with a power or an exponential function gives:

• for data recorded after stopping the wedge (static test)

$$\frac{da}{dt} = 3.82 \ 10^{-5} \ G^{16.89} \ ; \ R^2 = 0.9875 \tag{5.4}$$

$$\frac{da}{dt} = 2.44 \ 10^{-14} \ e^{21.66G} \ ; \ R^2 = 0.9875 \tag{5.5}$$



Figure 5.5 : Debond growth rate, da/dt, as function of the debond driving energy, G<sub>c</sub>, in laboratory air (R.H. 50%) at 22° C. The points of Figure 5.2 belonging to region I are also plotted.

• for data recorded during testing and corresponding to region I of Figure 5.2 (steady-state test)

$$\frac{da}{dt} = 3.500 \ 10^{-7} \ G^{12.88} \ ; \ R^2 = 0.9754 \tag{5.6}$$

$$\frac{da}{dt} = 1.086 \ 10^{-11} \ e^{10.53G} \ ; \ R^2 = 0.986 \tag{5.7}$$

The difference between the two sets of data of Figure 5.5 comes from the slight difference in toughness that can appear between two different samples, even if they were submitted to the same surface treatment. However, the values of the different coefficients of the fitting curves are in the same order, justifying that the rate dependent crack propagation mechanism is the same for these two sets of data. Moreover, the large coefficients or exponents in G indicate the extreme sensitivity of the velocity to the applied load as suggested in Refs. [31, 56].

An important consideration for the present subcritical debonding phenomenon is the existence of a debond-growth rate threshold value,  $G_{TH}$ , below which debond growth is not observed. Such thresholds are typically observed during subcritical crack-growth processes in bulk glasses at growth rates typically below  $10^{-10}$ m/s. The threshold is thought to arise from adsorbate and corrosion products formation along the crack surfaces which block access of the environmental species to the crack tip.

Crack growth rates in region II and III cannot be fitted by typical relationships because of the lack of experimental data. More information on the chemistry occurring at the crack tip can be found in [31, 56, 57].

# 5.2 Influence of geometry effects on the bond toughness

### 5.2.1 Influence of the flatness and the roughness of wafers on the overall bond toughness

Surface flatness is a global, macroscopic measure (at a scale larger than the wafer thickness) of the deviation of the front surface of a wafer from a specified reference plane. During silicon wafer bonding at room temperature, each wafer of the pair is elastically deformed to achieve conformity of the surfaces. During this stage, the work of adhesion,  $W^1$ , must be sufficient to cause the wafers to deform elastically in order for their surfaces to be in contact. This criterion can be rewritten:

$$W \ge \frac{dU_E}{dA} \tag{5.8}$$

where W is the work of adhesion,  $U_E$  is the elastic energy accumulated in the wafers as they conform to one another, A is the area of interface created. The term  $dU_E/dA$  is a function of the wafer geometry and material properties and will be referred to as the strain energy accumulation rate.

The contacting of two silicon wafers having opposite bowing as sketched in Figure 5.6 supports the building of high elastic strain energy in the wafers.

<sup>&</sup>lt;sup>1</sup>The work of adhesion is equal to twice the surface energy,  $\gamma$ , and is then almost equal to the interface toughness.



Figure 5.6 : Schematic of the bonding process. Wafers are contacted at a point from which surface or electrostatic forces pull the wafers into contact.

For the particular case of Figure 5.6, the strain energy accumulation rate can be determined (see Ref. [62]):

$$\frac{dU_E}{dA} = \frac{1}{24} E h_1^3 \left(\frac{1+\nu}{1-\nu}\right) \left[\frac{(\kappa_2 - \kappa_1)^2}{1+\eta^3}\right]$$
(5.9)

where E is the Young's modulus of the silicon wafers,  $h_1$  is the thickness of the upper wafer,  $\nu$  is the Poisson's ratio of silicon,  $\kappa_1$  and  $\kappa_2$  are the curvatures of the silicon wafers and  $\eta = h_1/h_2$ . Note that the curvature,  $\kappa$  is the inverse of the curvature radius, R, and that  $\kappa_1 = -\kappa_2$  in the case of the configuration shown in Figure 5.6.

The typical curvature of silicon wafers being on the order of 0.01-0.1 m, the strain energy accumulation rate,  $dU_E/dA$ , ranges from 0.15 to 15 mJ/m<sup>2</sup>. Bonding will thus occurs at room temperature if the surface energy is greater than 15 mJ/m<sup>2</sup>. This is the case in hydrophilic bonding where an expected value for  $\gamma$  is 80-100 mJ/m<sup>2</sup>.

When a crack propagates at the interface, the stored elastic energy (equal to the strain energy accumulation rate i.e equal to 15 mJ/m<sup>2</sup>) will facilitate the crack propagation and contribute to decrease the overall interface toughness. This negative contribution due to flatness variation of the wafers, called  $G_{curvature}$  in Eq. (3.20), can be easily determined. It is equal to the energy stored in the silicon wafers i.e:

$$G_{curvature} = 15 \text{ mJ/m}^2. \tag{5.10}$$

This value is largely lower than the value of interface toughness obtained after appropriate surface and thermal treatments where a interface toughness larger than  $2~{\rm J/m^2}$  is expected. Therefore, the bending of wafers does not usually affect the interface toughness of the final assembly.

Microroughness is a local, microscopic parameter of wafer surface quality that is crucial for wafer bonding. Microroughness is usually characterized by the Root Mean Square (RMS) value, abbreviated  $R_q$ , which can be measured by Atomic Force Microscopy (AFM). A value of  $R_q$  less than 5 Å is adequate for silicon wafer bonding at room temperature via hydrogen bonds [63]. This average roughness value is typical for commercially available prime grade bulk silicon wafers.

# 5.2.2 Influence of long unbonded area on the quality of wafer bonding

Most of the real microsystems including wafer bonding as a step of their fabrication process present complex architecture. The bonding of patterned silicon wafers is often necessary to achieve the complex structure of real MEMS as depicted in Figure 5.7.



Figure 5.7 : Cross section of a microperistaltic pump (from [64]).

Silicon wafers with channels were thus processed in order to study the influence of patterns and unbonded areas on the bonding behavior. The description of the fabrication steps required to make these channels is presented in Section 2.2.4. As previously seen (see Section 4.1.2), bubbles can form during the annealing treatment of the wafer bonding process. Such samples cannot then be used for any applications and are often thrown away or recycled. One of the common idea to avoid formation of annealed bubbles is to bond wafers (at least one) with controlled cavities which should collect the byproducts of the chemical reactions occurring at the interface. In Figure 5.8, wafers without (Figure 5.8 (a), (b)) and with (Figure 5.8 (c), (d)) channels have been bonded and submitted to the same annealing treatment.



Figure 5.8 : Infrared transmission images for O<sub>2</sub> plasma treated Si/Si wafers. Annealing conditions are: 120° C for 70 hours followed by a subsequent annealing at 260° C for 90h in air. a) before annealing, without channels. b) after annealing, without channels. c) before annealing, with channels. d) after annealing, with channels.

It appears clearly that annealing bubbles are not present in samples with channels. Indeed, byproducts of the polymerization reaction (see chemical reactions (4.3) and (4.4)  $(H_2O \text{ and/or } H_2)$  can reach the cavities during the annealing stage and escape from the interface. Thus, the byproducts cannot promote the formation of bubbles during the annealing step.

# 5.2.3 Influence of long channels on the toughness of the bonding

Samples with channels were tested using the steady-state wedge-opening test developed during this thesis and presented in Section 3.3.2. The orientation of the channels is depicted in Figure 5.9.



Figure 5.9 : Orientation of the channels of the specimens.

In all of the tested specimens, the wedge move down along the [010] direction, parallel to the channels.

A cross-section of specimens with channels is presented in Figure 5.10.



Figure 5.10 : Cross-section of the samples with channels:  $w_b$  is the width of the bonded areas contacting the two wafers and  $w_c$  is the width of the channels.

Because of the presence of the channels, one needs to correct the relationship

$$G_c = \frac{3Ed^2h^3}{16a^4} \tag{5.11}$$

The interface toughness<sup>2</sup> must be scaled proportionally to the ratio of the total area divided by the bonded area [41]:

$$G_c = \frac{3Ed^2h^3}{16a^4} \left(\frac{A_{tot}}{A_{bonded}}\right) \tag{5.12}$$

which leads for the channel geometry of Figure 5.10 to

$$G_c = \frac{3Ed^2h^3}{16a^4} \left(\frac{w_b + w_c}{w_b}\right).$$
 (5.13)

Samples with channels ( $w_b=400\mu m$ ,  $w_c=200\mu m$ ) have been compared to samples containing no patterns. The two sets of samples (with and without channels) were subjected to the same surface activation (a 5 sec.  $O_2$  plasma activation) and annealing step (10 h at 120°C). The evolution of the toughness as a function of the cracking rate is shown in Figure 5.11 for the two types of samples.

<sup>&</sup>lt;sup>2</sup>From a theoretical point of view, the interface toughness does not depend on the geometry of the specimens. This the strain energy release rate that have to be scaled proportionally to the ratio  $A_{tot}/A_{bonded}$ . However, in the tests developed in this thesis where crack propagates continuously, G is always equal to  $G_c$ .



Figure 5.11 : Interface fracture toughness,  $G_c$ , as a function of the cracking velocity for samples without and with channels ( $w_b=400\mu m-w_c=200\mu m$ ). Surface activation (5 sec.  $O_2$  plasma) and annealing step (10 h at 120° C).

The toughness of patterned samples is always lower than the toughness of samples made of two plain wafers whatever the cracking velocity. Different reasons developed in the next sections can be proposed in order to explain this effect.

### Influence of channels on the action of water molecules during crack propagation

As previously seen in Chapter 5, the effect of water molecules can lower the interface toughness by dissociating the strong covalent bond bridging the two wafers. Hence, when one of the wafers is patterned with channels, the access of water molecules to the crack front is increased as sketched in Figure 5.12. The easier access of water molecules to the crack front could explain the low toughness of bonded patterned wafers interface. Thus, samples tested at high crack velocity (10 mm/min or higher) should not present any difference between samples with and without



Figure 5.12 : Water molecules access to crack front for samples with and without channels. Note: in the case of samples with channels, only one channel was drawn (patterned).

channels. As seen in Figure 5.11, the toughness at high crack velocity is different for samples with and without channels and then, the easiest access to the crack front for water molecules during crack propagation cannot explain the lower toughness for patterned samples. If it is not during crack propagation, this easier access of water molecules to the crack front can promote the decrease of the interface toughness during the storage between samples fabrication and samples testing. Indeed, water molecules trapped and circulating in the channels can promote the dissociation of the strong covalent bonds and then decrease the toughness of the interface. This assumption does not fit with many results of the literature [1] which demonstrate that the toughness increases and reaches a constant value with the storage time. The easiest access to the crack front for water molecules cannot thus explain the lower toughness for patterned samples.

#### Influence of patterns on the contact wave propagation

When two silicon wafers are put in contact, a contact wave propagates from the point of contact (ideally the center of the wafer) to the boundary of the wafer as sketched in Figure 5.13. The presence of the channels could disturb the propagation of the contact wave resulting in a lower quality pre-bonding when compared to values involving no channels. In order to validate this assumption, samples with



Figure 5.13 : Infrared picture of initiation and propagation of a bonding wave in wafer bonding of a 4-in. silicon wafers. (a) Onset of the contact wave, (b) extension after about 2 sec, and (c) complete bonding after about 5 sec. The radial dark line comes from the tong used to initiate the bonding (from [1]).

a particular arrangement of channels shown in Figure 5.14(a) were processed and tested with the steady-state wedge test [43]. As shown in Figure 5.14(b), the



Figure 5.14 : (a) Specific channel arrangement used to evaluate the influence of channels on the propagation of contact wave and (b) toughness of the different samples (1, 2 and 3) (3s plasma O<sub>2</sub>, 400° C-12h, 10mm/min) (from [43]).

toughness of samples located in regions 1 and 3 is similar to the toughness measured for sample 2 (where bonding is initiated). Hence, the presence of the channels does not seem to perturb the propagation of the contact wave, the interface toughness being the same in regions on both sides of the channels.

### Influence of the contact width, $w_b$

In order to understand the effect of channels, Zhang [53] modified the width of the bonded area,  $w_b$  while keeping the width of channels,  $w_c$  constant. The result is shown in Figure 5.15. On Figure 5.15, a second x-axis taken into account



Figure 5.15 : Evolution of the toughness with the contact width,  $w_b$  (from [53]). The interface fracture toughness has been properly corrected following Eq. (5.12).

for the conversion of the width of channels and the contact width into the ratio  $A_{total}/A_{bonded}$  is also represented. The conversion was performed using the simple relationship

$$\frac{A_{total}}{A_{bonded}} = \frac{w_b + w_c}{w_b}.$$
(5.14)

Samples with lower contact area definitely present lower interface toughness. One last possible explanation is developed in the next section dealing with the mechanical influence of channels.

### Influence of channels - Mechanical point of view [65]

The relationship (5.8) establishes the criterion for the pre-bonding step of bowed silicon wafers. It is based on the competition between the adhesion work, W, and the strain energy accumulation rate, $dU_E/dA$ . The presence of channels will reduce the bonding area, dA. As the stiffness of the wafers is not significantly affected by the presence of channels (the height of the channels is equal to 1  $\mu$ m whereas the thickness of the wafers is about 380  $\mu$ m), the strain energy  $dU_E$  that is required to deform the wafers does not change significantly. So, the presence of the wafers increases the value of the ratio  $dU_E/dA$  leading to a less effective pre-bonding step. This pre-bonding step playing a major role in the complete bonding process, this less effective pre-bonding will lead to a lower final interface fracture toughness. A "pre-bonding quality index" can be defined as:

$$Q = W - \frac{dU_E}{dA} \tag{5.15}$$

and we can assume that the lower is the pre-bonding quality index, the lower will be the final interface fracture toughness. In Figure 5.15, the samples having the lower interface toughness have also the lower bonded area (and thus the lower "prebonding quality index").

### 5.3 Conclusions

Environmental and geometrical parameters can modify the behavior of the interface subjected to external loading.

The effect of water molecules on the crack propagation along the interface must be taken into account for technology development. Microsytems working in high relative humidity environment or within water (e.g. microfluidic devices) will be more likely to crack corrosion.

Patterns and cavities decrease the presence of bubbles and then lead to uniform

bonding. However, the presence of these patterns decreases the toughness of the bonding. Some tentative explanations have been given to explain this effect but it is not yet fully understood. In order to fully explain the effect of channels on the toughness of the interface, some other tests should be undertaken: modifying the width of channels while keeping the contact width constant, modifying either the width of channels and the contact width keeping the ratio  $A_{total}/A_{bonded}$  constant should explain more precisely the role of channels. Moreover, the effect of channels may depend on the toughness of the interface and be increased/decreased for very high bond toughness. These tips should be studied in future works in order to explain the decrease of the interface toughness obtained with patterned wafers.

# Influence of a ductile interlayer on the toughness

In microelectronics, ductile materials are used as interconnects between the different levels of microsystems (Al, Cu, AlCu, ...), as metal contacts (W, PtSi,  $TiSi_2$ , ...) or as solder connections (PbSn alloys,...). These ductile layers also affect the distribution of stresses into the systems when they are subjected to external or internal (residual stresses of various origins) loading.

In this work, we were interested at looking at the effect of the presence of a ductile layer close to the interface on the toughness of bonded wafers. This ductile layer could be envisioned as a dissipative layer to protect the interface against debonding.

The first part of this chapter is dedicated to a theoretical modeling of the influence a ductile interlayer on the global toughness of the assembly. A numerical study was undertaken in order to analyze the influence of various parameters (mechanical and geometrical properties of the ductile interlayer, strength of the interface) on the global toughness. The parameters window in which first order effects are expected was determined. Real samples with and without a ductile interlayer were then processed and tested to verify the prediction of the model and demonstrate the practical interest of the idea.

This ductile interlayer can also serve as a "strength sensor" allowing the determination of the strength of the interface. The procedure used to determine the strength of interfaces is presented in the second section of this chapter.

# 6.1 Influence of the ductile layer on the toughness Numerical study

## 6.1.1 Principle of the shielding dissipation by the interlayer

The geometry used is depicted in Figure 6.1.



Figure 6.1 : Samples with ductile interlayers.

After depositing a ductile layer of controlled thickness  $(h_p)$  onto the silicon substrate of thickness,  $h_s$ , a PECVD silicon dioxide of thickness,  $h_{el}$ , is deposited onto the ductile interlayer surface. This silicon oxide is used to perform the bonding between the two wafers. Details about the fabrication of these samples can be found in Chapter 2. The influence of these ductile interlayers on the global toughness is represented in Figure 6.2.



Figure 6.2 : Influence of the size of the stress field generated by the crack propagation. (a) The size of the region involving intense stress field is too small and does not generate permanent deformation in the ductile interlayers. No effect on the overall interface toughness is then expected. (b) The size of the region involving intense stress field is large enough to promote plastic dissipation into the ductile interlayer. The toughness of the assembly will be improved.

When a crack propagates, a stress field is generated ahead of the crack tip. Depending of the size of the region involving intense stress field, it will promote or not a plastic permanent deformation into the ductile interlayers lying on both sides of the crack plane. This plastic dissipation will thus increases the global interface toughness of the assembly with ductile interlayers. In such samples, the global interface toughness writes:

$$\Gamma = \Gamma_0 + \Gamma_p \tag{6.1}$$

where  $\Gamma$  is the global interface toughness of the assembly,  $\Gamma_0$  is the intrinsic toughness of the interface and  $\Gamma_p$  represents the increase of toughness due to plastic dissipation into the ductile interlayers. A multiscale numerical modeling strategy (see Figure 6.3) has been used in order to define the parameter window for which first order effects can be expected.



Figure 6.3 : The macroscopic scale (>1 mm) sketch is the typical geometry and loading configuration used to estimate the interface fracture energy. The first level window around the crack tip depicts the microscale problem ( $\approx 1.5 \ \mu m$ ), involving the interface between two thin elastic layers ( $h_e \approx 50-500$  nm) obtained by molecular bonding, the ductile interlayers  $(h_p \approx 100-2000 \text{ nm})$ on both sides of the elastic layers and the zone of large stresses surrounding the crack tip. The stresses can be predicted quite accurately within the thin elastic layer using linear elastic behavior and linear elastic fracture mechanics concepts except very near the crack tip within a zone of typically 5 atomic spacings. In the nanoscale zone (1-3 nm) shown on the lower-right, the modeling of the non linear effects related to the large distorsion and atomic plane separation requires the introduction of a traction-separation law. The response of the metallic interlayer under stresses larger than yield stress lead to plastic deformation and strain hardening mechanism related to the generation and gliding of dislocations along slip systems. A typical stress-response is shown in middle-right.

The different models used in this multiscale approach are presented in the next sections.

### 6.1.2 Constitutive models

### a. The interface traction-separation law

Following earlier efforts by Tvergaard and Hutchinson [66], the fracture process at the interface is modeled using a traction-separation law which relates the normal stress,  $\sigma$ , to the interface normal displacement,  $\delta$ , and which is characterized by the fracture energy of the interface noted,  $\Gamma_0$ , and a peak stress noted,  $\sigma_c$ . A schematic representation of this law is depicted in Figure 6.4.



Figure 6.4 : Schematic representation of a traction-separation law used to represent the response of the interface.

This type of model is called a Cohesive-Zone Model (CZM) and it was extensively used to model fracture at interfaces [36]. The traction-separation law proposed by Tvergaard and Hutchinson [66] (the trilinear function of Figure 6.4) has been chosen for this investigation.

The work of separation writes

$$\Gamma_0 = \sigma_c \delta_c \left( 1 - \frac{\lambda_1 + \lambda_2}{2} \right) \tag{6.2}$$

where  $\delta_c$  is the maximum separation (at fracture),  $\sigma_c$  the peak stress and  $\lambda_1$  and  $\lambda_2$  are two shape parameters of the curve. As discussed by Tvergaard and Hutchinson [66, 67], the parameters  $\lambda_1$  and  $\lambda_1$  are of secondary importance, and they will be taken equal to 0.15 and 0.5 in this study.

### b. Properties of the different layers

The different layers involved in the simulation were modeled using isotropic linear elasticity for the two elastic layers  $(Si \text{ and } SiO_2)$ :

$$\sigma = E\epsilon \tag{6.3}$$

and the isotropic elastic-plastic  $J_2$  flow theory for the ductile layer characterized by the uniaxial tension behavior given by:

$$\sigma = \begin{cases} E\epsilon & (\sigma \le \sigma_0) \\ \frac{\sigma_0}{(\sigma_0/E)^n} \epsilon^n & (\sigma > \sigma_0) \end{cases}$$

where E is the Young's modulus,  $\sigma_0$  is the yield stress and n is the work-hardening coefficient.

### 6.1.3 Numerical methods

The three layers are discretized according a typical finite element procedure (elements and nodes). The mesh used in the simulations is presented in Figure 6.5.



Figure 6.5 : FEM mesh used for modeling the opening wedge test with the steady-state framework. Drawing not to scale.

A linear kinematics finite element formulation for the study of steady-state crack propagation in elastoplastic materials was first applied by Dean and Hutchinson (1980) [68] and later implemented by several other authors [68, 69, 70]. This formulation has also been applied to rate dependent fracture of epoxy by Landis etal. [71]. The steady state FE method is based on finding an equilibrium solution for the displacements based on a previous approximate distribution of plastic strains in the ductile layer and then integrating the plasticity laws along streamlines to determine new approximations for stresses and plastic strains. This procedure is then repeated until convergence is achieved. More details about the formulation of the code can be found in Ref. [72]. Since the test is symmetrical, only half of the sandwich needs to be analyzed. Plane strain conditions are assumed. The wedge is modeled with a fixed boundary condition at a normalized distance from the plane of symmetry. Extremely fine meshes are required near the crack tip between the two elastic layers to properly resolve the fracture process taking place at the nanoscale and involving very small displacements. Very fine meshes are also necessary within the ductile interlayer to get converged solutions. Note that the analysis of the limit problem of a very thick ductile layer has been already provided by Wei and Hutchinson [73].

### Dimensional analysis of the problem

Dimensional analysis shows that the global toughness is a function of the following parameters:

$$\frac{\Gamma}{\Gamma_0} = F\left(\frac{\sigma_c}{\sigma_0}, \frac{E_p}{\sigma_0}, n, \frac{h_{el}}{h_p}, \frac{\Gamma_0}{\sigma_0 h_p}, \frac{E_p}{E_{el}}, \frac{E_p}{E_{el}}, \nu_{el}, \nu_p, \nu_s\right)$$
(6.4)

where  $h_i$  is the thickness,  $E_i$  is the Young's modulus,  $\nu_i$  is the Poisson ratio of the different layers. The ductile layer is also characterized by  $\sigma_0$ , the yield stress and n, the work-hardening exponent. The interface is characterized by the interface toughness,  $\Gamma_0$  and the peak stress,  $\sigma_c$ .

The different parameters involved in this analysis are summarized in Table 6.1

interface
$$\Gamma_0, \sigma_c$$
silicon oxide $h_{el}, E_{el}, \nu_{el}$ aluminum $h_p, E_p, \nu_p, \sigma_0, n$ silicon $h_s, E_s, \nu_s$ 

 Table 6.1 : List of parameters involved in the dimensional analysis.

The value of  $\Gamma$  represents the overall toughness of the sample and is composed of two terms:

$$\Gamma = \Gamma_0 + \Gamma_p \tag{6.5}$$

The value of  $\Gamma_p$ , required to calculate  $\Gamma$ , is computed using :

$$\Gamma_p = \int \sigma_{ij} d\epsilon^p_{ij} \tag{6.6}$$

where  $\epsilon^{p}_{ij}$  are the components of the plastic strains tensor.

One important parameter is the estimation of the size of the inelastic zone defined as [74]:

$$R_0 = \frac{E_p}{3\pi (1 - \nu^2)} \frac{\Gamma_0}{\sigma_0^2}.$$
(6.7)

This is the zone where the condition for plasticity is satisfied at initiation of cracking if the ductile interlayer was infinitely thick.

### 6.1.4 Numerical results

In most numerical results, the global toughness  $\Gamma$  divided by the toughness of the interface  $\Gamma_0$  is plotted as a function of the size of the inelastic zone  $R_0$  divided by the height of the inner elastic zone,  $h_{el}$ , as often presented in literature [73]. A typical numerical result is shown in Figure 6.6. All of the predicted curves show



**Figure 6.6 :** Typical evolution of the overall toughness as a function of  $R_0/h_{el}$  in the presence of an interlayer with  $h_p/h_{el}=10$ ,  $\sigma_c/\sigma_0=13$ , n=0.

the same evolution. First, the ratio  $\Gamma/\Gamma_0$  increases with increasing  $R_0/h_{el}$  (zone I), reaches a maximum (II) and then decreases (zone III). In the first zone, the increase is due to an increase of the ratio  $\Gamma_p/\Gamma_0$ ,  $\Gamma_p$  growing faster than  $\Gamma_0$  ( $\Gamma_0$  is proportional to  $R_0$ ). At the maximum of the curve, plasticity is fully developed in the ductile interlayer and the ratio  $\Gamma_p/\Gamma_0$  reaches a maximum. When  $R_0/h_{el}$  increases even more,  $\Gamma_0$  also increases whereas  $\Gamma_p$  now increases much slowly than  $\Gamma_0$ . Hence, the ratio  $\Gamma_p/\Gamma_0$  decreases leading to a decrease of  $\Gamma/\Gamma_0$ .

### Influence of the ductile interlayer thickness

Figure 6.7 shows the effect of the thickness of the ductile layer on the global toughness.



Figure 6.7 : Influence of the thickness of the ductile interlayer on the global interface toughness of the assembly. The other parameters are:  $\sigma_c/\sigma_0=13$ ; n=0.1;  $E_p/\sigma_0=1400$  i.e.  $E_p=70$  GPa and  $\sigma_0=50$  MPa.

Thicker ductile layers allow much larger dissipation when increasing  $R_0$ . The magnitude of  $\Gamma_p$  directly depends on the thickness of the ductile layer,  $h_p$ . At the maximum of the curve, the value of  $\Gamma_p$  is equal to 6.27 J/m<sup>2</sup> for the thicker ductile interlayer  $(h_p/h_{el}=10)$ , 2.94 J/m<sup>2</sup> for the intermediate thickness  $(h_p/h_{el}=5)$  and 0.77 J/m<sup>2</sup> for the thinner layer  $(h_p/h_{el}=1)$  for values of  $\Gamma_0$  equal to 1 J/m<sup>2</sup> in the three cases. Compared with samples without ductile layer, the toughness can thus be improved by more than 700% for ductile interlayer ten times thicker than the inner elastic layer. When the thickness of the ductile layer is reduced (e.g. for  $h_p/h_{el}=1$ ), the increase of the toughness is still equal to 77%.

### Influence of the ductile interlayer yield stress

Simulations have been run for three different ratio  $E_p/\sigma_0$  equal to 200, 700 and 1400. These values cover most of the metallic materials. For instance, in aluminum alloys (with E=70 GPa), yield stress ranging between 50 and 350 MPa are realistic. Figure 6.8 shows the variation of the ratio  $\Gamma/\Gamma_0$  as a function of  $\Gamma_0/(h_{el}\sigma_0)$ . The x-axis parameter was modified because one must look here at the result for the same  $\Gamma_0$  and E but changing only  $\sigma_0$ .



Figure 6.8 : Influence of the yield stress of the ductile interlayer on the global toughness of the assembly. The other parameters are equal to:  $\sigma_c/\sigma_0=13$ ;  $h_p/h_{el}=10$ , n=0.1.

For low value of  $\Gamma_0$ , the largest increase of global toughness is obtained for the low yield stress ductile interlayer. This trends is reversed when the toughness of the interface,  $\Gamma_0$ , increases i.e. the global toughness becomes higher for the ductile interlayer with the largest yield stress. At low values of  $\Gamma_0$ , the crack propagates easily along the interface and the stress field intensity ahead of the crack tip is relatively low. This low stress field allows plastic deformation only in lower yield stress ductile interlayer, the higher yield stress ductile interlayer remaining elastic. As the interface toughness increases, plasticity becomes fully developed in lower yield stress interlayer whereas only the near-to-crack tip regions of high yield stress ductile interlayer are deformed plastically. When the toughness of the interface increases again, the value of  $\Gamma_p$  increases slowly and finally saturates for ductile interlayer with low yield stress leading to a decrease of the ratio  $\Gamma/\Gamma_0$ . On the contary, in higher yield stress ductile interlayer,  $\Gamma_p$  still increases faster than  $\Gamma_0$ and the overall toughness keeps increasing further.

#### Influence of the work-hardening exponent of the ductile interlayer

The work-hardening exponent of a metal characterizes its ability to harden during deformation. The influence of the work-hardening exponent on the global toughness of the assembly is shown in Figure 6.9.



Figure 6.9 : Influence of the work-hardening exponent of the ductile interlayer on the global toughness of the assembly. The other parameters are equal to:  $\sigma_c/\sigma_0=13; h_p/h_{el}=10, E_p/\sigma_0=1400 \text{ i.e. } E_p=70 \text{ GPa and } \sigma_0=50 \text{ MPa.}$ 

At low value of  $R_0/h_{el}$  i.e. when  $\Gamma_0$  is low, the largest increase of the toughness is obtained with a ductile layer with the lowest work-hardening exponent. When  $R_0/h_{el}$  increases, the best improvement of the global toughness is obtained with an intermediate work-hardening exponent and we may expect that at higher value of  $R_0/h_{el}$ , the highest global toughness will be obtained with the highest workhardening exponent. This evolution matches the effect of  $\sigma_0$  presented in Figure 6.8.

### Influence of the strength of the interface

Figure 6.10 shows the variation of the overall toughness,  $\Gamma/\Gamma_0$  for different interface strength  $\sigma_c/\sigma_0$ .



Figure 6.10 : Influence of the strength,  $\sigma_c$  of the interface on the global toughness of the assembly. The other parameters are equal to: n=0.1;  $h_p/h_{el}=10$ ,  $E_p/\sigma_0=1400$ , i.e.  $E_p=70$  GPa and  $\sigma_0=50$  MPa.

The interface strength has a very important effect on the maximum toughening which can be expected by inserting a ductile interlayer. Increasing the crack tip stress increases the plastic work,  $\Gamma_p$ , hence the ratio  $\Gamma/\Gamma_0$ . In Figure 6.11, the global toughness is now plotted as a function of the ratio  $\sigma_c/\sigma_0$  keeping the ratio  $R_0/h_{el}$  constant as done in [73].



Figure 6.11 : Influence of the strength,  $\sigma_c$  of the interface on the global toughness of the assembly. The other parameters are equal to : n=0.1;  $h_p/h_{el}=10$ ,  $E_p/\sigma_0=1400$ . i.e.  $E_p=70$  GPa and  $\sigma_0=50MPa$ .

For a given value of the ratio  $R_0/h_{el}$  i.e. a given value of the toughness of the interface,  $\Gamma_0$ , the global toughness increases with the strength of the interface in any case. If  $\sigma_c/\sigma_0$  is smaller than 3, the yielding condition is never attained in the ductile interlayer and  $\Gamma_p \approx 0$  [66]. The increase of toughening with increasing interface strength is very large when  $\sigma_c/\sigma_0$  increases from 3 to about 5-7. Then the the rate of increase levels off. Note that a maximum of toughening is attained for intermediate value of  $R_0/h_{el}$ .

It seems thus that an optimum in the value of  $\Gamma_0$  leads to a maximum increase of the global toughness. Indeed, when the value of  $\Gamma_0$  is too large  $(R_0/h_{el} = 80$ in Figure 6.11), the relative improvement of the global toughness is smaller than for  $R_0/h_{el} = 50$ . This effect can be explained by the fact that if  $\Gamma_0$  increases,  $\Gamma_p$
saturates leading to a decrease of the relative improvement of the global toughness.

An important outcome of this numerical study is the possibility to select the properties of the ductile layer leading to a high improvement of the global toughness,  $\Gamma$ . A thicker ductile layer with a low yield stress and a low work-hardening coefficient is the combination of the parameters providing maximum toughening for low toughness interfaces,  $\Gamma_0$ . When the toughness of the interface increases, maximum toughening is obtained for ductile layer with high yield stress and work-hardening exponent.

## 6.2 Influence of a the ductile layer Experimental results

The profilometer test method (presented in Chapter 3) was applied both on samples with and without a ductile interlayer deposited in between a silicon oxide and the silicon substrate. The complete procedure used to manufacture these samples can be found in Chapter 2. The main characteristics of the ductile layer and the silicon oxide are recalled hereafter.

The ductile interlayer is made of pure aluminum deposited by an evaporation process. The thickness of this aluminum layer is equal to 1  $\mu$ m and its mechanical properties are E=70 GPa and  $\sigma_0$  is about 100 MPa (see Appendix B). The silicon oxide involved in the bonding between the two wafers is a PECVD silicon oxide, subjected to a CMP surface treatment in the two types of samples resulting in a thickness of about 350 nm.

The crack length and the toughness<sup>1</sup> measured for the two types of specimens are given in Figure 6.12.

<sup>&</sup>lt;sup>1</sup>The relationship used to obtain the toughness from the crack length (see Eq. (3.30)) is only valid in the elastic regime which is true here because the thickness of the ductile layer (1  $\mu$ m) is very small compared to the thickness of the silicon substrate (380  $\mu$ m).



(b)

Figure 6.12 : (a) Crack length values (thickness of the wedge= $100\mu m$ ) and (b) fracture toughness of samples without and with a ductile interlayer.

	Without ductile layer	With ductile layer
$a \ [mm]$	$9.4 \pm 0.3$	$8.4 \pm 0.3$
$G_c  [\mathrm{J/m^2}]$	$2.2 \pm 0.3$	$3.4 \pm 0.5$

The mean values of Figure 6.12 are summarized in Table 6.2.

**Table 6.2 :** Comparison of crack length (thickness of the wedge= $100\mu m$ ) and toughness obtained on samples with and without a ductile interlayer.

The effect of the presence of the ductile layer is obvious : the toughness of samples with ductile layer is larger than the toughness of specimens without ductile interlayer. This relative improvement of the toughness is equal to

$$\frac{\Gamma}{\Gamma_0} = \frac{3.4}{2.2} = 1.55. \tag{6.8}$$

This increase of the toughness is still limited when compared to the maximum possible toughening predicted by the model. This can be explained by the fact that the ratio of the ductile interlayer thickness to the thickness of the elastic layer,  $h_p/h_{el}$ , is equal approximately to 3 ( $h_p = 1\mu m$ ,  $h_{el} = 350 \text{ nm}$ ). From Figure 6.7 and by calculating  $R_0/h_{el}$  to be equal to about 5, a ratio  $\Gamma/\Gamma_0$  between 1.5 and 2.5 is expected.

## 6.3 Inverse determination of the interface strength

As previously explained in Section 3.4, the determination of the strength  $\sigma_c$  is not possible using a simple tensile test. A new methodology based on the coupling between experiment and simulation is proposed and applied on a first set of results. More precisely, the idea is to study exactly the same interfaces in the presence of a thin ductile interlayer inserted near the interface for the first type of samples and without ductile interlayer for the second type. Interfaces with and without ductile interlayer being exactly the same<sup>2</sup>, a unique common couple ( $\Gamma_0, \sigma_c$ ) should be

 $<sup>^{2}</sup>$ We assume that the interface is similar for the two types of specimens even if the deposition of PECVD silicon oxide on a silicon substrate or on a aluminum thin layer can lead to silicon oxide with different internal stresses and thus different chemical reactivity.

found for these interfaces by using Eq. (3.30). The crack length of samples without a ductile interlayer is determined from experimental tests. This crack length is used to determine the interface fracture toughness,  $\Gamma_0$ , of the interface.

The crack length of samples with a ductile interlayer and the material properties  $(E, \sigma_0, \nu, n)$  of the different layers  $(SiO_2, Al \text{ and } Si)$  are used as input in the simulations. These material properties are determined previously by nanoindentation (see Appendix B). By varying  $\sigma_c$  in the simulations, an infinity of couples  $(\Gamma_0, \sigma_c)$  which matches the experimental crack length can be found. The value of  $\Gamma_0$  is known from specimen without ductile layer and the value of  $\sigma_c$  can be thus deduced from samples with ductile interlayer.

This procedure is schematically shown in Figure 6.13.



Figure 6.13 : Schematic representation of the procedure used to determine the strength,  $\sigma_c$ , of the interface.

Applying this procedure to experimental results presented in the previous section allows determining the properties of the (PECVD) $SiO_2$ / (PECVD) $SiO_2$  interface. The different couples ( $\Gamma_0$ ,  $\sigma_c$ ) matching the experimental crack length of samples with a ductile interlayer and the constant toughness of samples without a ductile interlayer are plotted in Figure 6.14 for various ductile interlayer yield strength. The intersection between the curves corresponding to specimens with a ductile interlayer and the constant amplitude curve (specimens without ductile interlayer) is the couple ( $\Gamma_0$ ,  $\sigma_c$ ) representative of the fracture properties of the interface.



Figure 6.14 : Influence of the yield strength of the ductile interlayer on the determination of the characteristic couple ( $\Gamma_0$ ,  $\sigma_c$ ) of the (PECVD)SiO<sub>2</sub>/(PECVD)SiO<sub>2</sub> interface.

As seen on Figure 6.14, the interface strength,  $\sigma_c$ , largely increases with the yield strength of the ductile interlayer. The value of the yield strength of the ductile interlayer determined by nanoindentation is equal to 100 MPa, giving a value of interface strength equal to 350 MPa. This value is too low regarding theoretical values obtained in the literature [44, 45]. However, the yield strength of fully constraint 1  $\mu$ m Al layer can be higher than the value of 100 MPa determined on free Al layer by nanoindentation. Increasing the yield strength of the Al interlayer from 100 MPa to 150 MPa largely increases the interface strength and a interface strength value,  $\sigma_c$ , of about 5 to 10 GPa is expected, in better agreement with theoretical values. The critical displacement at which fracture occurs,  $\delta_c$ , is thus equal to about 3 to 6 Å. Chapter 7

# **Conclusions and Perspectives**

Wafer bonding is a process by which two wafers adhere to each other at room temperature without the addition of any macroscopic gluing layer.

Even if the process of molecular bonding has been known for many centuries, the application of this process in the microelectronic industry emerged only since the end of 70's. Nowadays, the application of the wafer bonding process to silicon wafers is largely used in the microelectronic industry for applications like pressure sensors, accelerometers, epitaxial transfer layer, ...

Unfortunately, and particularly in the case of Micro-Electro-Mechanical Systems (MEMS) where some parts of the system move during the operation of these devices, the interfaces created by the wafer bonding process constitute a preferential locus of failure and, thus, significantly decrease the reliability of the MEMS. The improvement of the uniformity and strength of the "wafer bonding" interfaces

This thesis aimed at the determination and the optimization of the cracking resistance of interfaces created by silicon wafer bonding.

is thus a key for the large scale use of MEMS.

Proper understanding of the molecular reactions occurring during the bonding process and of the bond breaking phenomena is essential for correctly interpreting the experimental results.

The adhesion of two silicon wafers is due to the formation of strong covalent bonds between the two wafers. This process is usually divided into three steps:

• the silicon oxide (native or "artificial") covering the silicon substrate reacts

with "external activators" (chemical species contained in the cleaning bath, plasma activation, ...) and form silanol groups (-Si - OH) on the top of the silicon wafers surface,

- these silanol groups form weak hydrogen bonds with water molecules and, previous weak links are achieved across the two silicon wafers,
- appropriate storage time and annealing transforms the weak hydrogen bonding into strong covalent bonds (Si - O - Si), responsible for the high strength of "wafer bonding" interfaces.

The complete process is depicted in Figure 7.1.



Figure 7.1 : Schematic representation of the chemical process occurring when two silicon wafers are contacted, stored and annealed.

The key point of the bonding process is probably the absorption of the byproducts of the polymerization reaction. Indeed, the formation of the strong covalent bond (Si - O - Si) from two silanol groups produces water molecules:

$$2Si - OH \longleftrightarrow Si - O - Si + H_2O. \tag{7.1}$$

Unfortunately, these byproducts are responsible of the formation of large unbonded areas and can promote debonding by dissociating the strong covalent bond. Some options can be used to absorb these byproducts. The byproducts can diffuse either to the outer boundary or into the silicon oxide or are trapped into cavities etched at the interface.

### Optical and mechanical tests have been developed for determining the uniformity and the strength of the "wafer bonding" interfaces.

The transparency of silicon to infrared radiation has been used to observe the uniformity of the bonding and to determine the Si/Si interface fracture toughness. A home-made steady-state wedge-opening test has been developed in order to determine the interface toughness by measuring the crack length initiated at the interface by the insertion of a wedge between the two silicon wafers [37]. The originality of the steady-state wedge test is that it continuously measures the crack length propagating at the interface. This method allows the determination of the real crack length avoiding edge effects and misorientation that can result from a manual insertion of the wedge. Using this technique, we have observed that the highest interface fracture resistance is obtained when the silicon wafers are submitted to an optimized  $O_2$  plasma treatment. These results have driven further research on that specific surface treatment [53, 54]. In some cases, the interface cracking resistance is so high that the crack propagates into the silicon substrate. The annealing step is also necessary to obtain a high interface toughness. The main interface toughness results obtained in the framework of this thesis are summarized in Appendix A.

A second experimental method based on the measurement of the curvature of wafers has been developed in order to measure the crack length resulting from the insertion of a wedge. This method avoids the error made on the crack length measurements by using IR radiation (see Appendix D) and allows the determination of the interface fracture toughness of non-IR transparent assemblies. Unfortunately, this method is only applied, for the moment, with the static version of the wedge test. The continuous determination of the wafer curvature during a steady-state wedge-opening test by using optical method (e.g. interferometry) could be an interesting procedure for the determination of interface fracture toughness of bonded wafers.

Crack propagation is influenced by environmental chemical species, and particularly water molecules.

The use of the steady-state wedge opening test has allowed evidencing the effect of environmental species, and particularly water molecules, on the crack propagation at the interface. Indeed, at low crack propagation rate, water molecules adsorb on the covalent bond linking the two silicon wafers, dissociate it, resulting in a lower measured interface fracture toughness. On the contrary, at high crack propagation, environmental species do not influence the crack propagation process and the "intrinsic" interface fracture resistance can be measured. This phenomenon can reduce drastically the reliability of devices working in wet atmosphere (e.g. microfluidic applications).

#### The interface fracture resistance of patterned wafers is lower than of plain wafers.

In real structures, bonded silicon wafers are often patterned. The cavities, required by the design of the device, can be used to trap the byproducts of the polymerization reaction. In order to observe the effect of these cavities, the influence of long channels on the interface toughness has been studied. The conclusion was that the interface toughness of patterned wafers is always lower than of plain wafers. The influence of these cavities on the bonding and debonding behavior of silicon wafers is still not fully understood. Some hypothesis have been advanced to explain this effect:

- an influence of the cavities on the bonding wave propagation;
- an influence of the cavities on the elastic deformation energy stored in the wafers;
- the cavities can contain chemical species which could promote an initial decrease of interface toughness during the storage time between the end of the fabrication process and the testing of the specimens;
- an influence of the cavities on the action of water molecules during crack propagation.

However, none of these assumptions were satisfactory to explain all the results. In order to explain the decrease of the interface toughness in patterned silicon wafers, some complementary tests should be performed by varying the size and the space between these long channels.

A ductile interlayer deposited in-between the substrate and the silicon dioxide is used to increase the global toughness of the Si/Si assembly.

Modeling and simulations tools have allowed us to quantify the effect of a ductile interlayer on the global toughness of the assembly. An increase of the global toughness by a factor ranging between 3 and 7 can be obtained by adjusting the mechanical properties and thickness of the ductile interlayer. Moreover, the numerical method can be used to determine the geometrical and material properties of the ductile layer leading to a pre-defined global toughness.

The specimens with a ductile interlayer have also been used in order to determine the "theoretical" strength of interface that cannot be determined experimentally by simple tensile tests. By coupling simulations and experimental results, we conclude that the "theoretical strength" of Si/Si interfaces depends largely on the yield strength of the ductile layer. For a 1  $\mu$ m thick Al layer with a yield strength of 150 MPa, a value of the interface strength ranging from 5 to 10 GPa is expected. This value is impossible to determine experimentally by tensile tests.

The three following sentences extracted from an article of the April 2006 issue of "*The international newsletter on micro-nano integration* - MST News" show us the importance of having reliable characterization, simulation and modeling tools for improving fabrication and reliability of microsystems.

"Many typical applications of microtechnologies require very high reliability, and suitable test methodologies, standards and instrumentation are often missing."

"From the modeling point of view, the most important challenges to address are those tools supporting multiphysics, multiscale and multirate simulations. That means that several phenomena at different abstraction levels shall be coupled." "Design, modeling and simulation tools enable users to assess, in "realtime", concepts and options for a variety of complex products. Rapid prototyping for micro- and nano-technologies will be delivered through the integration of such tools within the fabrication process."

At the scale of the silicon wafer bonding process which is more and more used in microelectronic industry, this thesis proposes test methodologies for a proper determination of the interface fracture properties (toughness and strength) of silicon bonded wafers. The determination of the fracture toughness AND the strength of "wafer bonding" interfaces is of practical importance for the future development of devices or MEMS using wafer bonding as a step of their fabrication process. The use of these properties in simple fracture models like cohesive-zone model will allow to predict the behavior of real structures under complex loading and guide the designers for choosing geometry, materials and process to make MEMS reliable. Appendix A

## Summary of the main results

The main interface toughness results obtained in the framework of this thesis are listed in Table A.1. For convenience, the standard cleaning procedure used in the clean rooms of UCL is recalled hereafter.

The wafers are first dipped in a sulfuric solution  $(H_2SO_4(96\%) : H_2O_2(30\%) = 5 :$ 2) for 10 minutes at 100 °C. Next, the wafers are rinsed in two separate DI (deionized) water containers for 5 minutes in each container. The wafers are dipped again in a second sulfuric solution. The native oxide which can contain impurities in the bulk or on the surface is then removed by dipping the wafers in fluorhydric acid (HF 2%) for about 15 seconds. In order to recover the hydrophilicity of the wafers, the wafers are dipped again in the second sulfuric solution. A clean "native" oxide of a few angströms is created. The wafers are finally rinsed and dried.

Wafers covered by thermal or PECVD oxide have been subjected to the same cleaning sequence prior the oxidation step. After the oxidation step, the wafers have been cleaned again following the same procedure except for the dipping in HF which remove the silicon oxide.

Wafers covered with an aluminum thin layer and a silicon oxide thin layer (see Chapter 6) are cleaned before aluminum deposition and after silicon oxide deposition except for the HF dipping.

The plasma treatment was optimized by Dr. Zhang during his postdoctoral visit at UCL-EMIC. The parameters are an  $O_2$  flow of 100 sccm, 90mTorr chamber pressure and about 300W bias. The wafers were exposed to an  $O_2$  plasma treatment during 5 sec.

Surface treatment	Annealing	Cracking	Interface fracture
	Conditions	rate	toughness
		[mm/min]	$[J/m^2]$
standard cleaning and storage during 150 hours with a	Noonoling	0.25	$0.05 {\pm} 0.015$
dead weight.	INO annicannig	25	$0.25 {\pm} 0.15$
		0.05	$1.1 {\pm} 0.10$
standard closning and storage during 150 bours with a		0.25	$1.3 {\pm} 0.10$
dood weight	150h @ 150°C	2.5	$1.4{\pm}0.10$
near weighte.		25	$2.3\pm0.15$
		200	$2.2{\pm}0.10$
standard cleaning, $O_2$ plasma treatment		0.25	$2.607 {\pm} 0.30$
(20W-10s-4Pa.) and storage during 150 hours with a dead weight	150h @ 150°C	25	$3.3 {\pm} 0.25$
		0.1	$2.2{\pm}0.10$
standard closning and entimized O- plagma treatment	10h @ 1900	1	$2.7{\pm}0.10$
Standard creating and optimized $O_2$ prasma treatment.		10	$3.5 {\pm} 0.20$
		50	$3.9\pm0.20$
standard cleaning, PECVD oxide, KEMESYS CMP and optimized $O_2$ plasma treatment.	24h @ 120°C	10	$2.2 {\pm} 0.30$
standard cleaning, 1 $\mu$ m Al interlayer, KEMESYS CMP, PECVD oxide, KEMESYS CMP and optimized	24h @ 120°C	10	$3.4{\pm}0.50$
$O_2$ plasma treatment.			

Table A.1 : Summary of the main results obtained in the framework of this thesis.

Appendix B

## The nanoindentation technique

The instrumented nanoindentation technique is used in order to determine the mechanical properties (Young's modulus, E and hardness, H) of thin layers ( $SiO_2$  and Al) deposited on the silicon substrate. This technique allows the measurement of several mechanical properties at the sub-micron scale and is, therefore, well suited for thin layers with thickness varying between 5 nm and 5  $\mu$ m. First, the basic principles of nanoindentation are presented. The application of this technique to thin films and the modifications used to take into account the

substrate effects on the measurements are presented in the second section.

Finally, results obtained on silicon, silicon oxide and aluminum are presented.

### **B.1** Experimental setup

The basic principle of any indentation technique is to penetrate a material with a tip of known and controlled geometry. The complete system is shown in Figure B.1.



Figure B.1 : Block diagram of nanoindenter coupled to AFM system.

In our case, the nanoindenter is mounted on an Atomic Force Microscopy (AFM) system that allows imaging surfaces before and after indentation. Parts of the AFM system are represented in Figure B.1 in non-shaded boxes whereas the nanoindentation system is represented in shaded boxes. The two most important components are the piezo-actuator which allows the surface to be scanned by the indenter tip and the transducer (a three-plate capacitive force/displacement sensor). A cross section of the transducer is shown in Figure B.2.



Figure B.2 : Cross section schematic of the transducer.

This transducer generates the load force and simultaneously measures the force and resulting displacement.

A typical load/displacement curve is shown in Figure B.3.



Figure B.3 : Typical load-displacement obtained during a nanoindentation test.

## B.2 Determination of the mechanical properties by nanoindentation

#### B.2.1 General analysis

The recorded force-displacement curve allows the determination of the Young's modulus and the hardness of the indented material. The method relies on that the displacements recovered during unloading are largely elastic, in which case elastic punch theory [75] can be used to determine the modulus, E, from a simple analysis of indentation load-displacement data. Referring to the schematic plot of indentation load, P, vs displacement, h in Figure B.3, the most classical method relates E to the initial unloading stiffness, S = dP/dh. The basic assumption is that during the initial withdrawal of the indenter, the contact area between the indenter and the specimen remains constant, in which case the analysis of Sneddon

for the indentation of an elastic half space by a flat, cylindrical punch approximates the behavior [75]. Sneddon's analysis leads to a simple relation between P and hof the form:

$$P = \frac{4\mu a}{1-\nu}h\tag{B.1}$$

where a is the radius of the cylinder (the area of the contact circle, A is then  $\pi a^2$ ),  $\mu$  is the shear modulus that can be related to the elastic modulus through  $E = 2\mu(1+\nu)$ , and  $\nu$  is the Poisson ratio. Differentiating P with respect to h leads to:

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}}\sqrt{A}\frac{E}{1-\nu^2}.$$
(B.2)

It is thus seen that the modulus can be computed directly from the initial unloading slope, provided a reasonable estimate of Poisson ratio and an independent measure of the contact area, A, are available. This contact area is determined using a specimen of well-known properties  $(E, \nu)$  and by using Eq. (B.2). Contact areas for several common indenter geometries are shown in Figure B.4.



**Figure B.4 :** Projected contact area for different type of indenter tip: (a) conical; (b) Vickers; (c) Berkovich.

In the case of the indenter itself has finite elastic constants,  $E_i$  and  $\nu_i$ , so that its deformation contributes to the measured displacement, it is convenient to define a reduced modulus,  $E_r$ ,

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}.$$
(B.3)

The relationship (B.2) reduces then to:

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}}\sqrt{A}E_r.$$
 (B.4)

It is important to note that as derived, Eq. (B.4) is formally limited to those instances which the indenter behaves as a flat cylindrical punch. Nevertheless, the indenters used most commonly in load-displacement sensing indentation techniques are not flat punches, but rather square-based or triangle-based pyramids (Vickers or Berkovich indenters). However, it has been shown recently that relationships (B.2) and (B.4) are more universal than has generally been recognized. In fact, these equations are valid for any indenter whose geometry can be described by a solid of revolution with a smooth function (cone, sphere, paraboloid of revolution, ...) [76]. Unfortunately, Berkovich and Vickers indenters cannot be described as bodies of revolution, but it appears that even this does not place severe restrictions on the use of Eq. (B.4). This last conclusion has been derived from 3D finite element calculations of the load-displacement characteristics of an elastic half space deformed by flat ended punches with circular, triangular and square cross section performed by King [77]. The latter two geometries, which cannot be described as bodies of revolution, are the flat-ended equivalents of the Berkovich and Vickers indenters. King found that for all three geometries, the unloading stiffness is given by

$$S = \frac{dP}{dh} = \beta \frac{2}{\sqrt{\pi}} \sqrt{A} E_r \tag{B.5}$$

where the values of the constant  $\beta$  are listed in Table B.1.

circular: 
$$\beta$$
=1.000  
triangular:  $\beta$ =1.034  
square:  $\beta$ =1.012

# **Table B.1 :** Values of the constant $\beta$ for the three typical geometries of indenters (from[77]).

The relationship (B.5) is similar to Eq. (B.4) except for the geometric factor  $\beta$  which deviates from the circular one by only 3.4 % and 1.2 % for the triangular and square geometries respectively. Vlassak and Nix [78] later conducted independent numerical calculations for the flat-ended triangular punch using a more precise

method and found a higher value of  $\beta$ =1.058. Hendricks, noting that the pressure distribution for elastic deformation by a flat punch is not representative of the real elastic-plastic problem, adopted another approach to estimate  $\beta$  [79]. Assuming the pressure profile to be perfectly flat, he used simple elastic analysis procedure to show that  $\beta$ =1.0055 for a Vickers indenters (square-based pyramid) and  $\beta$ =1.0226 for the Berkovich. Larson *et al.* [80] conducted full 3D finite element calculations of true Berkovich indentation for a purely elastic material and four different elastic-plastic materials that simulate the behavior of aluminum alloys. For the purely elastic material, they found that  $\beta$  = 1.14. However, this solution applies to indentation of flat, elastic half-space and thus inherently ignores the fact that plasticity severely distorts the surface during the formation of hardness impression. Because of this, one might expect better results from the elasto-plastic simulations because they account for plasticity. For the four elastic-plastic materials examined, the value  $\beta$  = 1.034 suggested by King was found to work well, producing errors no more than 6.5 % in the contact area and hardness.

The wide range of  $\beta$  values makes it difficult to decide for a single preferred value. However, there are strong reasons to expect that  $\beta$  for the Berkovich indenter should be greater than unity. The deviation from circular cross-section appears to play an important role, as does the fact that the original analysis from which Eq. (B.5) was derived ignores the radial displacements of the surface for a conical indenter. Ignoring values coming from elastic analysis, the remaining values fall in the range  $1.0226 \leq \beta \leq 1.085$ . Thus,  $\beta=1.05$  is probably a good choice , with a potential maximum error of approximately  $\pm 0.05$ . Carefully performed experiments and 3D finite element simulations that take care to insure good convergence and accuracy could help resolving this issue.

Looking at Eq. (B.5), the two parameters we need to determine the Young's modulus are the initial unloading stiffness, S = dP/dh, and the contact area of the indenter, A. Moreover, the hardness of the indented material can be determined by

$$H = \frac{P_{max}}{A} \tag{B.6}$$

where  $P_{max}$  is the maximum force imposed to the indenter and A is the contact area of the indenter.

#### Determination of the initial unloading stiffness, S

Unloading data are well described by a power law relationship:

$$P = K(h - h_f)^m \tag{B.7}$$

where the constants K, m, and  $h_f$  are all determined by a least square fitting procedure. The initial unloading slope S is then found by analytically differentiating this expression and evaluating the derivative at the peak load and displacement.

$$S = \left. \frac{dP}{dh} \right|_{h_{max}} = mK(h_{max} - h_f)^{m-1}.$$
 (B.8)

#### Determination of the contact area A

Figure B.5 shows a cross section of an indentation profile and the definition of the parameters used in the analysis.



Figure B.5 : A schematic representation of a section through an indentation showing the various parameters used in the analysis.

At any time during loading, the total displacement h is written as

$$h = h_c + h_s \tag{B.9}$$

where  $h_c$  is the vertical distance along which contact is made (hereafter called the contact depth) and  $h_s$  is the displacement of the surface at the perimeter of the contact. At peak load, the load and displacement are  $P_{max}$  and  $h_{max}$ , respectively.

Upon unloading, the elastic displacements are recovered, and when the indenter is fully withdrawn, the final depth of the residual hardness impression is  $h_f$ . The experimental parameters needed to determine hardness and modulus are shown in the schematic load-displacement data in Figure B.6.



Figure B.6 : A schematic representation of load vs. indenter displacement showing the parameters used in the data reduction, as well as graphical interpretation of the contact depth.

The area of contact at peak load, A, is determined by the geometry of the indenter and the depth of contact,  $h_c$ . The area of contact can be described by an area function F(h) which relates the cross-sectional area of the indenter to the distance from its tip, h. Given that the indenter does not itself deform significantly, the projected contact area at peak load can then be computed from:

$$A = F(h_c). \tag{B.10}$$

The functional form of F must be established experimentally prior to analysis by indenting a material of well-known mechanical properties (E, H). In order to

determine the contact depth from experimental data, it is noted that:

$$h_c = h_{max} - h_s. \tag{B.11}$$

Since  $h_{max}$  can be measured experimentally, the key to the analysis is to infer the displacement of the surface at the contact perimeter,  $h_s$ , from the load-displacement data. The deflection of the surface at the contact perimeter depends on the indenter geometry. For a conical indenter, Sneddon's expression [75] for the shape of the surface outside the area of contact can be used to give:

$$h_s = \epsilon \frac{P_{max}}{S} \tag{B.12}$$

where the geometric constant  $\epsilon$  is given in Table B.2 for different tips geometry.

Conical: $\epsilon = 0.72$ Flat punch: $\epsilon = 1$ Paraboloid of revolution: $\epsilon = 0.75$ 

**Table B.2** : Value of the constant  $\epsilon$  for different geometries of the indenter.

The contact depth can then be determined by inserting Eq. (B.12) into Eq. (B.11):

$$h_c = h_{max} - \epsilon \frac{P_{max}}{S} \tag{B.13}$$

where all the parameters are known from the load vs displacement curve (see Figure B.6). It is now possible to determine the Young's modulus and hardness of the indented materials:

$$E_r = \frac{\sqrt{\pi}S}{2\beta\sqrt{A}};\tag{B.14}$$

$$H = \frac{P_{max}}{A} \tag{B.15}$$

where S is the initial unloading slope,  $A = F(h_c)$  is the contact area,  $P_{max}$  is the maximum force and  $\beta$  is a constant dependent of the shape on the indenter. Unfortunately, the use of these relationships can lead to relatively large errors on the values of E and H. These errors are mainly due to errors on the determination of the contact area  $A = F(h_c)$ . Indeed, contact area may be modified by pile-up and sink-in effects occurring when an elasto-plastic material is indented as explained in the next section.

#### B.2.2 Pile-up and sink-in

In an indentation into an elastic material, the surface of the specimen is typically drawn inwards and downwards underneath the indenter and sinking-in occurs. When the contact involves plastic deformation, the material may sink-in or pile-up around the indenter. Pile-up and sink-in are shown in Figure B.7. The influence of these two effects on the contact area are also shown in Figure B.7.



Figure B.7 : Piling-up (a) and sinking-in (b) occuring during an indentation test. The effect of pile-up and sink-in on the contact area are shown on the plan view of indentation. Dashed lines represents the indenter cross-sectionnal area and solid lines represents the actual contact area.

A well-annealed soft metal that exhibits a high strain-hardening rate will tend to show far-field plasticity. Strain hardening near the indenter tip will cause plastic deformation to occur further and further away from the contact, causing material to be displaced far away from the indentation and resulting in sink-in behavior. This sink-in of the surface causes the actual contact area to be smaller than the cross-sectional area,  $A = F(h_c)$ , of the indenter at this depth. By contrast, strainhardened materials and metallic glasses that exhibit a low strain-hardening rate will deform more locally, creating a pile-up of material against the sides of the indenter. The displacement of the material near the indentation results in a significant increase in contact area into a given depth.

Some techniques are proposed in the literature to take account the effect of pile-up and sink-in on A [81, 82]. Chen and Vlassak [26] make a comprehensive analysis of nanoindentation with full account of pile-up and sink-in. In Figure B.8, a comparison between the Oliver and Pharr model presented in Section B.2 and a numerical study of the indentation process shows that for hard materials (characterized by a low value of the parameter  $Etan(\beta_0)/\sigma_y$ ), there exists a good agreement between the analytical model of Oliver and Pharr and the numerical study.



**Figure B.8**: Plot of the normalized height of pile-up as a function of the yield strength (from [26]).

The value of  $\beta_0$  is about 20° and is equal to 90°  $-\alpha$  where  $\alpha$  is the half apex angle of a rigid cone indenter. This type of indenter was chosen for FEM analysis, so that the ratio of cross-sectional area to indentation depth is the same one as for a Berkovich or Vickers indenter. The value of  $\delta_p/\delta$  is the height of the pile-up divided by the indentation depth. This parameter is negative for material presenting sinking-in

and positive for materials showing piling-up effect. The mechanical properties (E,H) of hard materials presenting sink-in can thus be determined using the Oliver and Pharr method. However, this model cannot be applied to indentations in soft materials. The discrepancy between the Oliver-Pharr model and the FEM results for large values of  $Etan(\beta_0)/\sigma_y$  is of course due to pile-up of material around the indenter and can lead to an underestimation of the contact area by as much as 66%. Numerical study have shown that extended pile-up occurs in materials where  $h_f/h_{max} > 0.7$  (see Figure B.6) [83]. In an indentation experiment, care must be exercised when  $h_f/h_{max} > 0.7$ , as use of the method can lead to large errors in the contact area. On the other hand, when the pile-up is small (i.e;  $h_f/h_{max} \leq 0.7$ ), the contact areas  $A(h_c)$  given by the classical method match very well with the true contact area obtained from finite element analysis. As a practical matter, if there is suspicion that pile-up may be important based on the value of  $h_f/h_{max}$ and/or other independent knowledge of the properties of the material, indentations should be imaged to examine the extent of pile-up and establish the true area of contact. For Berkovich indenter, indentations with a large amount of pile-up can be identified by the distinct bowing out at the edges of the contact impression. If pile-up is large, accurate measurement of H and E cannot be obtained using the contact area deduced from load-displacement data; rather, the area measured from the image should be used to compute H and E from Eq. (B.14) and Eq. (B.15). Cheng and Cheng developed a method that can be used to correct for pile-up in a manner that does not involve imaging the contact impression [84, 85, 86]. The method they proposed, based on finite element calculations, to account for pile-up is based on the work of indentation [86], which can be measured from the areas under indentation loading and unloading curves. They established two independent relationships between H and E allowing the determination of these two parameters:

$$\frac{W_{tot} - W_u}{W_{tot}} \cong 1 - 5\frac{H}{E_r} \tag{B.16}$$

$$\frac{4}{\pi} \frac{P_{max}}{S^2} = \frac{H}{E_r^2} \tag{B.17}$$

where  $W_{tot}$  is the total work of indentation (area under the loading curve) and  $W_u$  is the work recovered during unloading (area under the unloading curve). These quantities are all measurable from load-displacement data. Unfortunately, these method has never been tested experimentally.

#### **B.2.3** Application of nanoindentation to thin films

Determination of the mechanical properties of thin films on substrates by indentation has always been difficult because of the influence of the substrate on the measured properties [26, 87, 88]. The indentation response of a thin film on a substrate is a complex function of the elastic and plastic properties of both the film and substrate. The standard method used for extracting properties from the measured load-displacement data were developed primarily for monolithic materials [89]. These same methods are often applied to film/substrate systems for determining film properties without explicit consideration of how the substrate influences the measurements. In order to measure film-only properties, a commonly used rule of thumb is to limit the indentation depth to less than 10% of the film thickness [89]. Unfortunately, this rule cannot be applied in the case of very thin film and is sometimes too restrictive when dealing with soft films on hard substrates as seen hereafter. In our case, we have to determine the properties of Allayers on Si substrate. In Figure B.9 [26], the ratio  $H/H_b$  is plotted versus the normalized indentation depth  $\delta/h$  for the case of soft film on hard substrate. H is the hardness of the film material,  $H_b$  is the hardness of the bulk material,  $\delta$  is the indentation depth and h is the film thickness.



Figure B.9 : Variation of the normalized film hardness with normalized indentation depth  $\delta/h$  as a function of  $\sigma_f/\sigma_s$  (from [26]).

Figure B.9 shows that the value of the ratio  $H/H_b$  remains constant and equal to 1 until the indentation depth is about 50% of the film thickness. This criterion is then less restrictive than the 10 % criterion allowing larger indentation depth in such type of film/substrate combination. This 50 % rule cannot be used in other configurations than soft film on hard substrate as explained in details in [26]. For the case of  $SiO_2$  films on silicon substrate, this rule cannot thus be applied. However, as shown in Figure B.10, the normalized hardness  $H/H_b$  remains constant and equal to 1 for material having the same mechanical properties (E and  $\sigma_y$ ) for normalized indentation depth  $\delta/h$  ranging from 0.2 to 1. Silicon and silicon dioxide presenting almost the same stiffness [24], the indentation depth will be not limited by the influence of the substrate. However, this indentation depth was limited to 50% of the film thickness as for aluminum films.



Figure B.10 : Variation of the normalized film hardness  $H/H_b$  with normalized indentation depth  $\delta/h$  as a function of  $\sigma_f/\sigma_s$  for material having the same Young's modulus E (from [26]).

## **B.3** Determination of the mechanical properties of the Si substrate, $SiO_2$ and Al thin layers by nanoindentation

#### B.3.1 Nanoindentation of silicon

The silicon wafers used in the framework of this thesis were 3in. commercially available one-side polished,  $\langle 100 \rangle$  oriented, p-type and 15-25  $\Omega cm$  resistivity. The evolution of the hardness and Young's modulus of the silicon is shown in Figures B.11 and B.12. The evolution of hardness of Si with indentation depth is relatively constant equal to about 10 GPa.



Figure B.11 : Evolution of the hardness, H, of silicon for various indentation depths.

This value is in agreement with values from the literature [90]. The value of the Young's modulus decreases from a value of 165 GPa to about 125 GPa with indentation depth.



Figure B.12 : Evolution of the Young's modulus, E, of silicon for various indentation depths.

The decrease of the Young's modulus can be explained by the phase transformation of silicon during a nanoindentation test. Indeed, it is well known from experiments and theoretical studies [91, 92, 93] that silicon transforms from the diamond cubic structure (dc) denoted Si - I into the metallic  $\beta$ -tin structure (Si - II) under an hydrostatic pressure in the range of 11-13 GPa. On release of the pressure, the material first reverts to Si - XII(r8) and then to Si - III (bc8) [93]. This multiple transformation can influence the shape of the unloading data and thus the initial unloading stiffness, S, leading to a decrease of the Young's modulus (see Eq. (B.4)).

#### B.3.2 Nanoindentation of PECVD silicon oxide

The hardness and Young's modulus of the PECVD silicon oxide are shown in Figures B.13 and B.14. As explained in Section B.2.3, the difficulty of measuring mechanical properties of thin film is due to the influence of the substrate on the response of the nanoindenter. This substrate effect can lead to errors in the determination of the properties of the thin film. In the case of a  $SiO_2$  film, the influence of the Si substrate is relatively small because of the similarities in the mechanical properties of Si and  $SiO_2$ . However, the indentation depth was limited to half the film thickness which is equal to 1  $\mu$ m in our case. The hardness of the PECVD silicon oxide is about 9-10 GPa (see Figure B.13) except for the first three results that show a lower hardness for smallest indentation depth. These "outside-the-range" results can be explained by the difficulty of performing correct measurement and treating them at this relatively small depth.



Figure B.13 : Evolution of the hardness, H, of a 1  $\mu m$  thick PECVD SiO<sub>2</sub> layer with indentation depth.

The values of the Young's modulus of PECVD silicon dioxide obtained by nanoindentation range from 75 GPa at low indentation depth to 110 GPa at higher indentation depth. Even if the true value of the Young's modulus of silicon dioxide lies in this range of values, this large variation indicates the difficulty of determining the properties of thin films on substrate with the nanoindentation technique.



Figure B.14 : Evolution of the Young's modulus, E, of a 1  $\mu$ m thick PECVD SiO<sub>2</sub> layer with indentation depth.

#### B.3.3 Nanoindentation of thermal silicon oxide

The hardness and Young's modulus of the thermal silicon oxide are shown in Figures B.15 and B.16. The value of the Young's modulus is equal to 75 GPa and is in good agreement with values coming from literature [24]. The value of hardness is also a typical value for a silicon oxide.



**Figure B.15 :** Evolution of the hardness of a 1  $\mu$ m thick thermal SiO<sub>2</sub> layer with the indentation depth.



Figure B.16 : Evolution of the Young's modulus of a 1  $\mu$ m thick thermal SiO<sub>2</sub> layer with the indentation depth.

# B.3.4 Nanoindentation of aluminum thin layer deposited by evaporation

The Young's modulus of aluminum thin films obtained by an evaporation process is shown in Figure B.17.



Figure B.17 : Evolution of the Young's modulus of a 2  $\mu$ m thick Al layer deposited by evaporation with the indentation depth.

The Young's modulus of aluminum increases from 80 to 100 GPa. These values are higher than typical Young's modulus values (around 70 GPa). The overestimation of the Young's modulus is due to the wrong determination of the contact area when using the Oliver and Pharr (O&P) method. Indeed, the O&P method does not take into account pile-up effects appearing during the indentation of soft metals. The contact area, A, determined by the O&P method is thus underestimated leading to an overestimation of Young's modulus and hardness (see Eq. (B.14) and Eq. (B.15)).

However, it is possible to correct the hardness value by considering that the Young's modulus has a constant value equal to 70 GPa in the case of aluminum thin films. By rearranging Eq. (B.14) and Eq. (B.15), we obtain that:

$$H = \frac{4\beta^2 P_{max} E_r^2}{\pi S^2}.$$
 (B.18)

The determination of the hardness considering that the Young's modulus of thin film is constant is called the  $E_{constant}$  method in the rest of the document. The evolution of the hardness obtained with the two different methods (O&P and  $E_{constant}$ ) is plotted in Figure B.18. The O&P method gives a quite constant hardness value whereas the hardness obtained with the  $E_{constant}$  method decreases and reaches a plateau value of approximately 300 MPa. This decrease of hardness with the indentation depth is typical of soft metals and is explained in details in [94].



Figure B.18 : Evolution of the hardness of 2  $\mu$ m thick Al layer deposited by evaporation with the indentation depth.

The hardness of the aluminum is quite low indicating a low value of the yield stress. According to [26], the value of the yield stress can be obtained by dividing the hardness by a factor 3 in the case of soft materials. The value of the yield stress is thus:

$$\sigma_y \approx \frac{H}{3} = \frac{300}{3} = 100 \text{ MPa.}$$
 (B.19)

This value of 100 MPa obtained for the yield stress is expected for pure aluminum thin films [23].

### B.3.5 Nanoindentation of aluminum thin layer deposited by sputtering

Aluminum thin films deposited by sputtering are composed of 99 % of aluminum and 1 % of silicon. The evolution of the Young's modulus with the indentation depth is depicted in Figure B.19.



**Figure B.19 :** Evolution of the Young's modulus of 2  $\mu m$  thick Al layer deposited by sputtering with the indentation depth.

As in the case of aluminum deposited by evaporation, the Young's modulus of aluminum thin film (determined according to the O&P method) increases with indentation depth. The indentation depth being below the 50%, the substrate does not influence the determination of the Young's modulus. The wrong determination of the contact area is thus responsible of this increase of the Young's modulus. The evolution of the hardness with indentation depth is shown in Figure B.20.


**Figure B.20** : Evolution of the hardness of  $2 \ \mu m$  thick Al layer deposited by sputtering with the indentation depth.

The application of the  $E_{constant}$  method showed that the hardness of aluminum thin films deposited by sputtering decreases with the indentation depth [94] to reach a constant value equal to 550-600 MPa. Using the relationship already used for aluminum deposited by an evaporation process, we obtain

$$\sigma_y \approx \frac{H}{3} = \frac{600}{3} = 200 \text{ MPa.}$$
 (B.20)

The small amount of silicon in the aluminum layer increases the yield strength by a factor of two. It is then possible to obtain a relatively wide range in yield strength depending on the process (and particularly the source) used to deposit the aluminum thin layer. For assemblies described in Chapter 6, this controlled variation of the yield strength can be used to optimize the property of the ductile interlayer and thus to optimize the global toughness of such an assembly.

Appendix	С
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# Determination of the elastic strain energy stored in a bended beam

Let us consider the configuration of Obreimoff's experiment, shown in Figure C.1.



Figure C.1 : Obreimoff's experiment

The elastic strain energy in the deformed upper cantilever can be determined as follows.

The strain energy per unit of volume is given by

$$w_e = \frac{1}{2} \epsilon_{ij} \sigma_{ij}$$

Using Hooke's law,

$$\epsilon_{ij} = \left(\frac{1+\nu}{E}\right)\sigma_{ij} - \frac{\nu}{E}\sigma_{mm}\delta_{ij}$$

it is possible to write that:

$$w_e = \left(\frac{1+\nu}{2E}\right)\sigma_{ij}\sigma_{ij} - \frac{\nu}{2E}(\sigma_{mm})^2$$

where  $\nu$  and E are the Poisson ratio and Young's modulus of the beam. In our case, the dominant stresses are  $\sigma_{xx}$  and  $\sigma_{xy}$  and all other stresses can be neglected. Consequently, the strain energy simplifies into:

$$w_e = \frac{\sigma_{xx}^2}{2E} + \frac{\sigma_{xy}^2}{2\mu}$$

where  $\mu$  is the shear modulus. It is convenient to express the stresses as a function of the internal loads M(x), N(x) and Q(x) where M(x) is the internal moment, N(x) the internal normal force and Q(x) the internal shear force occurring in the beam when external loads are applied [95]. From beam theory, the stress  $\sigma_{xx}$  writes:

$$\sigma_{xx} = \frac{M(x)}{I}y + \frac{N(x)}{A}$$

where I is the inertia and A the area the beam with the following expressions for a rectangular beam:

$$A = wh$$
 and  $I = \frac{wh^3}{12}$ 

with w is the width of the beam (out-of-plane in Figure C.1) and h is the thickness of the beam. By definition of Q(x), the average value of  $\sigma_{xy}$  over a cross section is

$$\overline{\sigma}_{xy} = \frac{Q(x)}{A}.$$

It is then possible to determine the value of the shear stress  $\sigma_{xy}$  (see [95]) as:

$$\sigma_{xy} = \frac{6Q(x)}{A} \left(\frac{1}{4} - \frac{y^2}{h^2}\right).$$

We can now rewrite the strain energy per unit of volume:

$$w_e = \frac{1}{2E} \left[ \frac{M(x)}{I} y + \frac{N(x)}{A} \right]^2 + \frac{1}{2\mu} \left[ \frac{6Q(x)}{A} \left( \frac{1}{4} - \frac{y^2}{h^2} \right) \right]^2$$

The strain energy of the entire beam is obtained by the integration of  $w_e$  over the volume:

$$U_E = \int_{\Omega} w_e \ dx dy dz$$

which leads to:

$$U_E = \int_L \frac{M^2(x)}{2EI^2} dx \int_S y^2 dy dz + \int_L \frac{N^2(x)}{2EA^2} \int_S dy dz + \int_L \frac{M(x)N(x)}{EIA} dx \int_S y \, dy dz + \frac{Q^2(x)}{2\mu A_1} dx$$

where  $A_1$  is the reduced shear area (see [95] for more explanation on the reduced shear area). Considering that:

$$\int_{S} y^{2} dy dz = I,$$
$$\int_{S} dy dz = A,$$
$$\int_{S} y dy dz = 0,$$

it is then possible to rewrite the strain elastic energy as:

$$U_E = \int_L \left[ \frac{M^2(x)}{2EI} + \frac{N^2(x)}{2EA} + \frac{Q^2(x)}{2\mu A_1} \right] dx.$$

The contribution of the shear forces Q(x) and the normal forces N(x) can be neglected. Then, the elastic strain energy reduces to

$$U_E = \int_L \left[\frac{M^2(x)}{2EI}\right] dx.$$

The moment M(x) for the configuration of Figure C.1 is equal to:

$$M(x) = \frac{Ewh^3d}{8a^3}(x-a)$$

and the elastic strain energy  $U_E$  is then equal to:

$$U_E = \frac{Ewh^3d^2}{32a^3}$$
 [J].

Appendix 1	D
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## Determination of the error on the crack length using IR radiation

The minimum height of the air gap which can be detected using IR radiation is equal to 0.275  $\mu$ m [1]. Knowing the thickness of the wedge, it is possible to determine the error made using IR radiation for determining the crack length. Schematic of the problem is presented in Figure D.1.



Figure D.1 : Schematic of the wedge test showing the error made by using IR radiation to detect the crack front. The parameters  $\Delta a$  and e were magnified to make the picture more readable.

On Figure D.1, d/2 is half of the thickness of the wedge, a is the crack length measured by IR,  $\Delta a$  is the error made by using IR radiation and e is the minimum gap height which can be detected by IR radiation.

Let us use the notations  $a=a_{measured}$  and  $a + \Delta a=a_{real}$ .

The bending of the wafer can be described by the relationship derived from simple

beam theory:

$$y = \frac{d}{4} \left( 2 - 3\frac{x}{a_{real}} + \left(\frac{x}{a_{real}}\right)^3 \right).$$
(D.1)

The point  $(a_{measured}, e)$  should verify the relationship (D.1). So, we have that:

$$e = \frac{d}{4} \left( 2 - 3 \frac{a_{measured}}{a_{real}} + \left( \frac{a_{measured}}{a_{real}} \right)^3 \right).$$
(D.2)

The minimum value of e is equal to  $0.1375\mu$ m and the value of d, the thickness of the wedge, is equal to  $100\mu$ m. So, it is possible to determine the value of the ratio  $a_{measured}/a_{real}$  by solving Eq. (D.2):

$$\frac{a_{measured}}{a_{real}} = 0.95687.$$

The error made on the crack length by the use of IR radiation is then:

$$error(a) = \frac{a_{real} - a_{measured}}{a_{real}} \times 100 \approx 4.3\%.$$

It is then possible to determine the error made on the toughness remembering that:

$$G_c = \frac{K}{a^4}$$

where K is a constant depending on geometrical and material properties. We have then that:

$$error(G_c) = \frac{G_c^{real} - G_c^{measured}}{G_c^{real}} \times 100 = \frac{\frac{K}{a_{real}^4} - \frac{K}{a_{measured}^4}}{\frac{K}{a_{real}^4}} \times 100 \approx -19.2\%.$$

So, due to the limitation of IR radiation, the measured toughness overestimates the real toughness by around 19 %. Using a wedge of thickness equal to  $230\mu$ m, the error on a is equal to  $\approx 2.8\%$  and the error on  $G_c$  is equal to  $\approx -12.2\%$ .

Appendix E

### List of publications

#### Papers in Journals

Y. Bertholet, F. Iker, J.-P. Raskin and T. Pardoen, "Steady-state measurement and modelling of wafer bonding failure resistance", Sensors and Actuators A: Physical, Elsevier, vol. 110, pp. 157-163, January 2004.

B. Olbrechts, X.X. Zhang, Y. Bertholet, T. Pardoen and J.-P. Raskin, "Effect of interfacial SiO2 thickness for low temperature O2 plasma activated wafer bonding", Microsystem Technologies - Micro-And-NanoSystems. Information Storage and Processing Systems, vol. 12(5), pp. 383-390, 2006.

Y. Bertholet, B. Olbrechts, J.-P. Raskin and T. Pardoen, "Molecular bonding aided by dissipative interlayers", Acta Materialia, in press.

#### Papers in Conference Proceedings

Y. Bertholet, F. Iker, J.-P. Raskin and T. Pardoen, "Steady-state measurement and modelling of wafer bonding failure resistance", 16th European Conference on Solid-State Transducers, Czech Technical University, Prague, Czech Republic, Sept. 15-18, 2002, pp. 294-297.

Y. Bertholet, F. Iker, J.-P. Raskin and T. Pardoen, "Steady-state measurement and modelling of wafer bonding failure resistance", Workshop on Wafer Bonding, Barcelona, Spain, Sept. 30 - Oct. 1, 2002, paper 19, 1 page. Y. Bertholet, X. X. Zhang, J.-P. Raskin and T. Pardoen, "Steady-state measurement of fracture energy in wafer bonding", of the 27th Annual Meeting of The Adhesion Society - From Molecules and Mechanics to Optimization and Design of Adhesives Joints (M.K. Chaudhury ed., The Adhesion Society Inc.), Wilmington, North Carolina, U.S.A., February 15-18, 2004, pp. 438-440.

Y. Bertholet, F. Iker, X. X. Zhang, J.-P. Raskin and T. Pardoen, "Fracture resistance of interfaces in bonded silicon wafers", 15th European Conference of Fracture, Stockholm, Sweden, August 11-13, 2004, paper ECF15, 9 pages.

B. Olbrechts, Y. Bertholet, T. Pardoen and J.-P. Raskin, "Direct wafer bonding issues: surface activation, high and low temperature annealing and insertion of a ductile layer for absorbing constraints", Workshop on Wafer Bonding for MEMS Technologies, Halle, Germany, October 11-12, 2004, pp. 25-26.

Y. Bertholet, J.-P. Raskin and T. Pardoen, "Cohesive zone-based modeling Si-Si and SiO2-SiO2 interfaces in a presence of a ductile layer", 11<sup>th</sup> International Conference on Fracture, Turin, Italy, March 20-25, 2005, pp. 5083-5087.

Y. Bertholet, J.-P. Raskin and T. Pardoen, "Influence of a ductile interlayer on the toughness of hydrophilic wafer bonding", Eighth Int. Symp. on Semiconductor Wafer Bonding; science, technology and applications, 205rd Meeting of the Electrochemical Society, Quebec City, Canada, May 15-20, 2005, in Semiconductor Wafer Bonding VIII: Science, Technology, and Applications edited by K.D. Hobart, S. Bengtsson, H. Baumgart, T. Suga, C.E. hunt, published by The Electrochemical society, Inc., 65 South Main Street, Pennington, New-Jersey 08534-2839, USA.

## Bibliography

- Q.-Y. Tong and U. Gösele. Semiconductor Wafer Bonding : Science and Technology. Wiley, New York, 1999.
- [2] J. Haisma. *Phillips J. Res.*, 49, pp. 165, 1995.
- [3] A. Plössl and G Kräuter. Materials Science and Engineering, R(25), pp. 1-88, 1999.
- [4] B. Anglicus. De genuinus rerum coelestium, terrestrium at inferarum proprietatibus rerum. Book 16, Chap. 4, pp. 718, 1601.
- [5] G. Galilei. Discorsi e dimostrazioni matematiche, intorno a due nuove scienze (giornata prima). 1638.
- [6] J.T. Desaguliers. Philos. Trans. R. Soc. Lond., 33, pp. 345, 1725.
- [7] D. Dowson. History of Tribology. Longman, London, 1979.
- [8] J.W. Obreimoff. Proc. Roy. Soc. Lond., A127, pp. 290, 1930.
- [9] Lord Rayleigh. Proc. Phys. Soc., A 156, pp. 326, 1936.
- [10] G.A. Antypas and J. Edgecumbe. Appl. Phys. Lett., 26, pp. 371, 1974.
- [11] M. Shimbo, K. Furukawa, K. Fukuda, and K. Tanzawa. J. Appl. Phys., 60, pp. 2987, 1986.

- [12] J.B. Lasky, S.R. Stiffler, F.R. White, and J.R. Abernathy. Silicon-on-insulator (soi) by bonding and etch-back. In *Proceedings of the IEEE International Electronic Device Meeting*, Piscataway, NJ, 1985.
- [13] J.B. Lasky. Appl. Phys. Lett., 48, pp. 78, 1986.
- [14] A.P. London, A.A. Ayon, A.H. Epstein, S.M. Spearing, T. Harrison, Y. Peles, and J.L. Kerrebrock. *Sensors and Actuators*, A 92, pp. 351, 2001.
- [15] MST News, International Newsletter on MICROSYSTEMS and MEMS, 2/02, April 2002.
- [16] http://www.nexus mems.com.
- [17] MST News, International Newsletter on MICROSYSTEMS and MEMS, 4/02, September 2002.
- [18] I. De Wolf. Reliability issues in mems. MicroElectroMechanical Systems : from microfabrication techniques to smart microsystems, May 13-15, 2002, Louvain-la-Neuve.
- [19] J. Bardeen and W.H. Brittain. Phys. Rev., 74, pp. 230, 1948.
- [20] F. Delannay and T. Pardoen. Introduction to the Deformation and Fracture of Materials. Cours MAPR 2481, Université catholique de Louvain, Edition 2002.
- [21] http://www.kaajakari.net.
- [22] J. Dolbow and M. Gosz. Mechanics of Materials, 23, pp. 311, 1996.
- [23] L.B. Freund and S. Suresh. Thin Film Materials : Stress, Defect Formation and Surface Evolution. Cambridge University Press, 2003.
- [24] http://www.memsnet.org.
- [25] F. Iker. 3-D self-assembled microsytems in thin film SOI. PhD thesis, Université catholique de Louvain, FSA/ELEC/EMIC, 2006.
- [26] X. Chen anf J.J. Vlassak. A numerical study on the measurement of thin film mechanical properties by means of nanoindentation. J. Mater. Res., 16 (10), pp. 2974-2982, 2001.

- [27] W. Kern and D.A. Puotinen. RCA Rev., 31, pp. 186, 1970.
- [28] G. Kissinger and W. Kissinger. Sensors and Actuators, A36, pp. 143, 1993.
- [29] H. Moriceau, B. Bataillou, C. Morales, A.M. Cartier, and F. Rieutord. In Semiconductor Wafer Bonding : Science, Technology and Applications VII, ECS PV 03-19, 2003.
- [30] A. Weinert, P. Amirfeiz, and S. Bengtsson. In Semiconductor Wafer Bonding : Science, Technology and Applications VI, ECS PV 01-27, 2001.
- [31] B. Lawn. Fracture of Brittle Solids 2<sup>nd</sup> edition. Cambridge University Press, 1993.
- [32] Z. Suo. Reliability of interconnect structures. Private Communication.
- [33] C.E. Inglis. Trans. Inst. Nav. Archit., 55, pp. 219, 1913.
- [34] Z. Suo and J.W. Huthinson. Advances in Applied Mechanics, 29, pp. 63, 1992.
- [35] A. Needleman. J. Mech. Phys. Solids, 38(3), pp. 289, 1990.
- [36] A.G. Evans, J.W. Hutchinson, and Y. Wei. Acta Mater., 47, pp. 4093, 1999.
- [37] Y. Bertholet, F. Iker, J.P. Raskin, and T. Pardoen. Sensors and Actuators, A 110, pp. 157, 2004.
- [38] W.P. Maszara, G. Goetz, A. Caviglia, and J.B. McKitterick. J. Appl. Phys., 64(10), pp. 4943, 1988.
- [39] T.S. Moss. *Photoconductivity in the elements*. Butterworths Scientific Publications, London, 1952.
- [40] T. Piotrowski and W. Jung. Thin Solid Films, 364, pp. 274, 2000.
- [41] Z. Liu and D.L. DeVoe. Robotics and Computer Integrated Manufacturing, 17, pp. 131, 2001.
- [42] M.W. Lane, J.M. Snodgrass, and R.H. Dauskardt. *Microelectronics Reliability*, 41, pp. 1615, 2001.

- [43] M. Legros. Encapsulation de microdispositifs par wafer bonding. Master's thesis, Université catholique de Louvain, FSA/ELEC/EMIC, 2005.
- [44] A.G. Evans, J.W. Hutchinson, and Y. Wei. Acta Mater., 45, pp. 4093-4113, 1999.
- [45] J.E. Raynolds, J.E. Smith, G.L. Zhao, and D.J. Srolovitz. Phys. Rev., B53, 1996.
- [46] R. Stengl, K.Mitani, V. Leman, and U. Gösele. In Proc. IEEE SOS/SOI Tech. Conf., pp. 123, 1989.
- [47] K. Mitani, V. Lehmann, R. Stengl, D. Feijoo, U. Gosële, and H.Z. Massoud. Jpn. J. Appl. Phys., 30, pp. 615, 1991.
- [48] R.H. Esser, K.D. Hobart, and F.J. Kub. Journal of the Electrochemical Society, 150(3) G, pp. 228, 2003.
- [49] Q.Y. Tong, G. Kaido, L. Tong, M. Reiche, F. Shi, J. Steinkrichner, T.Y. Tan, and U. Gösele. J. Electrochem. Soc., 142 L, pp. 201, 1995.
- [50] M. Reiche, I. Radu, M. Gabriel, M. Zoberbier, S. Hansen, and M. Eichler. In Semiconductor Wafer Bonding VIII : Science, Technolgy and Applications, pages 326–337. The Electrochemical Society, 2005.
- [51] J.J. Kim, H.H. Park, and S.H. Hyun. Thin Solid Films, 384, pp. 236, 2001.
- [52] H. Moriceau, F. Rieutord, C. Morales, S. Sartori, and A.M. Charvet. In Semiconductor Wafer Bonding VIII : Science, Technolgy and Applications, pages 34–49. The Electrochemical Society, 2005.
- [53] X.X. Zhang and J.-P. Raskin. Electrochemical and Solid-State Letters, 7(8), 2004.
- [54] X.X. Zhang and J.-P. Raskin. Journal of the Electrochemical Society, 151(9), 2004.
- [55] B. Olbrechts, X. X. Zhang, Y. Bertholet, T. Pardoen, and J. P. Raskin. Microsystem Technologies - Micro-And-NanoSystems. Information Storage and Processing Systems, 12(5), pp. 383, 2006.

- [56] S. M. Wiederhorn. J. Amer. Ceram. Soc., 50, 1967.
- [57] S. M. Wiederhorn. J. Amer. Ceram. Soc., 53, 1970.
- [58] R. F. Cook and E.G. Liniger. Am. Ceram. Soc., 1992.
- [59] T. A. Michalske and S. W. Freiman. *Nature*, 295, 1981.
- [60] T. Michalske and B. Bunker. J. Amer. Ceram. Soc., 70 (10), pp. 780, 1987.
- [61] S.M. Wiederhorn, S.W. Freiman, E.R. Fuller JR, and C.J. Simmons. *Journal of Materials Science*, 17, pp. 3460, 1982.
- [62] K.T. Turner, M.D. Thouless, and S.M. Spearing. Mechanics of wafer bonding: effect of clamping. *Journal of Applied Physics*, 95(1), pp349-355, 2004.
- [63] T. Abe and J.H. Matlock. Solid State Technol., 39, 1990.
- [64] A. Folta, F. Raley, and E.W. Hee. In *IEEE Solid-State Sensor and Actuator Workshop*, Hilton Head, South Carolina, 1992.
- [65] K.T. Turner and S.M. Spearing. Journal of Applied Physics, 92(12), pp. 7658, 2002.
- [66] V. Tvergaard and J.W. Hutchinson. Journal of the Mechanics and Physics of Solids, 40, pp. 1337, 1992.
- [67] V. Tvergaard and J.W. Hutchinson. *Philosophical Magazine*, A 70, pp. 641, 1994.
- [68] R.H. Dean and J.W. Hutchinson. In 12th Conference, ASTM STP 700, American Society for Testing and Materials, pp. 383, 1980.
- [69] Y. Wei and J.W. Hutchinson. International Journal of Fracture, 93, pp. 315, 1998.
- [70] T. Ferracin, C. Landis, F. Delannay, and T. Pardoen. Int. J. Solids Struct., 40, pp. 2889, 2003.
- [71] C.M. Landis, T. Pardoen, and J.W. Hutchinson. *Mechanics of Materials*, 32, pp. 663, 2000.

- [72] T.Ferracin. Mechanics of Failure in Adhesively Bonded Steel Assemblies. PhD thesis, Université catholique de Louvain, FSA/MAPR/PCIM, 2003.
- [73] Y. Wei and J.W. Hutchinson. International Journal of Fracture, 95, pp. 1, 1999.
- [74] V. Tvergaard and J.W. Hutchinson. J. Mech. Phys. Solids, 41(6), pp. 1119, 1993.
- [75] I.N. Sneddon. Int. J. Engng. Sci., 3, pp. 47, 1967.
- [76] G.M. Pharr, W.C. Oliver, and F.R. Brotzen. J. Mater. Res., 7(3), pp. 613, 1992.
- [77] R.B. King. Int. J. Solids Structures, 23, pp. 1657, 1987.
- [78] J.J. Vlassak and W.D. Nix. J. Mech. Phys. Solids, 42, pp. 1223, 1994.
- [79] B.C. Hendricks. J. Mater. Res., 10, pp. 255, 1995.
- [80] P-L. Larson, A.E. Giannakopoulos, E. Soderlund, D.J. Rowcliffe, and R. Vestergaard. Int. J. Solids Structures, 33, pp. 221, 1996.
- [81] K.W. McElhaney, J.J. Vlassak, and W.D. Nix. Determination of indenter tip geometry and indentation contact area for depth-sensing indentation experiments. J.Mater. Res., 13(5), pp. 1300, 1998.
- [82] J. Alcala, A.C. Barone, and M. Anglada. Acta Mater., 48, pp. 3451, 2000.
- [83] A. Bolshakov and G.M. Pharr. J. Mater. Res., 13, pp. 1049, 1998.
- [84] Y.T. Cheng and C.M. Cheng. J Appl. Phys., 84, pp. 1284, 1998.
- [85] Y.T. Cheng and C.M. Cheng. Int. J. Solids Structures, 36, pp. 1231, 1999.
- [86] Y.T. Cheng and C.M. Cheng. Appl. Phys. Letters, 73, pp. 614, 1998.
- [87] R. Saha and W.D. Nix. Acta Mater., 50, pp. 23, 2002.
- [88] D. Beegan, S. Chowdhurry, and M.T. Laugier. Thin Solid Films, 466, pp. 167, 2004.
- [89] W.C. Oliver and G.M. Pharr. J. Mater. Res., 7(6), pp. 1564, 1992.

- [90] B. Bhushan and X. Li. J. Mater. Res, 12(1), pp. 54, 1997.
- [91] J.C. Jamieson. Science, 139, 1963.
- [92] K.J. Chang and M.L. Cohen. Phys. Rev. B, 31, pp. 7819, 1985.
- [93] J.Z. Hu, L.D. Merkle, C.S. Menoni, and I.L. Spain. Phys. Rev. B, 34, pp. 4679, 1986.
- [94] W.W. Gerberich, N.I. Tymiak, J.C. Grunlan, M.F. Horstemeyer, and M.I. Baskes. J. Appl. Mech., 69, pp. 443, 2002.
- [95] I. Doghri. Mechanics of Deformable Solids. Springer, 2000.