STEADY-STATE MEASUREMENT OF THE INTERFACE FRACTURE RESISTANCE IN WAFER BONDING

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Abstract

A steady-state wedge-opening test has been developed in order to measure the work of separation of bonded silicon wafers. Non-steady-state and steady-state measurements are compared. Significant influence of i) the surface treatment, ii) the annealing time and temperature and iii) the crack velocity on the toughness is observed and related to the interface chemistry. A methodology based on the insertion of a thin plastically deforming layer near the interface is proposed in order to extract the critical strength of the interface as well as to increase the fracture toughness.

Introduction

Wafer bonding is a key microfabrication technique that allows assembling MEMS parts and packaging of microsystems. Reliability of MEMS requires the development of characterization and modelling tools that allow the assessment of the integrity of bonded interfaces towards fracture and delamination. According to the recent literature [1,2], a minimum of two parameters is necessary to fully characterize the mechanical response of an interface : i) the work of separation which represents the energy per unit area needed for propagating a crack, noted G_c or Γ and ii) the maximum stress, σ_c, reached in front of the crack tip in the so called fracture process zone. In order to measure G_c and evaluate various bonding techniques and rate effects, a steady-state wedge-opening test has been developed following earlier works in the literature on the static wedge-opening method [3,4]. The second parameter, σ_c, could, in principle, be determined by simple uniform tensile or shear tests providing a value of these critical stress required for wafer separation. However, such measurements heavily depend on interface defects and do not deliver the intrinsic strength of the bond [3]. An indirect method has been developed in order to extract the critical stress. The idea is to insert a thin ductile interlayer near the interface in samples presenting identical interfaces. The amount of energy dissipated by plastic deformation of the interlayer is very much dependent on the critical strength of the interface. This interlayer method serves as a "strength sensor". A model has been developed in order to quantify the link between the global toughness taking into account for plastic dissipation and the strength.

   The samples preparation is presented in a first section. Then, the measurement is described followed by a presentation and discussion of the main experiment results (without

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ductile layer). The model used to extract the critical stress in the presence of a ductile interlayer is presented in the last section.

Experimental methods

Sample preparation. After standard cleaning (in H_2SO_4 and H_2O_2 mixture (5:2)), the wafers undergo a surface treatment, which is either (i) oxygen plasma, (ii) thermal oxidation, or (iii) Plasma Enhanced Chemical Vapor Deposition (PECVD) oxidation. (i) The surfaces of bonded wafers were activated by O_2-plasma for different exposure time periods. O_2-plasma activation parameters were an oxygen flow of 100 sccm, 90 mTorr chamber pressure and about 300 W bias. (ii) Thermal oxide is obtained by the transformation of Si in SiO_2 at high temperature (950 °C) in the presence of vapor water. The thickness of the thermal oxide is about 4000 Å. (iii) The PECVD oxide is obtained by a continuous circulation of gas (SiH_4) on top of the wafers. This gas react with O_2 to form SiO_2 on top of the wafers. The thickness of the PECVD oxide is about 2500 Å. Bonding experiments were carried out manually in cleanroom environment. Annealing steps at 150°C for 150 hours and 400 ^{0}C for 120 hours were performed in order to test the stability of the bonded interface quality after long thermal budget: 400 ^{0}C is usually the annealing temperature limit allowed for the fabrication and co-integration of MEMS with CMOS electronics in post-process. The chemical reaction occurring during annealing is:

$$Si\text{-}OH + Si\text{-}OH \leftrightarrow Si\text{-}O\text{-}Si + H_2O. \tag{1}$$
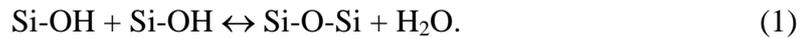
First, when the wafers are in contact, the link between the wafers is made only through hydrogen bonds. Then, during the annealing, the formation of covalent bonds Si-O-Si (see Eqn. 1) between the two wafers induces the cohesion between these wafers. Elimination of water occurs following two different ways: (i) water molecules diffuse outside the sample, (ii) water molecules diffuse through the oxide layer and react with silicon.

Infrared (IR) imaging system was used to inspect the bonding interface before and after annealing. Long rectangular specimens of 10 mm width are finally diced from the bonded wafers for bonding surface energy testing

Measurement techniques. The measurement set-up is represented in Fig. 1 (more details can be found in [5]). A razor blade of thickness h is inserted between the two wafers of thickness d at a constant rate using a home made set-up mounted on a universal mechanical testing machine. The crack length is continuously measured owing to the IR transparency of silicon and the contrast resulting from the presence of air between the wafers.
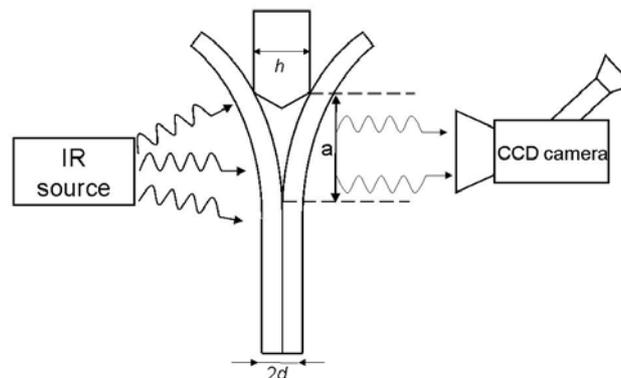
FIGURE 1: The wedge-opening test set up.

Simple beam theory leads to the following relationship between the crack length and the toughness for the configuration presented in Fig. 1 [1]:

$$\Gamma = G_c = \frac{3\,E\,h^2\,d^3}{16\,a^4} \tag{2}$$

where E is the Young's modulus of bonded wafers (taken to be equal to 200 GPa in our analysis), a is the crack length. This type of steady-state method allow to (i) generate accurate measurement of G_c, (ii) to detect heterogeneities in the bond quality from the side to the centre of the wafer-wafer structure, (iii) to address coupled strain rate/environmental effects by controlling the crack propagation rate. All tests were performed at room temperature ($\cong 21°C$), under atmospheric pressure and relative humidity of about 50%.

Results - *effect of the surface preparation and testing conditions on the interface toughness.*

Fig. 2 shows the variation of the crack length as a function of the displacement of the wedge for two annealed samples. Three zones can be distinguished. The 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 (Fig. 2) even if they undergo the same treatment. The second zone corresponds to the true steady state regime giving relevant crack length values. So, only crack length values be longing to this plateau can be considered in relation (2) to determine the toughness of the bonding. In the third zone, the crack tip stress field is starting to interact with the specimen end and therefore the value of the crack length is not representative anymore of the intrinsic toughness of the bonded wafers.
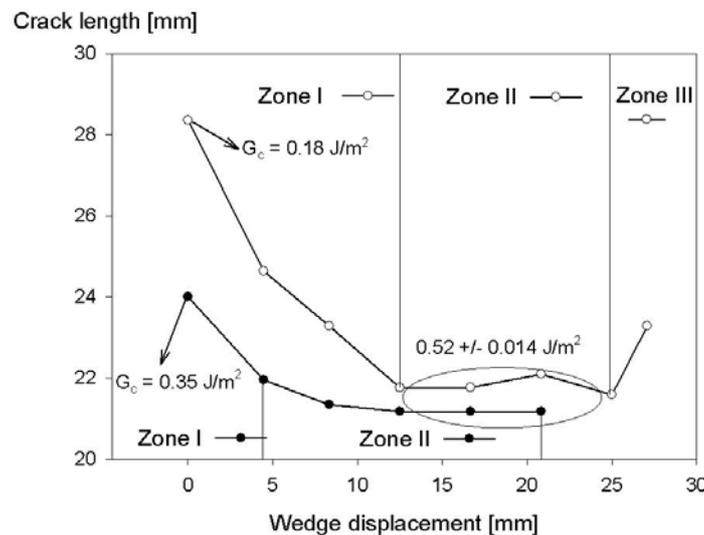
FIGURE 2: Evolution of the crack length as a function of the wedge displacement.

Influence of the wedge velocity. Fig 3 shows the variation of the fracture toughness as a function of crack velocity for annealed samples (no specific surface treatment except cleaning and 150 h annealing at 150°C). A significant increase of the bond interface fracture toughness with increasing crack velocity is observed. At high crack velocity, the toughness reaches a plateau value. Another plateau is also expected to appear at very low velocity [1,6].
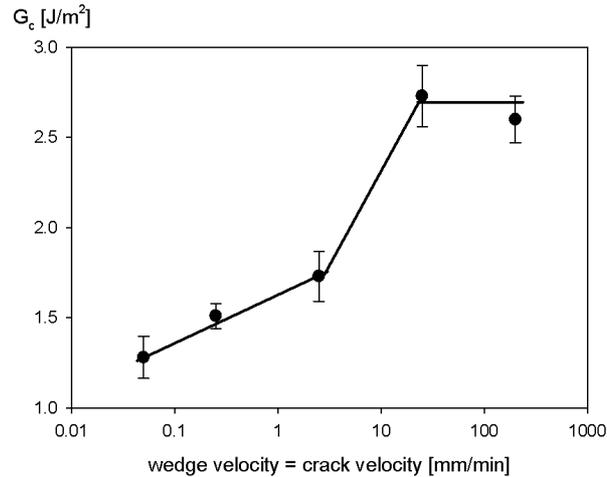
FIGURE 3: Variation of the fracture toughness as a function of crack velocity. Measurements are performed on samples prepared using the same procedure (no specific surface treatment – 150 h annealing at 150 °C).

Influence of the surface treatment. The fracture toughness measured for different specimens preparation procedures for a low (0.25 mm/min) and a high (25 mm/min) value of the crack velocity are gathered in Fig. 4. The PECVD oxide treatment does not appear because wafers did not bond at contact due to a roughness (RMS 1,5 nm) larger than the maximum value of roughness required for pre-bonding [1].
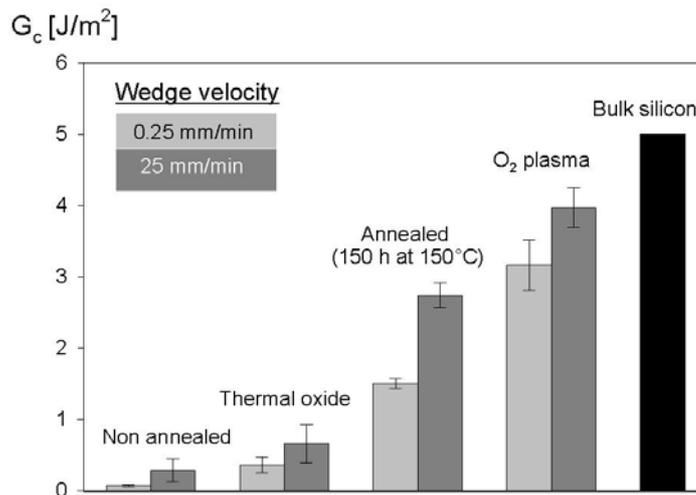
FIGURE 4: Fracture toughness at two different crack velocities for various surface treatments.

Plasma treated wafers give the highest fracture toughness. The toughness is then so large that some of specimen failed by the cracking of the silicon substrates. The method has thus bee selected for further researches and an effort has been placed on improving the reproducibility and homogeneity of the bonds. For instance, exposing wafers to a long plasma time increase the presence of voids at the interface as showed in Fig.5. In order to prevent annealing voids and meanwhile to increase the bonding toughness, an optimal O_2 plasma exposure time has been determined for Si-Si and SiO_2-SiO_2 bonding interfaces. The SiO_2 oxide is a thermal oxide with a thickness of about 1000Å. The variation of the bond

toughness as a function plasma treatment duration is shown on Fig. 6 for Si/Si and SiO$_2$/SiO$_2$ bonding wafers annealed at 400 °C for 120 hours. An optimal value for adhesion is reached for approximately 3s for Si/Si and 5s for SiO$_2$/SiO$_2$ O$_2$ plasma pretreatment prior to bonding.

(a)   (b)
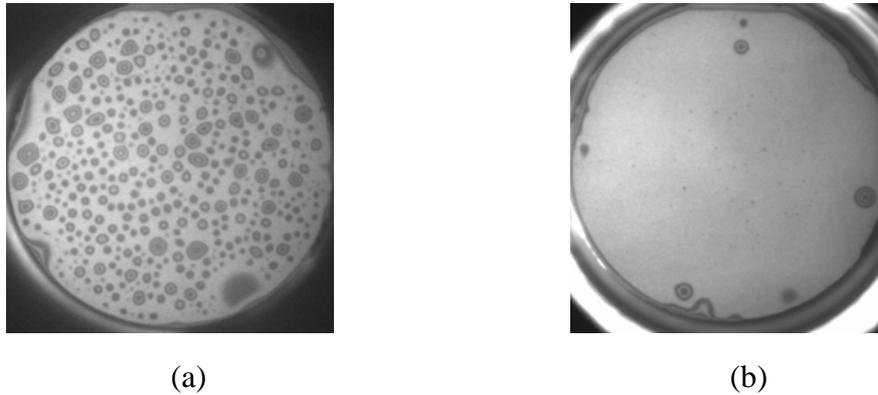
FIGURE 5: Infrared images of Si-Si SDB after annealing at 400^{0}C for 120 hours and for O$_2$ plasma pretreatment exposure time of (a) 20 s and (b) 3 s prior to bonding
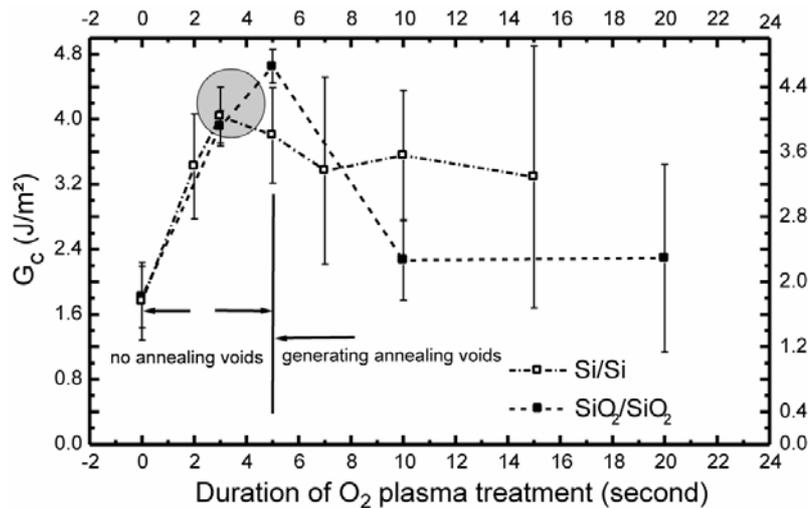
FIGURE. 6. Toughness as a function of plasma exposure time for Si/Si and SiO$_2$ (1000Å)/SiO$_2$ (1000Å) wafer bonding, (same exposure time for each wafer of bonded pair).

Discussion of the experimental results

Measurement method. Fig. 2 shows that care should be taken when performing wedge opening tests. Indeed, small misalignment of the razor blade with respect to the crack plane leads to an effective opening that can be significantly larger than the blade thickness. The error can be very important as G_c is proportional to the square of this effective opening, h (2). Having the specimen mounted on a free rotation grip is essential to allow perfect alignment of the blade during testing. Continuous crack length measurement also contributes to evaluate more representative average rate dependent bond fracture toughness.

Influence of the surface treatment. The toughness obtained for the different surface treatments were expected considering the interfacial chemistry, see ref. [3]. In non-annealed

samples, only hydrogen bonds contribute to the adhesion of the two wafers leading to very small bond toughness. The poor results obtained for the oxidized wafers come from a too large roughness. The annealed samples show relatively good bond toughness due to the formation, during the annealing, of Si-O-Si bonds between the wafers [3]. The higher value of bond toughness obtained for O_2 plasma treated samples is due to the higher density of these Si-O-Si bridges. These bond values of the toughness almost reach the fracture toughness of bulk silicon.

Influence of the wedge velocity. In Fig. 3, the evolution of the bond fracture toughness with wedge velocity can be attributed to the interaction with the environment, essentially the moisture. At high velocity, air water molecules cannot be adsorbed at the silicon surface and react with the Si-O-Si bond to dissociate it into two Si-OH groups as explained in [1,3,6]. When the crack velocity decreases, bond rupture by chemical reaction and healing events take place. Toughness is then much smaller than at high crack velocity

Model for the plastic deformation on a thin ductile interlayer

The idea developed here is to keep exactly the same Si/Si and SiO_2/ SiO_2 interfaces but in the presence of a thin ductile layer inserted near the interface. These tests supplement regular tests performed on samples without ductile layers. Fig. 7 schematically summarizes the principle of the method by considering two interfaces with similar fracture energy but different strengths. In the first case (see Fig 7.a), the interface is characterized by a low strength. In the second case (see Fig 7.b), significant plastic dissipation occurs in the ductile layer, which can significantly increase the overall interface toughness, Γ, as evaluated from (2). The global toughness is equal to $\Gamma_p + \Gamma_0$ where Γ_0 is the work of interface separation and Γ_p is the plastic work per unit area dissipated in the ductile layer. The strength can be obtained from the results of the tests with and without the ductile interlayer following the method described hereafter. Introducing the thin ductile layer is also a way to shield the interface and increase the global work of fracture.

| (a) | (b) |
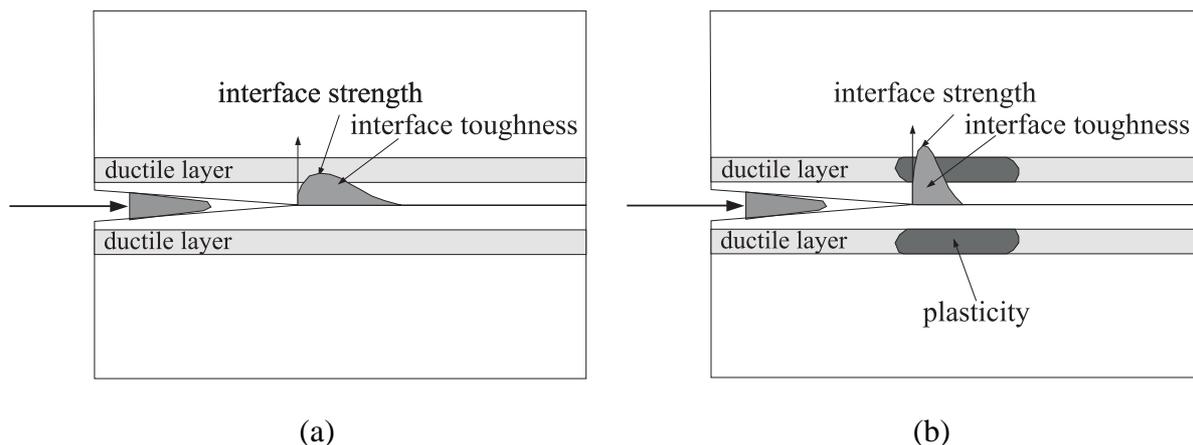
FIGURE 7: Schematic of the method used to probe the interface strength.

Following earlier efforts by Tvergaard and Hutchinson [2], the fracture process at the interface is simulated using as an interface traction-separation law which relates the normal stress σ to the normal displacements δ and which is characterized by the fracture energy of the interface noted Γ_0 and a peak stress noted σ_c. The traction-separation law proposed by

Tvergaard and Hutchinson [2] has been chosen for this investigation. The work of separation writes

$$\Gamma_0 = \sigma_c \delta_c \left(1 - \frac{\lambda_1 + \lambda_2}{2} \right) \tag{3}$$

where δ_c is the maximum separation, σ_c the peak stress and λ_1 and λ_2 are two shape parameters of the curve. As discussed by Tvergaard and Hutchinson [2], λ_1 and λ_2 are parameters of secondary importance, and they will be taken equal to 0.15 and 0.5 for the reminder of this study

The different layers involved in the simulation were modelled using isotropic linear elasticity and the isotropic J_2 flow theory for the ductile layer. The main output of the model allow the determination of the ratio Γ/Γ_0 , i.e. the overall toughness divided by the toughness of the interface. Γ can be determined using (2) if the wafer thickness is large enough. From dimensional analysis,

$$\frac{\Gamma}{\Gamma_0} \equiv F\left(\frac{\sigma_c}{\sigma_0}, \frac{E_p}{\sigma_0}, n, \frac{h_{el}}{h_p}, \frac{\Gamma_0}{\sigma_0 h_p}, \frac{E_p}{E}, v, v_p \right) \tag{4}$$

where σ_0 is the yield strength of the ductile layer, E_p is the Young's modulus of the ductile layer, n is the work-hardening coefficient of the ductile layer, v_p is the Poisson's ratio of the ductile layer, E and v are the Young's modulus and the Poisson's ratio of the two elastic layers surrounding the ductile one, h_p is the thickness of the ductile layer and h_{el} is the thickness of the thin elastic layer. An important length controlling the energy dissipation in the ductile layer is R_0 by

$$R_0 = \frac{E}{3\pi\left(1 - v^2\right)} \frac{\Gamma_0}{\sigma_0^2} \tag{5}$$

which represent the size of the plastic zone that would exist without the thin elastic layer if the ductile interlayer was very thick, see Wei and Hutchinson [7].

Numerical methods

A steady state finite element formulation for small strain-small rotation crack propagation problems was first applied by Dean and Hutchinson [8] and later implemented by several other authors [7]. The formulation consists of finding an equilibrium solution for the displacements based on a previous approximate distribution of plastic strains and then integrating the plasticity laws along streamlines to determine new approximations for stresses and plastic strains. This procedure is repeated until convergence is achieved. A small strain, large rotation formulation is used. More details about the formulation of the code can be found in [9]. Since the test is symmetrical, only half of the sandwich needs to be analysed. Plane strain conditions are assumed. The wedge is modelled with a fixed boundary condition at a normalised distance from the plane of symmetry.

One selected numerical result

The evolution of the Γ/Γ_0 ratio with σ_c/σ_0 for various R_0/h_{el} is shown in Fig. 8. The other parameters are taken constant: $E/\sigma_0 = 4000$, $n = 0.1$, $h_p/h_{el} = 5$. These are reasonable values for typical Al interlayer deposited on Si substrate. The influence of the ductile layer on the overall toughness is maximum for a ratio σ_c/σ_0 ranging from 6 to 7. The value of Γ/Γ_0 increases with increasing R_0/h_{el}, reaches a maximum value when it is equal to about 30 times the thickness of the thin elastic layer (see Fig. 8) and then decreases with further increase of R_0/h_{el}
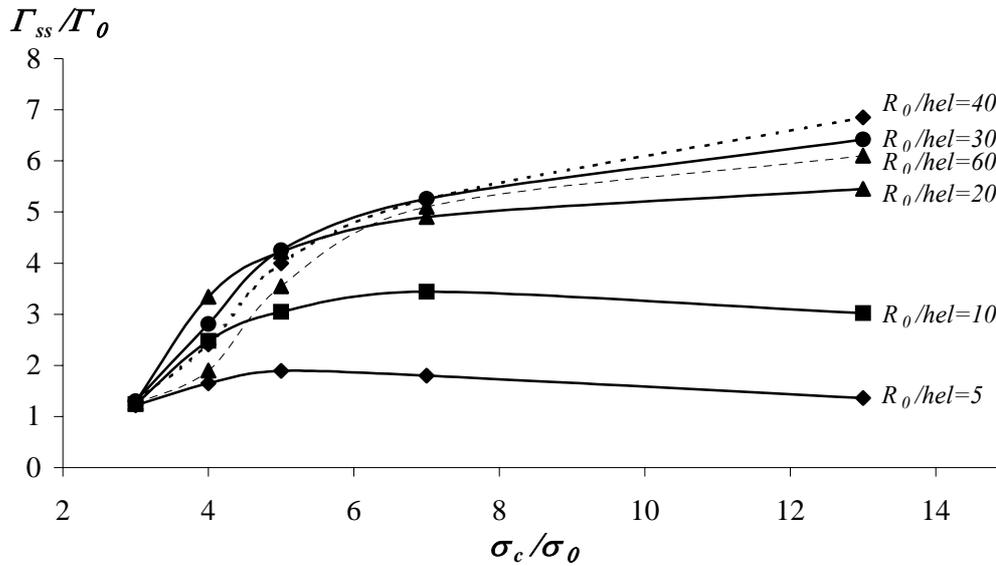
FIGURE 8: Influence of the critical stress of the interface on the global toughness for samples with a ductile layer.

The increase of Γ/Γ_0 with increasing σ_c/σ_0 at constant R_0 results from the increasing plastic dissipation in the ductile layer. Keeping R_0 constant means that Γ_0 is constant. Thus, when σ_c increases, the critical opening δ_c decreases proportionally and a point is sometimes attained where the decrease of δ_c becomes predominant in influencing the plastic dissipation. Increasing R_0 has the same effects as increasing σ_c. A maximum is attained because even if R_0 is increased, the increase of Γ_p is not high enough to keep the ratio $(\Gamma_p+\Gamma_0)/\Gamma_0$ increasing as Γ_0 increases proportional to R_0.

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