Indentation and Imprint Mapping for the Identification of Interface Properties in Film-Substrate Systems

M. Bocciarelli, G. Bolzon
Politecnico di Milano, Milano, Italy

The life-time of industrial products and the performance of materials for advanced mechanical applications are more and more often enhanced by coatings which, for instance, can act as thermal barriers in turbine engineering, constitute electrical connectors in silicon technology, improve wear and corrosion resistance in a variety of working tools [1]. Thermal, electrical and mechanical characteristics of coatings are of great interest for their application, but a further important macroscopic property to be estimated is adhesion across the interface region, strictly dependent on the deposition technique and on the mechanical/physical properties of the film and of its substrate, see e.g. [2].

Indentation tests are at present frequently employed for the identification of material parameters at different scales [3]. A recently proposed inverse analysis technique, combines the traditional indentation curves with the mapping of the residual deformations (imprint), thus providing experimental data apt to be used to identify material parameters in film-substrate systems in an almost no-destructive manner [4, 5]. In this methodology, the available experimental information deduced from indentation, performed on the external specimen surface, is combined with the simulation of the test and the material parameters, entering the numerical model, are estimated by minimizing the difference between experimental and their computed counterparts.

In the present communication the above mentioned inverse analysis technique is applied to estimate the material parameters governing the interface behaviour. Data about the applied load versus the penetration depth, usually returned by instrumented indentation are usually enough to discriminate situations where delamination does or does not occur, but does not return reliable estimates of the interfacial strength and toughness [4]. A novel set of experimental data, namely the displacement field measured in and around the residual imprint, is hence exploited in order to improve the performance of the identification methodology for the interface properties. The bulk material parameters and the friction coefficient between the indenter and the sample are supposed to be a priori known. Their values can be determined by the inverse analysis procedure proposed in [5], indenting the specimen at a penetration depth which does not induce delamination, an event usually clearly reflected by the indentation curves, and which can be further verified by impact-echo techniques or by mechanical impedance.

The sought material parameters are recovered through recursive calculations of the mechanical response of the film-substrate system, performed by the simulation of the test in finite strain regime. In these analyses, based on the finite element (FE) method, the interface response is interpreted through the cohesive model originally proposed for mode I fracture by Rose et al. [6] for metals and bimetallic interfaces and extended to two-dimensional situations in [7] and [8], based on the assumption of the existence of the following free energy density function:

$$\varphi(w) = e\sigma_c w_c \left[1 - \left(1 + \frac{w}{w_c}\right)\exp\left(-\frac{w}{w_c}\right)\right] \tag{1}$$

where e is the Neper number, σ_c is the maximum cohesive normal traction, w_c is a characteristic opening displacement and *w*, originally [6] corresponding to the opening displacement, is a scalar measure of the displacement jump vector across the interface, defined as follows:

$$w = \sqrt{w_n^2 + \beta w_s^2} \tag{2}$$

Parameter β assigns different weight to opening (w_n) and sliding (w_s) discontinuities. The total fracture energy dissipated in pure either opening or sliding mode is equal to $G_c = e\sigma_c w_c$.

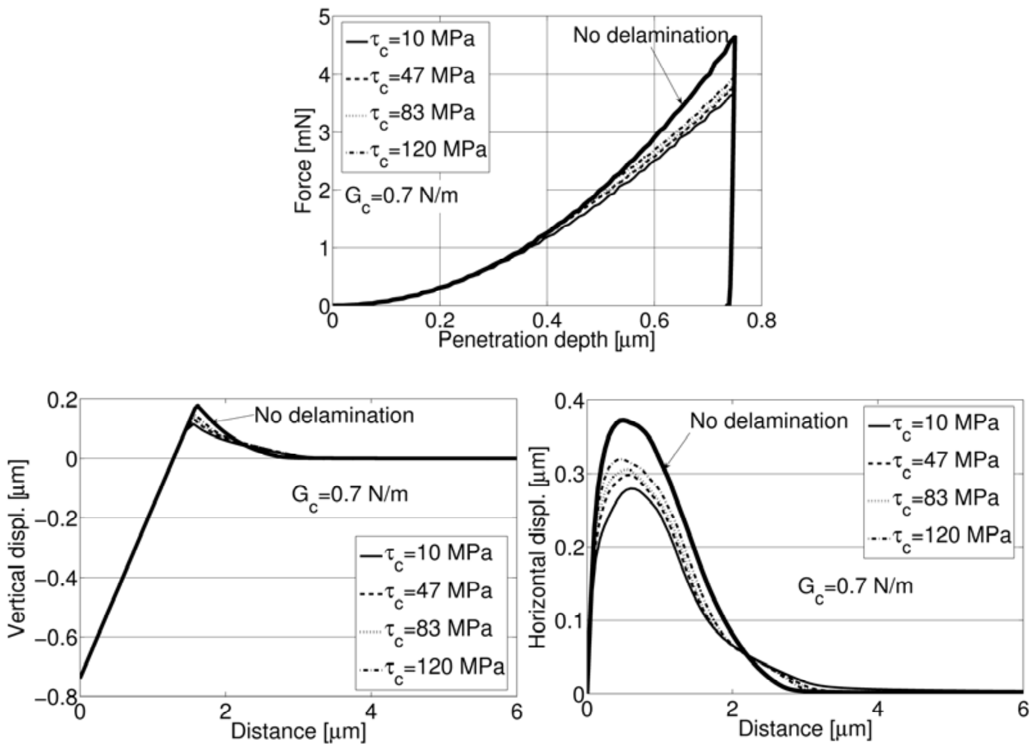

Fig. 1: Simulated response to conical indentation of a thin gold layer on a silicon substrate, in terms of: (a) reaction force versus penetration depth; (b) axis-symmetric profile of the residual imprint; (c) horizontal displacement component in and around the residual imprint.

Cohesive tractions under progressive fracture are defined through the derivatives of the free energy function defined in Eq.(1) with respect to the relative displacements w_n and w_s, while the irreversible behaviour of the interface is governed by the maximum effective displacement jump (2), which represents the only considered internal variable. Details can be found e.g. in [4].

Parameters σ_c, w_c and β (or the equivalent set: σ_c, $\tau_c = \beta\sigma_c$ and G_c) characterize the interface properties to be identified through the envisaged inverse analysis procedure. Input data are the results of the indentation test, see Fig. 1, in terms of: (a) the reaction force versus the penetration depth curves, usually returned by standard instrumentation; (b) the profile of the residual imprint, obtained through atomic force microscope, scanning interference microscopy or laser profilometer, depending on the imprint size [9,10]; (c) the distribution of the horizontal displacements on the flat surface outside the indented area, which can be accurately recovered by digital image correlation (DIC) techniques, see e.g. [11,12].

The value of the sought fracture parameters, here collected by vector $\mathbf{z}$, is then recovered through the minimization of the following objective function, which measures the discrepancy between experimental data and the result of the FE modelling of the laboratory (or in situ) test:

$$\omega(\mathbf{z}) = \sum_{i=1}^{M}\left(\frac{u_{ci}^{cur}(\mathbf{z}) - u_{mi}^{cur}}{u_{\max}^{cur}}\right)^2 + \sum_{j=1}^{N}\left(\frac{u_{Hcj}^{imp}(\mathbf{z}) - u_{Hmj}^{imp}}{u_{H\max}^{imp}}\right)^2 + \sum_{k=1}^{T}\left(\frac{u_{Vck}^{imp}(\mathbf{z}) - u_{Vmk}^{imp}}{u_{V\max}^{imp}}\right)^2 \quad (3)$$

where: indices m and c distinguish measured and computed (through the FE model) corresponding quantities; u^{cur} refer to measured/computed displacement along the indentation curve, under load control; u_H^{imp} and u_V^{imp} represent horizontal and vertical displacement components measured/computed around and along the residual imprint. A representative sample of all these displacements is input in the inverse analysis procedure; the total numbers of them (M, N and T in the definition of the discrepancy function ω) is selected on the basis of a reasonable compromise between accuracy and computing costs.

Merits and limitations of the proposed approach can be verified, first, by means of extensive numerical exercises based on pseudo-experimental data, i.e. data generated by the numerical model employed for the test simulation, to be input in the inverse analysis procedure in order to check if this is able to recover the parameters adopted to generate the pseudo-experimental data. Modelling errors are ruled out from this preliminary validation phase, while the significance of experimental noise on measurable quantities is addressed by introducing random errors in the computer generated data to be input in the inverse analysis. Some preliminary results relevant to strong interfaces, where shear delamination dominates without any apparent film buckling, are summarised in the following. The properties of the film-substrate system under consideration are typical of thin gold layer, 1 μm thick, deposited on silicon and subjected to conical indentation. In the simulations, gold is described by linear elasticity and von Mises perfect plasticity model, endowed by the following material constants: modulus of elasticity E_f = 80 GPa; Poisson's ratio ν_f = 0.4; yield stress σ_{0f} = 200 MPa. The silicon substrate is interpreted as linear elastic, characterized by the material parameters: E_s = 200 GPa; ν_s = 0.3. The conical tip is considered perfectly sharp with 60° (semi-)opening angle. The contact interface between the indentation tool and the specimen is characterized by Coulomb friction without dilatancy; the friction coefficient has been set equal to 0.15. Some representative results are shown in Fig. 1.

Table 1 reports the results obtained in terms of identification error with respect to the expected value of the fracture parameters. Results are given in terms of mean value, standard deviation and identification error for four different combinations of interface parameters. Pseudo-experimental information is extracted from the indentation curve and from the vertical and horizontal components of the displacement field. Data are corrupted by random noise, uniformly distributed within the range ±2.5% of the exact value. Dimensions are in [MPa] for σ_c and τ_c, in [N/m] for G_c.

Results listed in Table 1 show that:

1. mode II resistance can always be identified with an error of the same order of magnitude of the added noise;

2. mode I resistance can be identified only if it is definitely larger than τ_c, such that film-substrate delamination occurs in mixed mode and the influence of the normal strength is appreciable; however, such parameter combination defines a quite undesirable situation for practical applications;

3. fracture energy can be always identified, even if with an error larger than that characterizing the identification of τ_c.

τ_c^{exp}	τ_c^{id}	$Err(\tau_c^{id})$	σ_c^{exp}	σ_c^{id}	$Err(\sigma_c^{id})$	G_c^{exp}	G_c^{id}	$Err(G_c^{id})$
30	30.18±1.04	4.07%	100	98.12±15.80	17.68%	0.544	0.549±0.041	8.46%
100	100.85±4.75	5.60%	30	30.18±1.15	4.43%	0.544	0.536±0.037	8.27%
30	29.86±0.74	2.93%	100	99.22±15.49	16.27%	1.359	1.352±0.058	4.78%
100	99.39±2.53	3.14%	30	30.12±1.65	5.90%	1.359	1.372±0.046	4.34%

Table 1. Results of the preliminary validation tests based on pseudo-experimental information.

A broader validation of the proposed procedure has been considered for the identification of τ_c and G_c only, in the most desirable situation of relatively high normal strength at the interface. A critical parametric study has been carried out, aimed at determining the minimum experimental data required for the present identification purposes, assuming the following possible sources of information: (i) the indentation curve only; (ii) the indentation curve and the horizontal displacement field; (iii) the indentation curve and the vertical displacement field; (iv) all available data, in terms of indentation curve and in-plane and out-of-plane displacement fields.

The results, in terms of identification errors, summarised in Table 2 refer to the assumed interface properties: G_c = 0.544N/m, τ_c = 80MPa and σ_c = 100MPa. The inverse problem of finding them back has been solved by considering 500 different noise extractions. Figures in Table 2 permit to draw the following further conclusions:

4. the only indentation curve seems not to provide adequate experimental information for the present identification purpose as 2.5% noise disturbance produce some 10% error on the identified parameters;

5. the additional use of the horizontal displacement components improves the accuracy of the estimates, keeping the identification error almost within the limit of the added noise;

6. the additional use of the vertical displacements does not improve the identification any further, circumstance which reflects the lower sensitivity of this experimental information with respect to the unknown properties;

7. the use of indentation curve and vertical displacement field only is not as efficient for the present identification purposes.

	case(i)		case(ii)		case(iii)		case(iv)	
Noise	$Err(\tau_c^{id})$	$Err(G_c^{id})$	$Err(\tau_c^{id})$	$Err(G_c^{id})$	$Err(\tau_c^{id})$	$Err(G_c^{id})$	$Err(\tau_c^{id})$	$Err(G_c^{id})$
2.5%	9.6%	10.7%	3.3%	3.4%	8.0%	8.6%	4.0%	3.4%
5.0%	15.0%	18.5%	9.9%	9.5%	12.9%	15.4%	8.7%	8.3%
7.5%	21.2%	25.5%	16.1%	14.8%	20.6%	22.9%	17.0%	15.1%
10.0%	27.9%	37.2%	22.7%	20.1%	27.8%	30.1%	22.5%	19.9%

Table 2. Results of the preliminary validation tests based on pseudo-experimental information.

The present results are quite encouraging in view of the envisaged possibility of identifying interface properties through the widely used indentation test, simply performed on the specimen surface. This positive perspective should however be corroborated by the results of parameter calibration exercises exploiting truly experimental data, which is the aim of ongoing research work.

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