

# USE OF MODELS TO PREDICT DEFORMATION DURING THE ELASTIC NANO-INDENTATION OF COATINGS

R A Hunt and L N McCartney

NPL Materials Centre  
National Physical Laboratory  
Teddington, Middlesex  
United Kingdom, TW11 0LW

## ABSTRACT

This report describes progress that has been made with the development of a methodology to estimate, from nano-indentation tests, the elastic properties of coatings whose thicknesses are in the micron range. The report summarises the results of new work that has been carried out, and intercomparison results that formed part of the international INDICOAT project.

The basis and performance of an NPL developed hybrid model for predicting the indentation behaviour for coated systems is described. The model uses analytical techniques supplemented with FEA analyses for flat circular punches. The hybrid approach was developed to avoid the problem of having to deal with the moving contact boundary during loading. The methodology enables the application of the analysis to any axisymmetric indenter shape, and leads to predictions of the effective indenter modulus that are independent of indenter shape and coating thickness.

For elastic axisymmetric deformations, the load-penetration curves and surface profiles predicted in the INDICOAT project, by FEA modelling, and various analytical approaches including the NPL hybrid model, are in agreement indicating that these models are consistent with one another. The Gao et al model developed for flat circular punches is, however, shown to be inaccurate when compared to FEA solutions.

The analysis of the elastic behaviour of a pre-dented coated sample (using a spherical indenter) has shown that the contact is not Hertzian implying that the stress concentration effect of the indent is having a significant effect on load-penetration behaviour. This indicates that the Oliver and Pharr analysis, conventionally used to extract the effective modulus from the unloading part of elastic-plastic load penetration curves, has no sound scientific basis.

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National Physical Laboratory  
Teddington, Middlesex, UK, TW11 0LW

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Approved on behalf of Managing Director, NPL, by Dr C Lea, Head of NPL Materials Centre

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## 1. Introduction

Coatings are generally applied to engineering components in order to improve their performance and sometimes their appearance. Examples include hard coatings to prevent the wear of the substrate, and thermal barrier coatings to enable metal components to operate safely in high temperature environments. For these examples, it is vital that the integrity of the coating is maintained throughout the normal service life of the engineering component. It is also vital that engineers have a proper understanding of the micro-mechanisms of coating deformation and failure. For example, the stresses that develop during the deposition of the coating and subsequent cooling can reach levels that, when added to the external stresses applied during service, may result in cracking and de-adhesion of the film. For cracking in layered materials, analytical models have been developed to predict the critical stress, crack spacing and progressive crack formation. Interfacial fracture energy has been associated with the de-adhesion process and attempts at modelling the critical stress for the de-adhesion of a thin film have been made. Steady state delamination has been analysed for conditions where yielding occurs in the film or in the substrate. In addition, spallation has been identified as a major mode of failure in thermal barrier coatings. If damage resistant coatings are to be designed using fracture mechanics methods then it is essential that their in-situ properties be known accurately. This is a difficult task when the coatings are very thin with thicknesses in the micron range. The principal objective of this report is to describe the results of an investigation to model the nano-indentation of coatings, and to assess methods of extracting coating properties from the load-penetration curves.

Another objective of this report is to summarise the results of an international modelling investigation into the axisymmetric elastic deformation of a coated sample that is loaded by means of nano-indentation. The primary objective is to investigate whether coating elastic properties (Young's modulus and Poisson's ratio) can reliably be estimated from the indentation response in the form of load-penetration curves. The report summarises both relevant results from the recently completed international INDICOAT project [1], and work carried out relating to the DTI CPM2.2 project. A summary of the results of modelling elastic-plastic deformations will be given in a future report.

Results have been obtained by using modelling methods to predict indentation behaviour (using analytical methods and finite element analysis (FEA)) in order to select the most reliable method(s) that can be used to help with the interpretation of indentation tests in the elastic regime of deformation. Analytical methods are candidates for becoming the basis of testing standards. However, the involved nature of FEA methodology means that it is unlikely that this technique will be required for the development of testing standards. The major role of FEA in the project is to provide a reliable and accurate yardstick against which the performance of semi-analytical and analytical methods can be measured. To achieve such comparisons it is essential that a set of test problems is formulated that can be solved by the various methodologies outlined above. It is important that each methodology adheres to the specified problem as closely as possible. Thermal residual stresses are to be ignored in the inter-comparison exercise. The following sections outline the approach and describe the results and conclusions.

## 2. Types of modelling considered

### Analytical models

Analytical models regard the linear elastic indentation of a coated substrate as a classical mechanics problem where it is vital that the laws of continuum mechanics are obeyed, such as the equilibrium equations, stress-strain relations and compatibility equations. In addition it is vital that solutions should obey the correct boundary and coating/substrate interface conditions characterising a perfectly bonded interface. This is especially important for indentation problems where the contact area varies with the applied load. Analytical models usually assume linear elastic behaviour, and axisymmetric stress and displacement fields, leading to non-linear load-deflection behaviour arising from the increase of contact area with load. Analytical methods may result in simple analytical formulae that have potential to be used to interpret the results of indentation experiments. Usually simple formulae will result only if some approximations are introduced when solving the classical elasticity equations. Analytical models that require numerical methods to derive predictions can, in principle, form part of a measurement standard provided that validated source code or executable code is available.

Three types of analytical model for coated systems have been considered as part of the INDICOAT project [1]. The first model [2] develops a methodology that is based on the assumption that the pressure distribution over the contact area is Hertzian in form. The second model [3] is based on the use of a Green's function technique where the loading is represented by a continuous distribution of point forces acting over the contact area. The function defining the distribution of point forces is the unknown function that is solved by integral equation techniques. The third model [4] developed at NPL is a hybrid technique that involves the use of analytical elasticity methods combined with simple FEA analysis for a flat-ended rigid cylindrical punch. From such solutions it is possible to predict the indentation behaviour of any axisymmetric rigid indenter subject to either friction-free contact or to perfect adhesion (the latter case has not been considered here). The attraction of the NPL approach is that there is no need to take account of varying contact area when carrying out the FEA for a rigid flat cylindrical punch, and that predictions can easily be made for any coating thickness between 0.2  $\mu\text{m}$  and 2  $\mu\text{m}$ , and for *any* axisymmetric indenter geometry. Significant refinements have been made to the hybrid model, leading to the analysis and results presented in Appendix 1.

### Finite element models

Finite element methods are thought to provide the most reliable and accurate predictions of the stress and displacement fields in the coating and substrate of an indented sample. The method is very likely to be the only technique that can adequately model non-linear deformations arising from localised plasticity that occurs during indentation. In this report, which is restricted to elastic deformations, the use of infinitesimal deformation theory was thought to be adequate.

As part of the INDICOAT project [1], three organisations (JRC (Italy), KTH (Sweden) and NPL (UK)) have carried out the FEA of the various coating problems. KTH have used the FEA system ANSYS to predict their results [5]. This FEA system does not have infinite

elements and steps were taken by KTH to ensure that their results were independent of the model size. Both JRC and NPL used ABAQUS, together with infinite elements, when carrying out FEA simulations of elastic indentation.

### 3. The hybrid model for a coated system

For the refined hybrid model developed by NPL (see Appendix 1 for details) any axially symmetric indenter is represented by a sequence of cylindrical punches as illustrated schematically in Fig. 1.

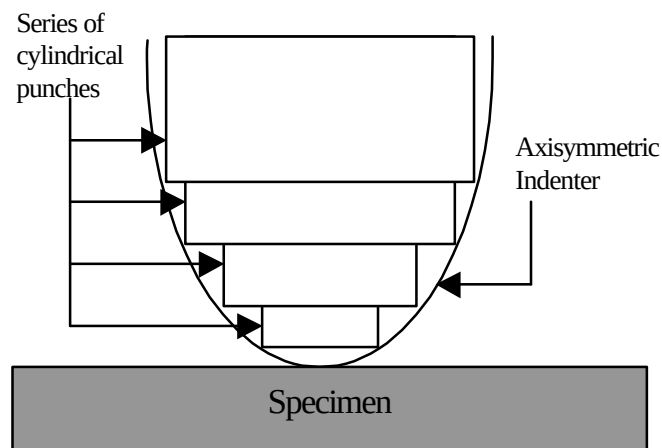


*Fig. 1: A schematic representation of the replacement of a single axisymmetric indenter with the superposition of a series of flat cylindrical punches.*

In the analysis, the limit is taken of the number of flat circular punches in the sequence tending to infinity (their thickness tending to zero) so that the approximate discrete representation of the axially symmetric indenter becomes exact. The required solutions have been obtained by a numerical technique. In this case the finite element method was used by applying flat cylindrical punches having a fixed radius to coatings having various thicknesses.

At first sight it would appear that a very large number of numerical solutions would be necessary to correspond to the infinite number of punches in the sequence representing the indenter. Fortunately it may be shown from dimensional considerations that the only distinct cases are distinguished by the value of the ratio of the coating thickness  $t_c$  and the radius of the contact area of the punch  $a$ . This means that the solutions for a given coating and substrate may be classified in terms of a single parameter  $t_c/a$  and this allows an interpolation scheme to be introduced which greatly reduces the number of finite element solutions necessary to achieve an acceptable accuracy.

The great advantage of the hybrid model is that it gives an integral representation of the solution for the load-penetration curve, the surface profile and the contact area. Once the solution is in this form it can be differentiated to estimate the effective modulus or manipulated to produce general results, which hold for any axially symmetric indenter. For the example of indentation into a homogeneous material (i.e. without a coating), it may be

shown that the effective modulus for an indentation test  $E_{IT} = E/(1-\nu^2)$  where  $E$  is Young's modulus and  $\nu$  is Poisson's ratio for the material. When indenting a coated sample the value of  $E_{IT}$  tends to the substrate modulus as the coating thickness  $t_c$  tends to zero and to the coating modulus as  $t_c$  tends to infinity. For other values of the coating thickness, the results lie on a single curve for any given combination of coating and substrate materials, when results are plotted as a function of  $t_c/a$  where  $a$  is the contact radius, regardless of the actual shape of the indenter. Thus, the *elastic*  $E_{IT}$  versus  $t_c/a$  curve, which may be determined experimentally from load-penetration curves derived from elastic indentation experiments, is predicted to have good prospects for producing accurate estimates of  $E_{IT}$  for both the coating and the substrate. It may be noted that, in order to ensure that the indentation experiments only involve elastic properties, stresses must be kept below the yield stress. This may be achieved by using relatively large radius spherical or flat circular indenters at low loads.

Unfortunately the analysis cannot predict separately the values of  $E$  and  $\nu$ , as only the combination  $E_{IT} = E/(1-\nu^2)$  can be predicted and measured. In order to achieve the required separation, a good analytic solution for the load-penetration curve for a cylindrical punch indenting a coated specimen needs to be found in a suitable form. In the light of the fact that this separation cannot yet be achieved for uncoated specimens it must be borne in mind that this will not be possible either for coated systems.

The performance of the hybrid model will now be tested by comparing its predictions with other methods including FEA, and with some experimental data.

#### 4. Material properties

The coating systems to be considered in the investigation are based on nominal values of coating and substrate properties, and they involve a hard coating of three nominal thicknesses applied to a soft substrate and vice versa.

##### Hard material

The properties of the selected hard material (similar to steels) are assumed to be linear elastic and isotropic, and are given by:

$$\text{Young's modulus} = 200 \text{ GPa}$$

$$\text{Poisson's ratio} = 0.3$$

##### Soft material

The isotropic properties of the selected soft material (similar to aluminium alloys) for elastic deformations are:

$$\text{Young's modulus} = 70 \text{ GPa}$$

$$\text{Poisson's ratio} = 0.33$$

## 5. Geometry

Predictions using axisymmetric models have been made for the indentation into coated systems for which the substrate material was assumed to be semi-infinite in extent. If only a finite volume is considered (as with the FEA package ANSYS that does not have infinite elements) then the size must be chosen to be large enough for the results to be independent of the substrate thickness and outer radius. The thicknesses of the coating were selected to be 0.2  $\mu\text{m}$ , 0.8  $\mu\text{m}$  and 2  $\mu\text{m}$ . The coated systems were indented by a rigid spherical indenter having a radius of 10  $\mu\text{m}$ . The direction of loading was taken as normal to the surface of the coating so that only axisymmetric modelling needed to be carried out.

## 6. Boundary and interface conditions

It was assumed that the origin of a set of cylindrical polar coordinates ( $r, z$ ) was located on the surface of the coating at the point of first contact by the spherical indenter. The  $z$ -axis was directed downwards into the coating and substrate. The state of loading was characterised by the radius,  $a$ , of the projected contact area. The displacement components were taken as being zero at the point on the free surface where first contact was made. The penetration depth of the indenter tip when the radius of the contact area was  $a$ , is denoted by  $w_o(a)$ . On the free surface of the coating :

$$\sigma_{rz}(r, 0; a) = 0, \quad \sigma_{zz}(r, 0; a) = 0, \quad r > a.$$

On the coating surface beneath the indenter one of the following three conditions were imposed (participants of the INDICOAT intercomparison could choose to attempt solutions involving one or more of these conditions)

i) for a freely slipping indenter :

$$\sigma_{rz}(r, 0; a) = 0, \quad u_z(r, 0; a) = w_o(a) + f(r), \quad r < a,$$

where  $z = f(r)$  describes the shape of the rigid indenter relative to the vertex of the indenter so that  $f(0) = 0$ . N.B.  $f(r)$  will always be negative.

ii) for a frictionally slipping indenter :

$$\sigma_{rz}(r, 0; a) = \mu \sigma_{zz}(r, 0; a), \quad u_z(r, 0; a) = w_o(a) + f(r), \quad r < a,$$

where  $\mu$  is the coefficient of friction. For predictions the values  $\mu = 0.1$  and  $0.9$  were used.

iii) for conditions of perfect adhesion :

$$\frac{\partial u_r}{\partial a}(r, 0; a) = 0, \quad u_z(r, 0; a) = w_o(a) + f(r), \quad r < a.$$

On the coating/substrate interface it was assumed that all the traction and displacement components are continuous across the interface. This means that interface remains perfectly bonded during deformation.

## 7. Results of the intercomparison

The methodology of the INDICOAT inter-comparison exercise was to provide participants with a clear specification of the indentation problems that needed to be solved. Participants were asked to submit their results to an independent scientist at NPL who was not involved in making any predictions. He collated the results and prepared the comparison graphs that will now be described. Each participant was given a letter code so that their results could be identified. The coding used to identify participants is as follows :

- Data set A - JRC (FEA - ABAQUS) Friction coefficient = 0.9
- Data set B - NPL (FEA - ABAQUS)
- Data set C - JRC (FEA - ABAQUS) Friction coefficient = 0.1
- Data set D - KTH (FEA - ANSYS)
- Data set E - NPL (Hybrid model - analysis & FEA for rigid punch)
- Data set F - TU Chemnitz (Analytical - Elastica)
- Data set G - VTT (Analytical)

### Load/penetration curves

The load/penetration results of predictions are shown in Figs. 2-7.

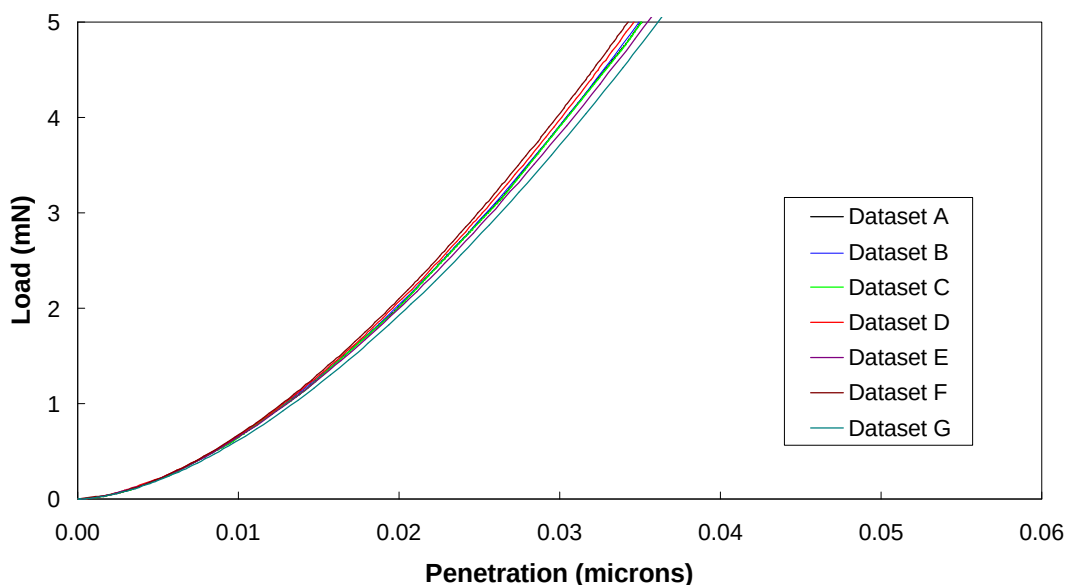


*Fig. 2 : Comparison of all models: Soft coating (0.2 mm) on hard substrate.*

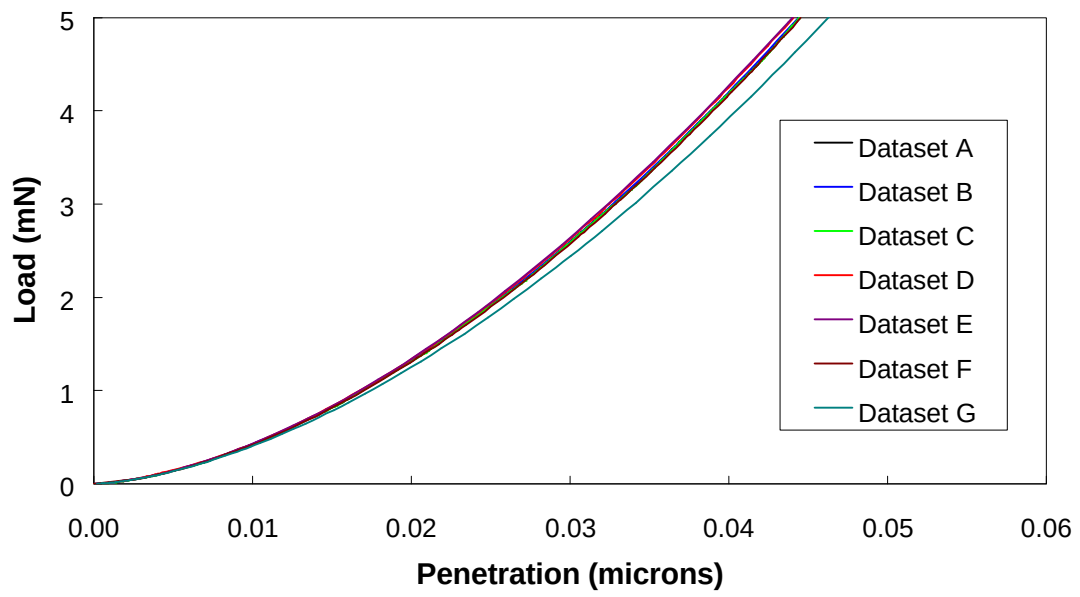


*Fig. 3 : Comparison of all models: Soft coating (0.8 mm) on hard substrate.*

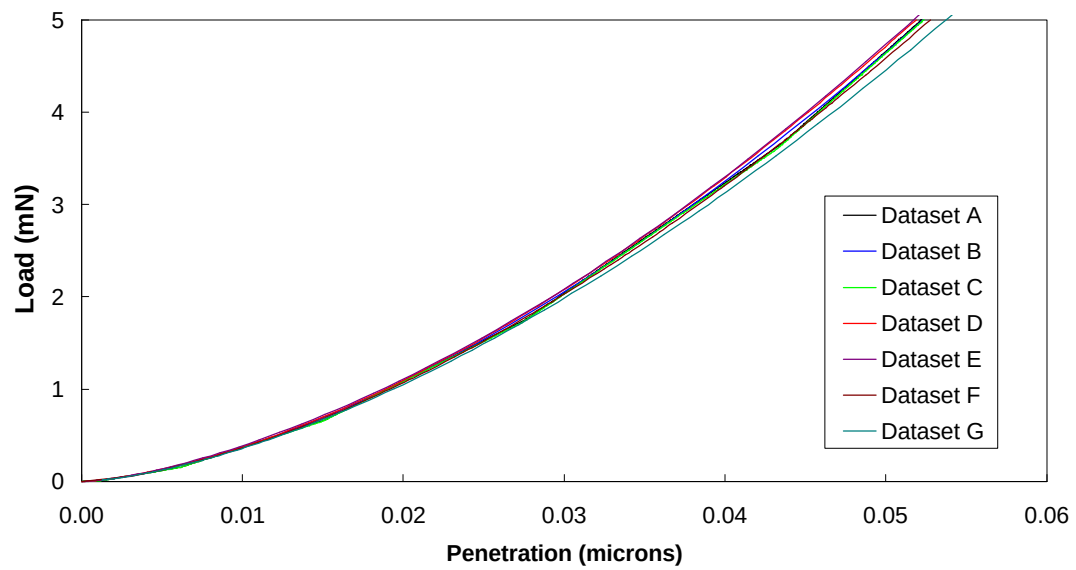


*Fig. 4 : Comparison of all models: Soft (2 mm) on hard substrate*

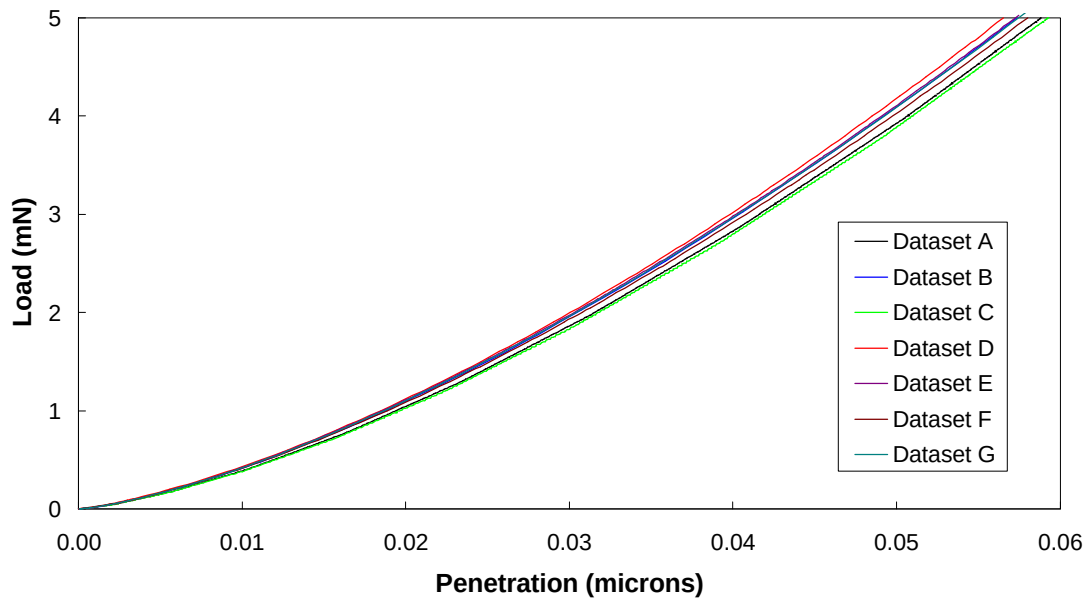


*Fig. 5 : Comparison of all models: Hard coating (0.2 mm) on soft substrate.*

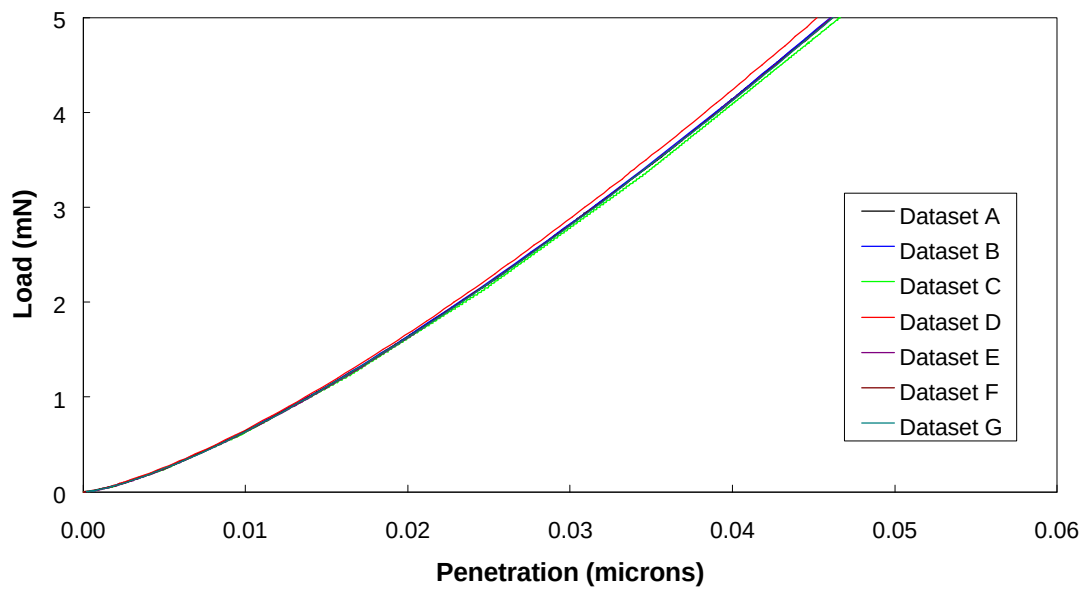


*Fig. 6 : Comparison of all models: Hard coating (0.8 mm) on soft substrate.*

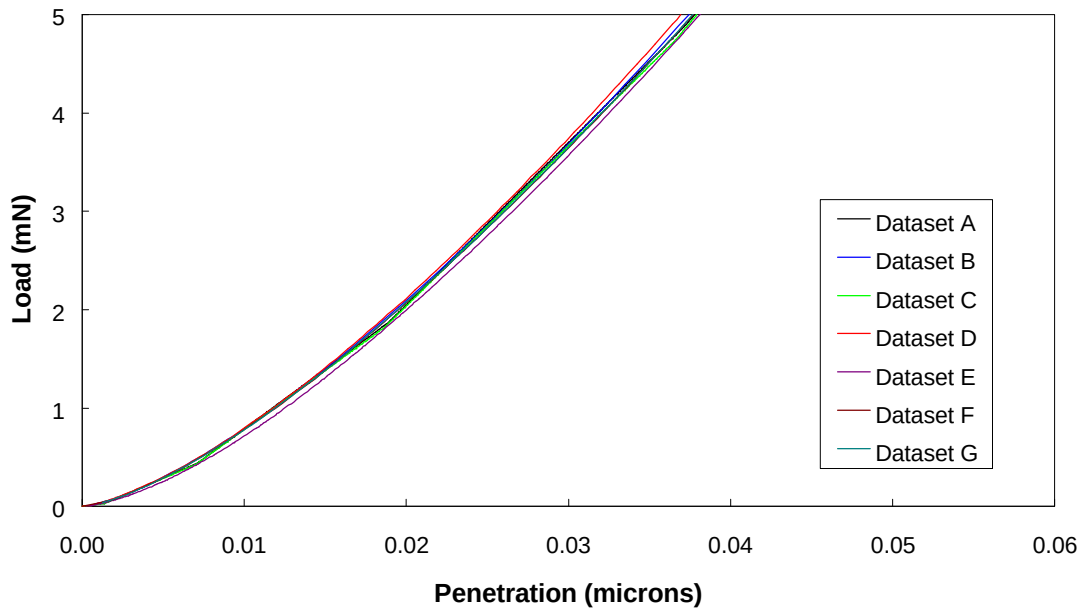


*Fig. 7 : Comparison of all models: Hard coating (2 mm) on soft substrate.*

From the results given in Figs. 2-7 it is seen that there is generally reasonable agreement between the various predictions, although one would have hoped for a unique curve rather than a set of curves that do indicate discrepancies arising from either analytical approximations or from differing finite element meshes. The results for a hard coating on a soft substrate show better agreement than those for a soft coating on a hard substrate. Dataset G certainly does not agree with the other datasets when analysing soft coatings.

### Surface profiles

When significant plasticity occurs during indentation, the profile of the deformed free surface can exhibit the phenomenon of pile-up where material has been displaced by the indenter. For elastic predictions such pile-up does not occur, but it is of interest to compare the surface deformations predicted by the various models. Such a comparison is shown in Figs. 8 & 9 for the case of the axial surface displacement component which is taken as positive where the load applied is 5 mN.

Fig. 8 shows the results for a hard coating of thickness  $0.2\ \mu\text{m}$  applied to a soft substrate. This curve represents the poorest agreement that has been obtained. The only real discrepancy is observed at very low values of  $r$  and this is small in magnitude. Fig. 9 shows the results for a soft coating of thickness  $2.0\ \mu\text{m}$  applied to a hard substrate. This curve represents the best agreement that has been obtained.

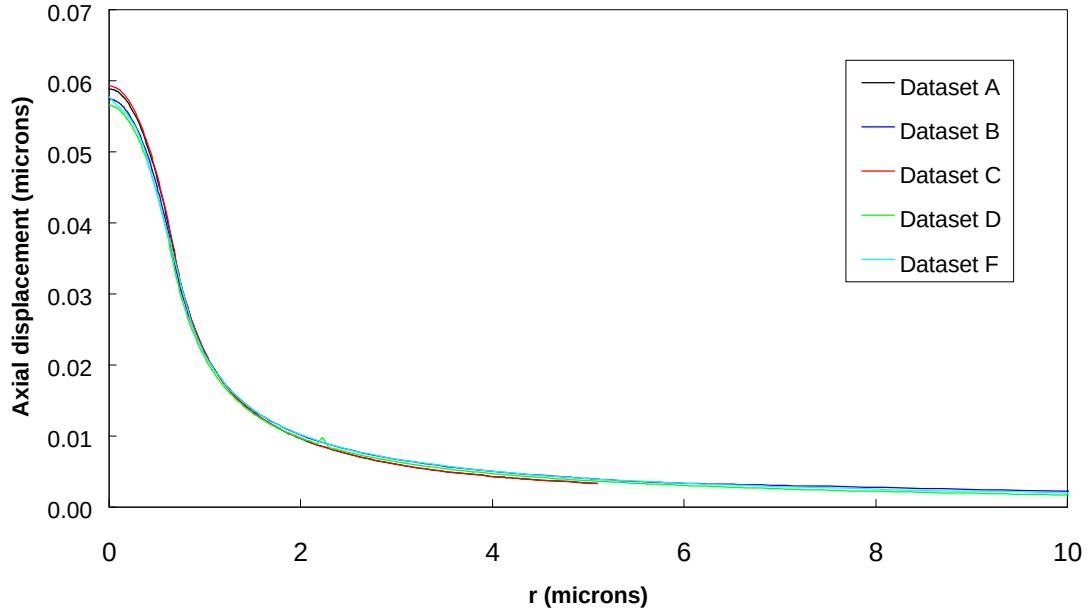


Fig. 8 : Comparison of surface profiles: Hard coating (0.2 mm) on soft substrate.

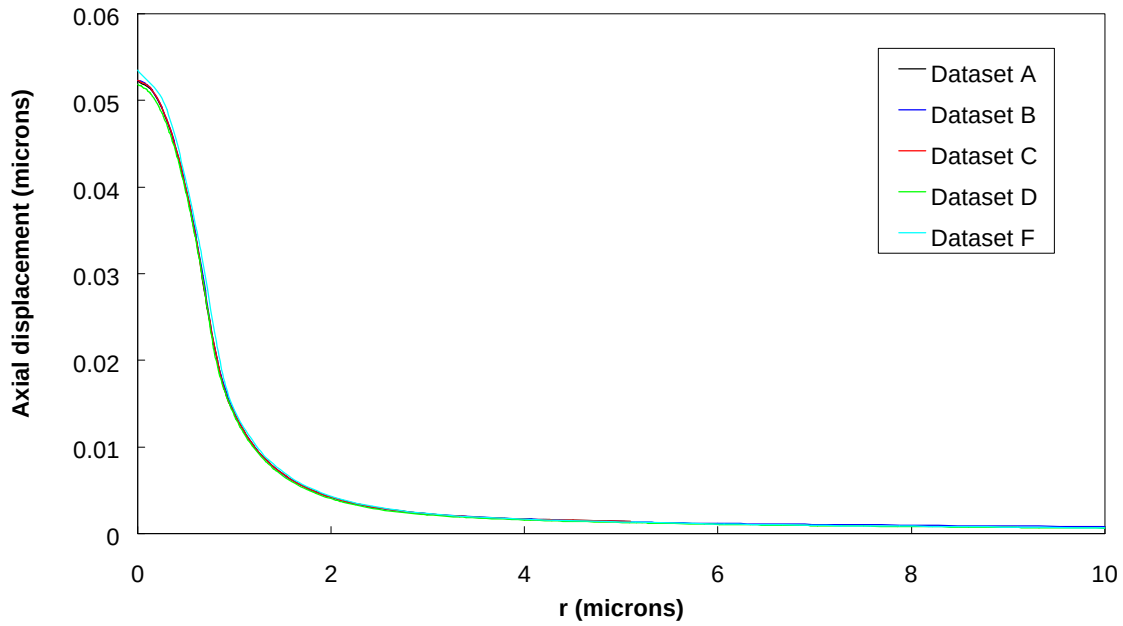


Fig. 9 : Comparison of surface profiles: Soft coating (2.0 mm) on hard substrate.

## 8. Estimation of $E_{IT}$

For elastic deformations of a monolithic sample of material, the method of estimating the properties of the elastic constants is to use the following exact result (to be referred to as Method 1) valid for any axisymmetric rigid indenter in the absence of friction (e.g. [6], but see Appendix, eq. (36))

$$E_{IT} = \frac{1}{2} \sqrt{\frac{\pi}{A_p}} \left( \frac{dP}{dh} \right), \quad (1)$$

where  $E_{IT}$  is the effective modulus measured in an indentation test,  $A_p$  is the projected contact area,  $P$  is the applied load and  $h$  is the corresponding penetration. This approach is also used for coated samples but for this case the value of  $E_{IT}$  is dependent upon the coating and substrate properties and the coating thickness.

For the Hertz solution, provided the contact radius is very much smaller than the indenter radius  $R$ , the value of  $E_{IT}$  is given by the approximation

$$E_{IT} = \frac{3P}{4h} \sqrt{\frac{\pi}{A_p}}, \quad (2)$$

which is a very useful result that avoids having to estimate the derivative  $dP/dh$  from load-penetration curves (a procedure that is well known to introduce significant errors if the data is not smooth). The projected contact area (and contact radius) may be approximated by

$$A_p \equiv \pi a^2 = \pi R h, \quad (3)$$

which leads to the following approximate expression for  $E_{IT}$  that is now independent of the contact area and does not require the estimation of the derivative  $dP/dh$

$$E_{IT} = \frac{3P}{4h\sqrt{Rh}}. \quad (4)$$

The result (4), and the approximation (3) for estimating the contact area and radius, will be referred to as Method 2. It is useful now to provide an example of estimates of the effective modulus  $E_{IT}$  based on the formula (1) and its convenient approximation (4). For this purpose the commercially available system ‘Elastica’ has been used that was developed by one of the INDICOAT project partners (TUC). Two cases have been considered corresponding to a hard coating on a soft substrate (Fig. 10) and vice versa (Fig. 11). The solid symbols show the estimates using the accurate Method 1 of the indentation modulus  $E_{IT}$  as a function of the ratio  $t_c/a$  where  $t_c$  is the coating thickness and  $a$  is the contact radius. The corresponding open symbols show the results obtained when using the approximate Method 2. It should be noted that various indenter radii and coating thicknesses have been used.

The first essential characteristic that follows from the results in Figs. 10 and 11, is that the curves generated by the results do not depend upon the thickness of the coating, nor on the radius of the indenter. In fact it has been shown using the hybrid model that the shape of the indenter does not affect this curve. These well known characteristics indicate that the method used to plot the results in Figs. 10 and 11 are the basis of a convenient measurement method for estimating coating properties.

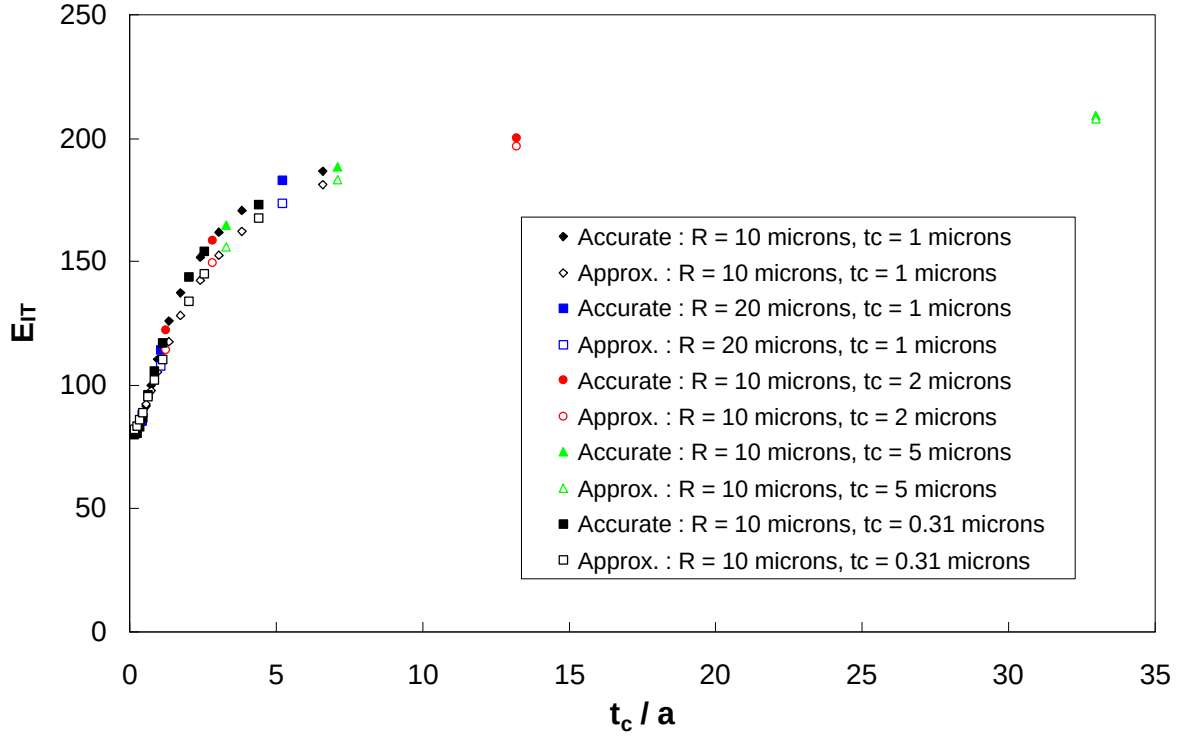


Fig. 10 : *Elastica* estimates of  $E_{IT}$  for a hard coating on a soft substrate by a rigid spherical indenter.

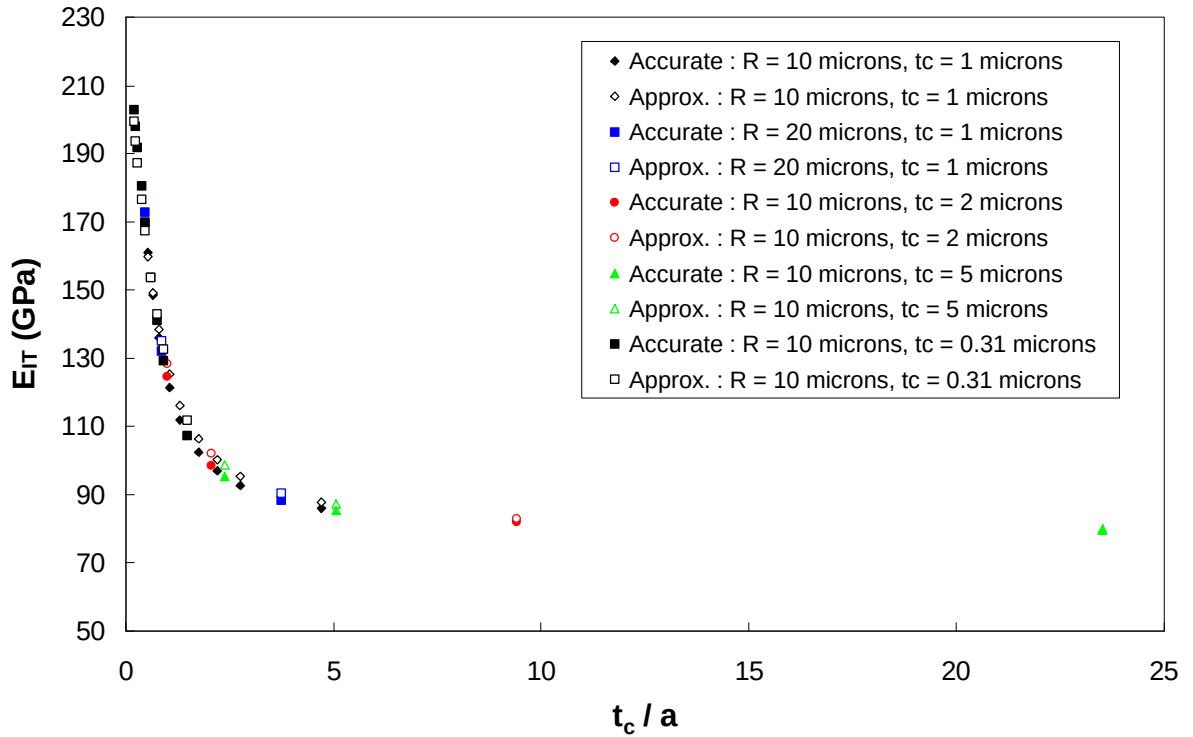


Fig. 11 : *Elastica* estimates of  $E_{IT}$  for a soft coating on a hard substrate by a rigid spherical indenter.

It is seen from Figs. 10 and 11 that Methods 1 and 2 lead to very similar predictions for all values of  $t_c/a$ . As  $t_c/a \rightarrow 0$  the results approach the value  $E_{IT}^s$  for the substrate, and as  $t_c/a \rightarrow \infty$  the curves approach the value  $E_{IT}^c$  for the coating. In these limits it should be noted that the predictions of Methods 1 and 2 tend to the same limiting values of  $E_{IT}$  corresponding to indentation into bulk substrate and coating respectively. It should also be noted that the open symbols predicted by the Method 2 are based on the approximate relation (4) that characterises indentation into homogeneous materials. The values of  $E_{IT}$  given by this approximation correspond to the effective reduced modulus of the coating and substrate when they are regarded as being smeared into an equivalent homogeneous medium having properties that will reproduce, to a good approximation, the load – penetration curves for penetration into the actual coating and substrate system. It is concluded from the comparison of predictions based on Methods 1 and 2 that the approximate Method 2 for the analysis of load penetration data will lead to very reasonable results having only a small error. This method avoids having to differentiate load-penetration data, and estimate the contact radius  $a$  from the indenter's area function.

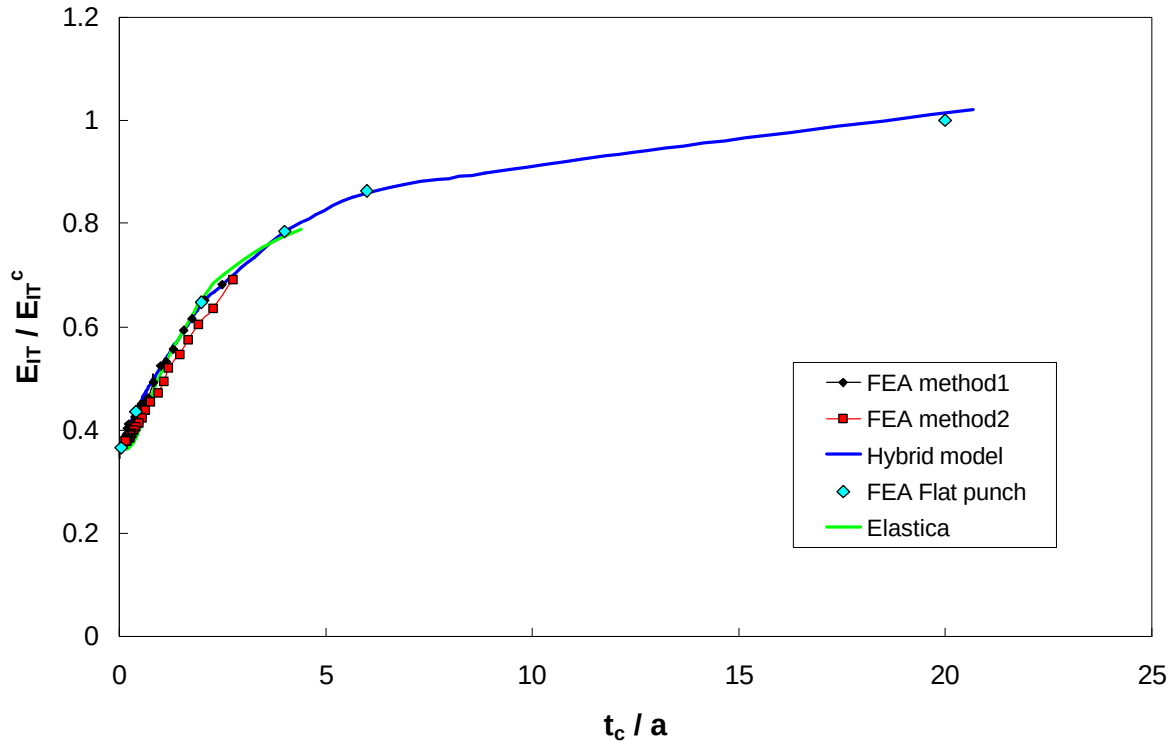


*Fig. 12 : Elastic estimates of  $E_{IT}$  for a hard coating of thickness 0.31 mm on a soft substrate by rigid indenters.*

For the case of the hard coating of thickness 0.31  $\mu\text{m}$  applied to the soft substrate, Fig.12 shows the dependence of  $E_{IT} / E_{IT}^c$  on the ratio  $t_c/a$ . This thickness of coating corresponds to one value used when carrying out FEA of a flat rigid punch for application to the hybrid solution technique. The results labelled ‘FEA Flat punch’ were derived using the refined hybrid solution technique (see Appendix 1). The results labelled ‘Hybrid-method’ have been obtained by applying (1) to the load-penetration data generated by the hybrid model for a spherical indenter of radius 10  $\mu\text{m}$ . It is seen that they correspond almost exactly to the FEA results for a flat circular punch. This is to be expected as, for the case of elastic deformations,

the results when plotted as shown in Fig. 12 should not depend on the geometry of the axisymmetric indenter. As another example of the geometry independence for axisymmetric indenters, it should be noted that the value of  $E_{IT}$  should be independent of the radius of the spherical indenter for all values of  $t_c/a$ : a property that has been confirmed using the hybrid model. This result implies that there is no effect of the shape and elasticity (i.e. shape change during loading) of the indenter, provided that the penetration is measured from the free surface. Experimentally such measurements are not usually possible with the result that corrections to account for the elasticity of the indenter are needed only to compensate for the contribution of the indenter to the penetration measurement. The results labelled 'FEA (sphere) – Method 1' are for a rigid spherical indenter of radius 10  $\mu\text{m}$  obtained using the accurate relation (1) for  $E_{IT}$ , which can only be implemented approximately due to inaccuracies in the numerical estimation of  $dP/dh$ , and of the contact area. The corresponding results obtained from the same data using the approximate, but easy to use, relation (4) are denoted by 'FEA (sphere) – Method 2'.

From the results presented in Fig. 12 it is clear that the methodology of the hybrid model is providing very consistent results for the case of elastic deformations as the same values of  $E_{IT}$  are predicted when using both spherical indenters (of any radius consistent with elastic deformations) and a flat circular punch. This verifies for another case, that all axisymmetric indenters described by the hybrid model, should give results lying on the same curve. In addition, the approximate method of estimating  $E_{IT}$  based on Method 2 above leads to results that are in very good agreement with those obtained using the accurate Method 1 except for small values of  $t_c/a$  which occur when the coating is very thin or the load is large (where plasticity would also be encountered). The 'Elastica' prediction is also in good agreement although the actual shape of the curve differs slightly from that of the other results at both low and high values of  $t_c/a$ .

The essential difference between the finite element results based on spheres and cylinders is the variation in contact area with load in the case of the spherical indenters. This is highlighted by the fact that the cylinder solution may be obtained from the finite element mesh as a linear perturbation solution whereas the sphere solution may not as the geometry always varies with load because of changes in contact area. Hertz avoids this problem by solving a moving boundary problem as though it were a fixed boundary problem having a continuity condition. It is reassuring that the finite element solutions for the spherical indenters give estimates of  $E_{IT}$  that are in agreement with those for flat cylindrical indenters.

Figures 13 and 14 show the results of comparing the predictions of FEA carried out by KTH (Method 1 only) with the NPL hybrid model (Methods 1 and 2), and with the Gao et al analytical model [7] for the case of the hard coating on the soft substrate and vice versa. It is seen that the trends of the FEA results for spherical indenters are, on the whole, in good agreement with the FEA results for the flat circular punch, and the hybrid model. The FEA results for spherical indentation, for the case of a hard coating on a soft substrate, do show a high degree of scatter that arises from the difficulties of estimating the contact area. This problem does not arise for the flat punch data which produce much smoother data. For the case of a soft coating applied to a hard substrate the FEA results for spherical indenters show some scatter and the data agrees very well with the flat punch and hybrid model results. The results of using the Gao et al model, which is thought to be the best available purely analytical technique, do not follow the FEA data, nor the hybrid model where the discrepancy is more marked for the case of a soft coating applied to a hard substrate.

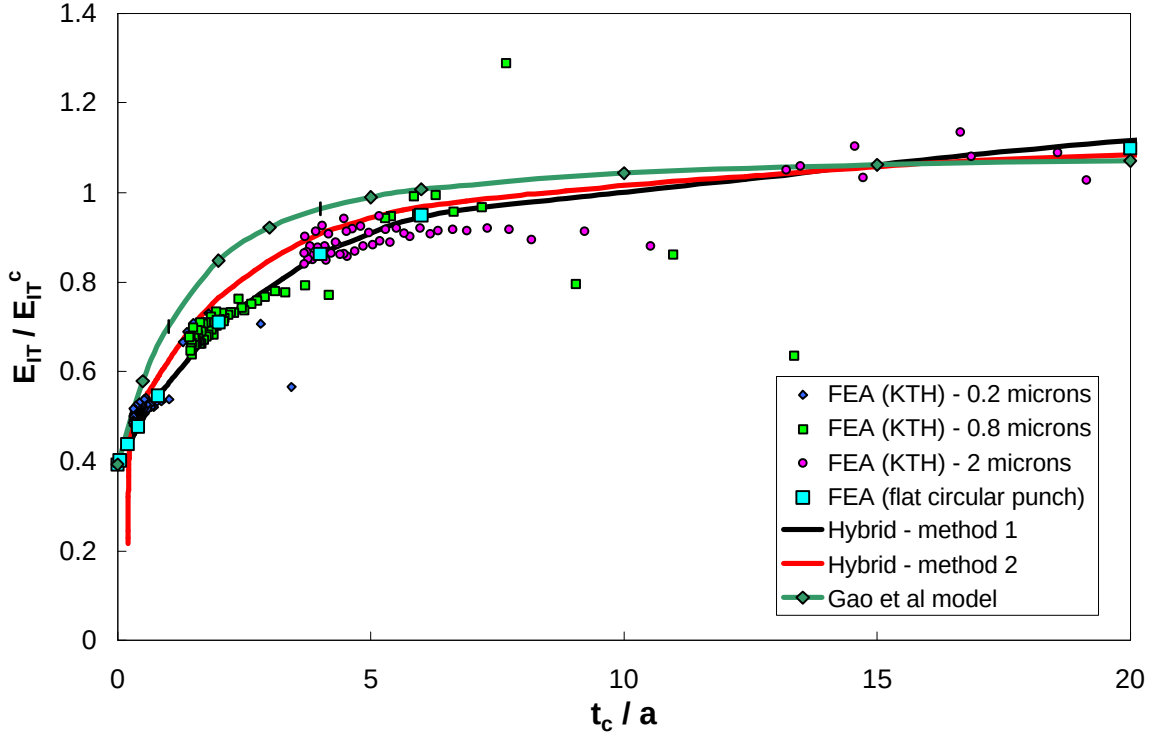


Fig. 13 : Comparison of elastic estimates of  $E_{IT}$  for hard coatings on a soft substrate by rigid indenters.

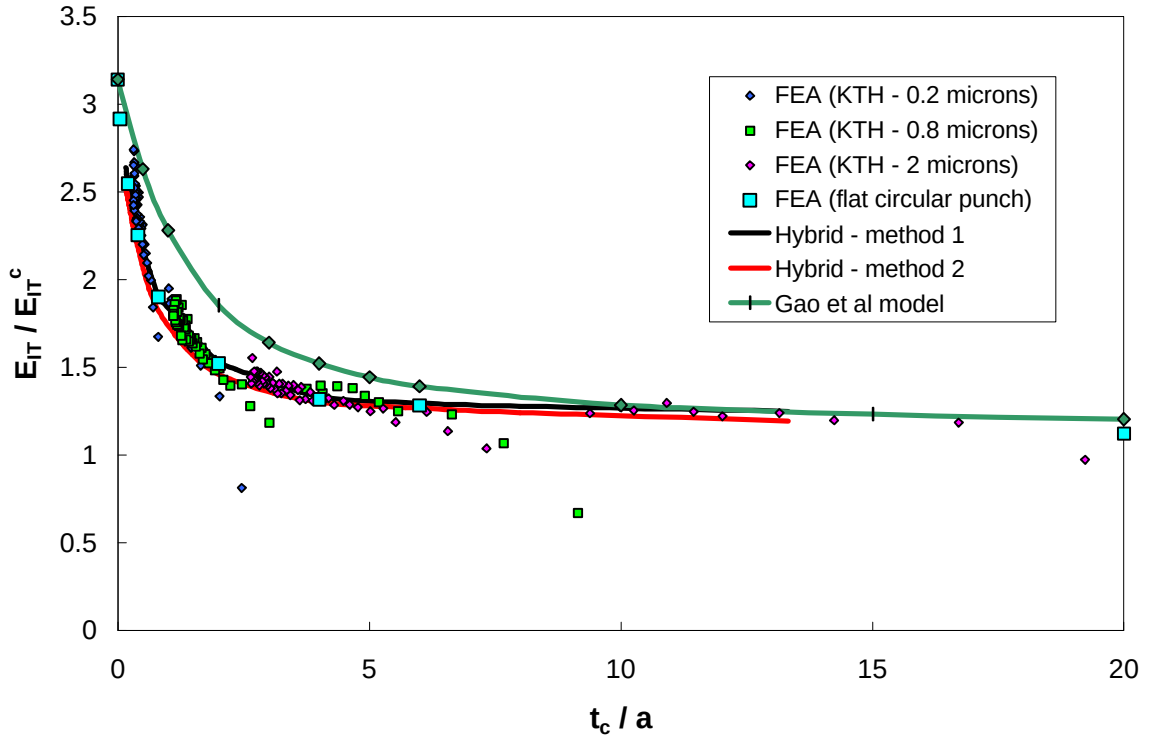


Fig. 14 : Comparison of elastic estimates of  $E_{IT}$  for soft coatings on a hard substrate by rigid indenters.

In Fig. 15 is shown the result of comparing experimental results with ‘Elastica’ predictions (using Methods 1 and 2) for  $E_{IT}$  for the case of a 2  $\mu\text{m}$  DLC coating applied to an M2 toolsteel

substrate using a spherical indenter of radius  $9.5\text{ }\mu\text{m}$ . For the ‘Elastica’ predictions the indenter has been assumed to be made of diamond rather than being rigid in order to simulate approximately the experimental conditions. It is seen that for values of  $t_c/a$  in the range  $0 \leq t_c/a \leq 5$  there is good agreement between predictions and experimental results [1]. For larger values of  $t_c/a$  the experimental data exhibit large variability arising when the applied loads are small.

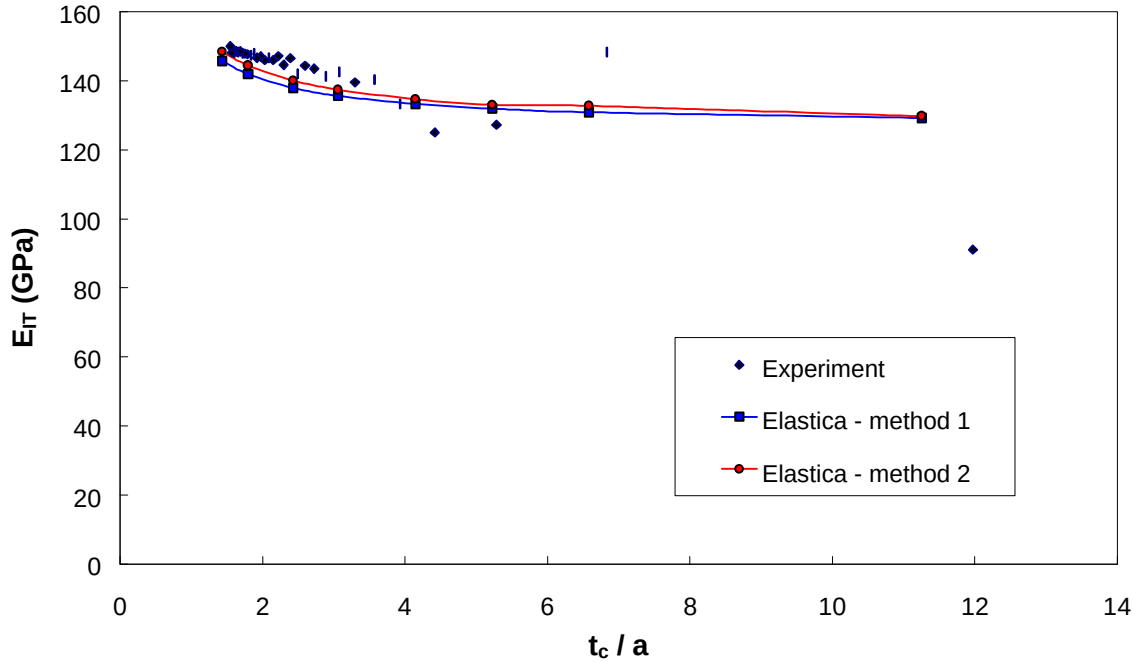


Fig. 15 : Comparison of experimental results with elastic estimates of  $E_{IT}$  for a 2 mm DLC coating on M2 toolsteel substrate indented by a rigid spherical indenter of radius  $9.5\text{ }\mu\text{m}$ .

From the above analysis of elastic indentation the following observations can be made:

1. FEA  $E_{IT}$  results for rigid spherical indenters can be subject to significant errors, especially at low loads where the accurate estimation of the contact area is very difficult,
2. FEA  $E_{IT}$  results agree reasonably well with the FEA for a rigid circular punch where the estimation of contact area is not a problem,
3. hybrid model predictions of  $E_{IT}$  using Methods 1 and 2 agree exceedingly well, and with both the FEA  $E_{IT}$  results for spherical indenters and those for a circular punch,
4. ‘Elastica’ software system produces results for  $E_{IT}$  that are in close agreement with corresponding FEA results. There is, however, a tendency for predictions to have slightly different shape to the FEA data and hybrid model results at both low and large values of  $t_c/a$ ,

5. Gao et al model [6], which is analytical in nature, does not agree well with the other results for values of  $t_c / a < 10$  but is in good agreement with the relatively sparse results in the region  $t_c / a > 10$ ,
6. experimental results obtained for purely elastic deformations agree very well with predictions based on 'Elastica', and hence with the hybrid model and FEA.

## 9. A consideration of 3D indenters

Many nanoindentation tests carried out in industry make use of indenter geometries that are 3D in nature, e.g. the Berkovich indenter. During the formulation of the project task it was thought that the NPL hybrid model (which has been shown above to be very successful for axisymmetric indenters) could be modified to deal with 3D indenter geometries. Using a sharp indenter tip would lead to almost instant plastic deformations during loading which are not included in the analyses presented here. Indeed, if the indenter were a perfect pyramid, then both the tip and the edges would be sharp leading to infinite stress concentrations in these regions. Using a Berkovich indenter, which is in the form of a triangular pyramid, together with a spherical tip that smoothly merges with the non-axisymmetric geometry of the Berkovich indenter, avoids the problem of instant plasticity, but means that when deforming in the elastic regime, the indentation behaves as an axisymmetric indenter that has already been analysed in detail. It is argued that in fact all indenters are rounded at the tip and this disposes of the stress singularity for small loads. Thus, a pyramidal indenter will behave as a spherical indenter for the purposes of investigating *elastic* indentation. For these reasons it was decided that the task objectives should be modified.

The limitation placed on the task where deformations are restricted to the elastic regime meant that there would be little benefit investigating 3D indenter effects. A more fruitful approach is to consider the elastic deformations that arise when an indenter (deformed to the point of significant plasticity) is unloaded elastically, as assumed by the Oliver and Pharr method [6] of analysing load-penetration data. The objective set was first of all to consider the elastic unloading of a spherical indenter applied to a homogeneous material where the only effect of plasticity during loading is to change the geometry of the free surface. The internal residual stress effects arising from plasticity are neglected as their inclusion requires a fully elastic-plastic solution that was outside the remit of the task. Only if satisfactory results are obtained from this investigation will it be sensible to consider 3D indenters.

## 10. Elastic indentation of a pre-dented specimen

Considerable difficulty has been encountered when matching the elastic-plastic FEA predictions of load-deflection curves with the corresponding results of experiments. A key issue is the provision for FEA of reliable elastic-plastic property data (to be discussed in a future report). It should be noted that the experimental results showed considerable scatter, whereas the comparison of FEA results, using different meshes, led to much more consistent FEA results. A critical test was needed in order to assess the accuracy and validity of the FEA results. FEA load-deflection curves were produced based on a model material with known elastic and plastic properties. The results of the FEA were then analysed to obtain estimates

of  $E_{IT}$  using the same methodology (Oliver and Pharr [6]) and associated software that is normally applied to experimental data. The results of this test were not very encouraging and suggested that  $E_{IT}$  could not be reliably estimated in this fashion. Nevertheless, experimentalists give assurances that such methods have been used in experimental analysis for many years and produce consistent results.

One reason for this apparent disagreement could be that the plastic deformation is not being modelled correctly in the finite element analysis. In order to circumvent this issue it was assumed that the plastic deformation merely changed the shape of the specimen without altering the effective modulus, i.e. the residual stresses arising from plastic flow are neglected, and there is no work hardening or induced anisotropy. On making this assumption, the unloading curve may be estimated by indenting a specimen for which its free surface topography corresponds to the, experimentally determined, final unloaded shape of a pre-dented specimen. Experimentally it has been found that the post indentation profile was approximately a spherical indentation in an otherwise plane surface.

The material system chosen for the investigation corresponded to an experiment carried out at QMWC using a spherical indenter having a radius of  $6.63\text{ }\mu\text{m}$ . The specimen had a  $5\text{ }\mu\text{m}$  coating of gold applied to a nickel substrate, and after the indentation an approximately spherical depression was left in the gold coating. The radius of the depression was given, as  $1.54\text{ }\mu\text{m}$  with a maximum depth of  $153\text{ nm}$  below the undeformed free surface, and the radius of curvature at maximum depth was approximately  $7.67\text{ }\mu\text{m}$ .

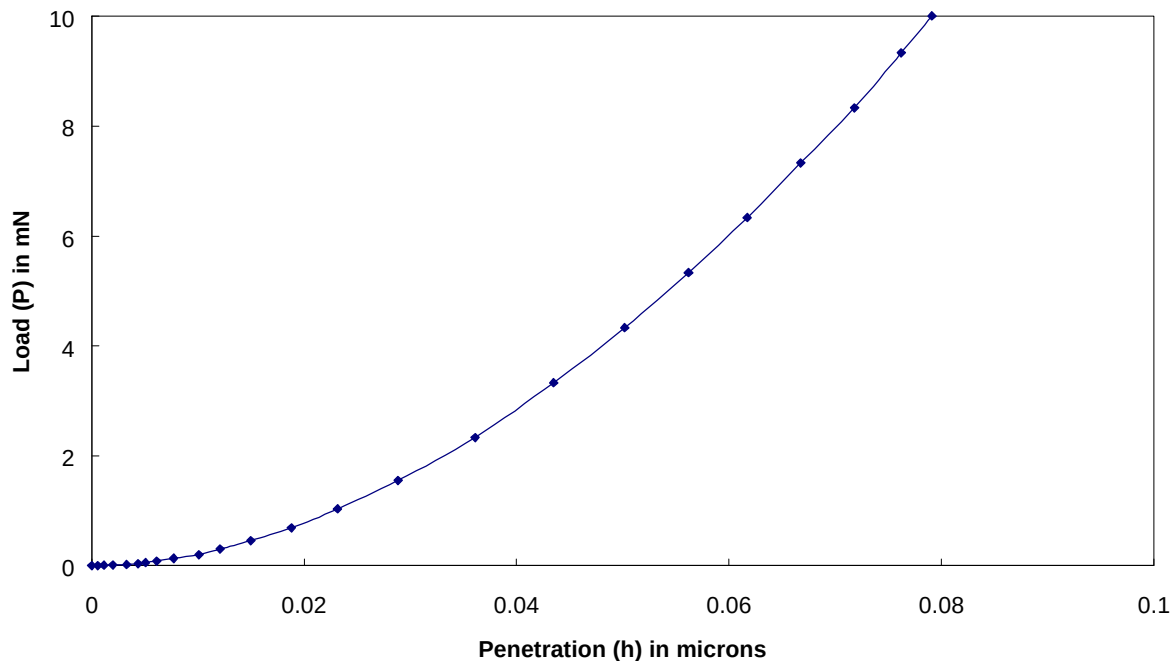


*Fig. 16: The load / penetration curve for a 5mm coating of gold on nickel.*

The results of predictions are given in Fig.16. On increasing the load beyond those plotted in Fig.16, a point is reached where the load-penetration curves become linear. This occurs when the surface of the indenter is in total contact with the surface of the pre-existing surface indentation. The linear response arises because a linear elastic analysis is being carried out,

and the contact area is not changing with increases in the applied load. It might be expected that the curve shown in Fig. 16 should correspond to a Hertzian case of the indentation of a sphere into a spherical cup of somewhat larger radius. If this were the case then a plot of the load  $P$  against  $h^{3/2}$  would be a straight line.

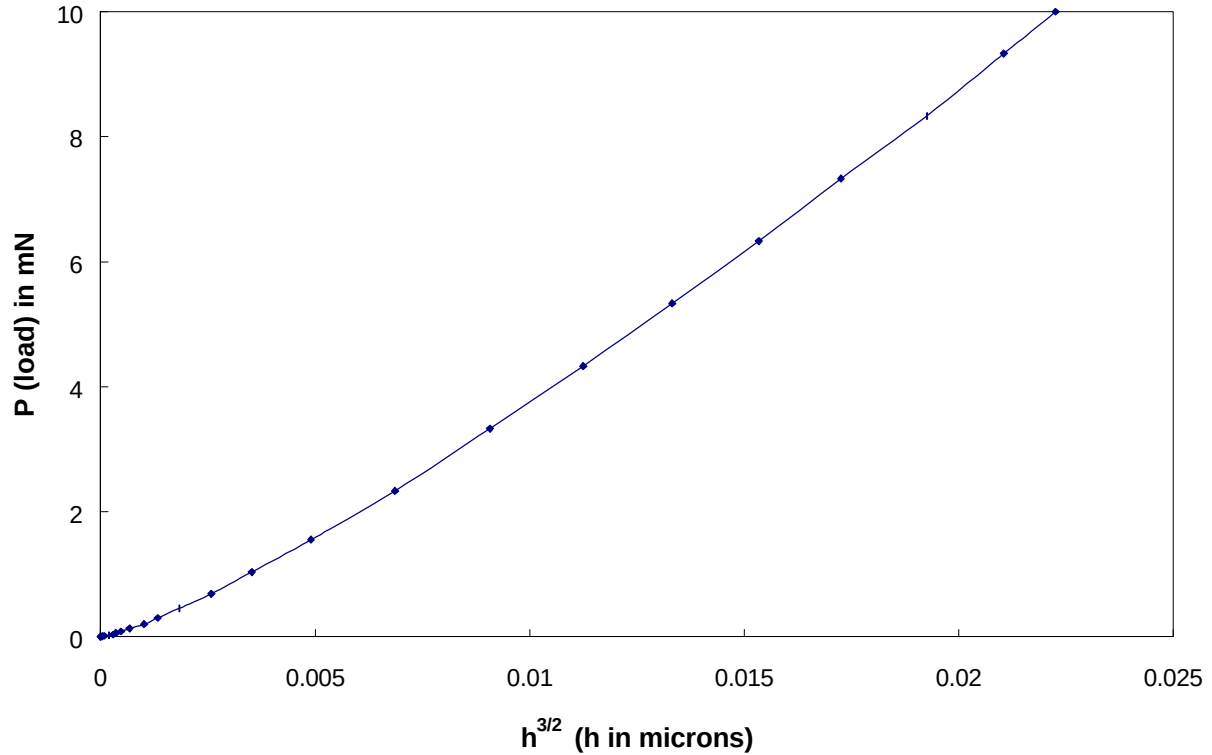


Fig. 17: Load versus  $h^{3/2}$  that would be a straight line in a Hertzian analysis.

It is seen from Fig. 17 that plotting load as a function of  $h^{3/2}$  does not quite transform the load-displacement plot into a straight line. As the analysis is indeed elastic, this solution suggests that the geometry of the pre-dented specimen is sufficient to significantly perturb the well known Hertzian 3/2 power law result. An important consequence of this result is that the Oliver and Pharr [6] technique for estimating  $E_{IT}$  may not lead to expected value due the influence of the geometry of the residual indentation created in experiments during loading. This result implies that there would be no benefit in repeating the analysis for the case of a 3D indenter.

## 11. Conclusions

1. For elastic analyses, there is generally good agreement between predictions of load-penetration behaviour based on analytical modelling and the corresponding FEA results for all thicknesses considered, and for both hard coatings on soft substrates and for soft coatings on hard substrates. This means that any of the prediction techniques can be used with confidence when predicting the elastic response of the indentation of coating and substrate.

2. The effects of the magnitude and nature of friction, at the low load levels considered for elastic responses, are very small.
3. When comparing theoretical predictions of load-penetration behaviour for elastic indentation with experimental results, use can be made of any of the predictive techniques considered.
4. When comparing predictions of load-penetration behaviour given by the analytical models, the agreement is better for the case of a hard coating on a soft substrate. The agreement is however good for the case of a soft coating on a hard substrate when the coating thickness is  $0.2 \mu\text{m}$ .
5. When comparing predictions of the load-penetration behaviour of FEA models, the agreement is better for the case of a soft coating on a hard substrate, which is very good for all the coating thicknesses considered. For the case of a hard coating on a soft substrate the agreement is good for the thicknesses  $0.8 \mu\text{m}$  and  $2.0 \mu\text{m}$  but when the thickness is  $0.2 \mu\text{m}$  there are some discrepancies at larger loads.
6. When comparing predictions of the axial displacement of the free surface when the applied load is  $5 \text{ mN}$ , there is very good agreement between the models.
7. When estimating for a coated system the indentation modulus  $E_{IT}$  very good agreement between the FEA analyses, 'Elastica' and the hybrid model is achieved for a wide range of properties, coating thicknesses and axisymmetric indenters (spheres of various radii and flat circular punches).
8. When estimating  $E_{IT}$  for a coated system it is found that the predictions obtained based on the use of FEA, 'Elastica' and the hybrid model differ from those of the analytical Gao et al model [7] except when  $t_c / a \rightarrow 0$  and  $t_c / a \rightarrow \infty$ . The discrepancy between the Gao et al model and the hybrid model is thought to arise from a basic inaccuracy of the former.
9. The validity of the Gao et al model [7] at large values of  $t_c / a$  means that it should be possible to unravel both Young's modulus and Poisson's ratio from the results of indentation tests by making use of thick coatings and applying low loads. This approach is consistent with the use of thick coatings, and large indenter radii and low loads that avoid plasticity so that the results of elastic modelling are applicable.
10. The elastic analysis of the effect of indentation into a pre-existing indent in the coating surface has shown that Hertzian unloading behaviour is not observed due to the stress concentration effect of the pre-existing indent, and this leads to uncertainties in the values of  $E_{IT}$  when using the Oliver and Pharr method to interpret the load-penetration curve.

## Acknowledgement

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## APPENDIX 1 : Indentation mechanics for a coated half-space - A hybrid model

### Background

The principal technical objective of this work is to attempt to predict the elastic load-deflection behaviour of a rigid spherical indenter that is applied to a coated semi-infinite substrate. A second objective is to assess whether it is possible to estimate the modulus of the coating from the load-deflection curve. For a spherical indenter, the load-deflection behaviour in the elastic regime of deformation is non-linear because of variations in the contact area during loading. A major problem encountered in an elastic analysis is knowing the contact area as a function of applied load. The elasticity problem thus involves the prediction of the movement during loading of the boundary of contact that affects the applied boundary conditions. This problem arises also when attempting a solution based on finite element analysis (FEA).

The approach of this report is to attempt an analysis where it is not necessary to identify the moving boundary determined by the contact area. When the indenter is a cylindrical flat rigid punch the contact area is regarded as being known, and the load-deflection behaviour is linear in the elastic regime of deformation. The load-deflection behaviour for a spherical punch is shown to be equivalent to an indentation that arises from the superposition of a distribution of flat cylindrical punches having a continuous range of diameters. The solution for a particular coating and substrate system will be constructed from linear load-deflection information obtained using FEA for a flat rigid cylindrical punch of unit radius applied to coatings having a range of different thicknesses. Such solutions can be used to predict behaviour for any other punch radius. This hybrid approach, involving both analytical modelling and FEA, avoids having to determine the contact area as a function of load when applying the solution procedure. Such information will be obtained directly as part of the solution to the problem that will be posed. The methodology can also be used to analyse different axisymmetric shapes of indenters, and can solve for a range of coating thicknesses without having to carry out further FEA. The proposed method thus has the potential for being very versatile.

### Superposition technique

A coated elastic half-space occupies the region  $0 \leq r < \infty$ ,  $z \geq 0$ . The coating of uniform thickness  $t_c$  occupying the region  $0 \leq r < \infty$ ,  $0 \leq z \leq t_c$  is assumed to be linear elastic and isotropic such that the Young's modulus is  $E_c$  and Poisson's ratio is  $\nu_c$ . The underlying half-space occupying the region  $0 \leq r < \infty$ ,  $z > t_c$  is also assumed to be linear elastic and isotropic such that the Young's modulus is  $E_s$  and Poisson's ratio is  $\nu_s$ . The non-zero stress components for axisymmetric indentation problems are denoted by  $\sigma_{rr}(r, z)$ ,  $\sigma_{zz}(r, z)$  and  $\sigma_{rz}(r, z)$ . The displacement components in the  $r$  and  $z$  directions are denoted respectively by  $u(r, z)$  and  $w(r, z)$ .

Consider the indentation of the coated elastic half-space by a rigid circular flat punch having radius  $a$  such that the vertical displacement  $w$  in the  $z$ -direction is uniform having the value  $w = 1$  on the part of the free surface specified by  $0 \leq r \leq a$ ,  $z = 0$ . Two types of boundary condition may be investigated. The first assumes that the indentation occurs such that the shear traction is everywhere zero on the surface  $z = 0$ , including in the region of the contact.

Such a boundary condition is relevant for friction-free indentation. The second type assumes that the shear tractions are zero on  $z = 0$  for  $a \leq r \leq \infty$  and that  $u(r,0) = 0$  for  $0 \leq r \leq a$ . This boundary condition corresponds to indentation for the case of perfect adhesion. The profile of the free surface resulting from an indentation is given by  $w = \Phi(r/a, t_c/a)$  where  $\Phi(r/a, t_c/a) = 1$  for  $0 \leq r \leq a$ . The geometry in the deformed state is illustrated schematically in Fig.1.

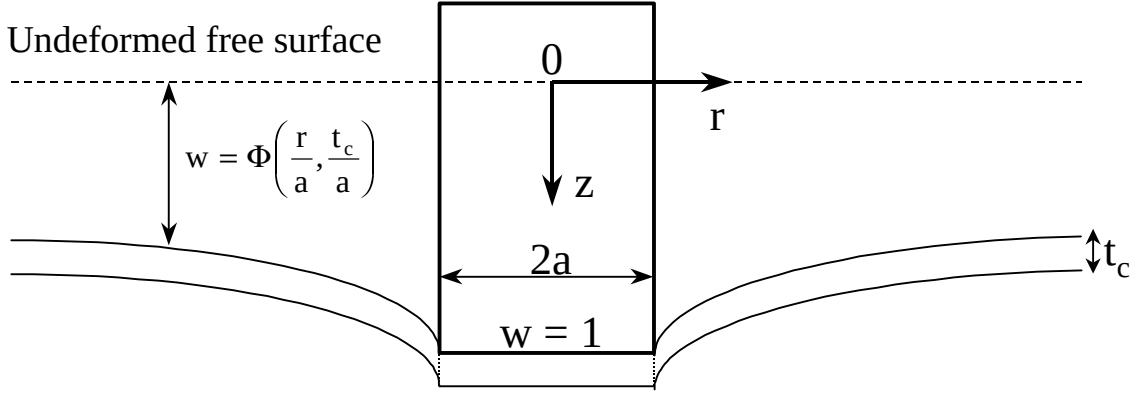


Fig.1 : Schematic diagram of a coated half-space indented by a rigid flat cylindrical punch.

The load applied to the punch is denoted by  $L$ . Because the material is assumed to behave linearly elastically it follows that for any uniform applied vertical displacement  $\omega(a) = w(0,0)$  the corresponding applied load is such that

$$L = a \omega(a) \alpha \left( \frac{t_c}{a} \right). \quad (1)$$

The parameter  $\omega(a)$  is the penetration of the flat punch when its radius is  $a$  and the function  $\alpha$  has the dimensions of modulus. For the special case when the coating is not present (i.e.  $t_c = 0$ ) it follows from Sneddon (1965) that

$$\frac{L}{a \omega(a)} = \alpha = \frac{2E}{1-\nu^2}. \quad (2)$$

In (1) it is expected that as  $t_c \rightarrow 0$  the value of  $\alpha$  is given by (2) where  $E$  and  $\nu$  are Young's modulus and Poisson's ratio for the substrate material.

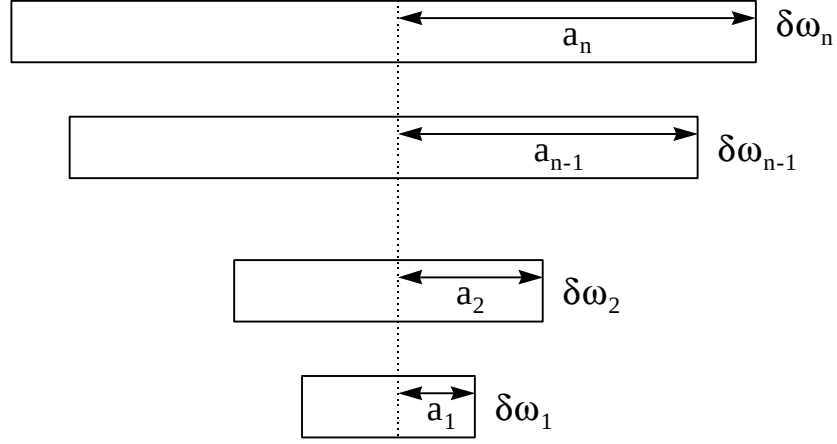


Fig.2 : Schematic diagram of indenter problems to be superimposed.

Consider now the set of indenter shapes shown in Fig.2 that have an axisymmetric geometry and comprise a series of flat circular punches of different radii. The radii of the flat indenters are given by the monotonic increasing sequence  $a_1, a_2, \dots, a_{n-1}, a_n = a$ , and it is assumed that  $a_0 = 0$ . The problem of interest is the superposition of indentation problems for  $i = 1 \dots n$ , where the flat punch having radius  $a_i$  is subject to a uniform vertical displacement  $\delta\omega_i$ . The resulting surface profile of the vertical displacement following superposition is given by

$$w(r,0) = \Phi\left(\frac{r}{a_1}, \frac{t_c}{a_1}\right)\delta\omega_1 + \Phi\left(\frac{r}{a_2}, \frac{t_c}{a_2}\right)\delta\omega_2 + \dots + \Phi\left(\frac{r}{a_{n-1}}, \frac{t_c}{a_{n-1}}\right)\delta\omega_{n-1} + \Phi\left(\frac{r}{a_n}, \frac{t_c}{a_n}\right)\delta\omega_n. \quad (3)$$

Since  $\Phi(r/a, t_c/a) = 1$  for  $0 \leq r \leq a$  the penetration of the indentation following superposition is given by

$$w(0,0) = \omega(a) = \delta\omega_1 + \delta\omega_2 + \dots + \delta\omega_{n-1} + \delta\omega_n. \quad (4)$$

It follows from (1) that the corresponding applied load is given by

$$L = a_1 \alpha\left(\frac{t_c}{a_1}\right)\delta\omega_1 + a_2 \alpha\left(\frac{t_c}{a_2}\right)\delta\omega_2 + \dots + a_{n-1} \alpha\left(\frac{t_c}{a_{n-1}}\right)\delta\omega_{n-1} + a_n \alpha\left(\frac{t_c}{a_n}\right)\delta\omega_n. \quad (5)$$

On letting  $a_{i+1} = a_i + \delta a_i$  and then taking the limit  $\delta a_i \rightarrow 0$ , it follows that  $\delta\omega_i \rightarrow \omega'(a_i)\delta a_i$  where  $\omega'(a)$  is the derivative, with respect to the contact radius  $a$ , of the penetration function  $\omega(a)$  such that  $\omega_i = \omega(a_i)$  for  $i = 1 \dots n$ . The relations (3) and (5) may then be written

$$w(r,0) = \int_0^a \omega'(t) \Phi\left(\frac{r}{t}, \frac{t_c}{t}\right) dt, \quad (6)$$

$$L(a) = \int_0^a t \omega'(t) \alpha\left(\frac{t_c}{t}\right) dt, \quad (7)$$

where the function  $\omega(a)$  denotes the penetration of the indenter when the contact radius has the value  $a$ , occurring when the applied load is  $L(a)$ . By definition  $\omega(0) = 0$ .

Since  $\Phi(r/t, t_c/t) = 1$  for  $0 \leq r \leq t$  result (6) may be written in the form

$$w(r,0) = \omega(a) - \omega(r) + \int_0^r \omega'(t) \Phi\left(\frac{r}{t}, \frac{t_c}{t}\right) dt, \quad 0 \leq r < a. \quad (8)$$

When the above representation of indentation is applied to a rigid axisymmetric indenter described by  $w = -f(r)$  for  $0 \leq r \leq a$ , it follows that  $w(r,0) = \omega(a) - f(r)$  where the function  $f(r)$  is defined so that  $f(0) = 0$ . It then follows from (8) that

$$\omega(r) = f(r) + \int_0^r \omega'(t) \Phi\left(\frac{r}{t}, \frac{t_c}{t}\right) dt, \quad 0 \leq r < a. \quad (9)$$

Since  $\omega(0) = 0$  the result (9) may be expressed as the following integral equation where the function  $f(r)$  is assumed known and the unknown function is  $\omega'(t)$

$$f(r) = \int_0^r \omega'(t) \left[ 1 - \Phi\left(\frac{r}{t}, \frac{t_c}{t}\right) \right] dt, \quad 0 \leq r < a. \quad (10)$$

### Solution for uncoated half-space

Consider now the case of the indentation of an uncoated half-space where the function  $\Phi = \Psi(r/a)$ . On setting  $t = r/\rho$  the integral equation (10) may be written in the form

$$f(r) = \int_1^\infty \frac{r}{\rho} \omega'\left(\frac{r}{\rho}\right) [1 - \Psi(\rho)] \frac{d\rho}{\rho}, \quad 0 \leq r < a. \quad (11)$$

The integral equation (11) may be solved analytically if the prescribed function  $f(r)$  defining the indenter shape can be expanded in the even form

$$f(r) = \sum_{k=1}^{\infty} A_k r^{2k}. \quad (12)$$

It is then clear from (11) that the required solution is given exactly by

$$\omega(a) = \sum_{k=1}^{\infty} B_k a^{2k}, \quad (13)$$

where

$$B_k = \frac{A_k}{1 - 2k \int_1^\infty \Psi(\rho) \frac{d\rho}{\rho^{2k+1}}}, \quad k = 1 \dots N. \quad (14)$$

### Indentation by a sphere

For a spherical indenter of radius  $R$  the function  $f(r)$  is given by

$$f(r) = R - \sqrt{R^2 - r^2} \quad (15)$$

On expanding, the spherical profile defined by (15) may be expressed

$$f(r) = R \sum_{k=1}^{\infty} \frac{1}{2k-1} \frac{(2k)!}{2^{2k}(k!)^2} \left(\frac{r}{R}\right)^{2k}, \quad (16)$$

so that the values of the coefficients  $A_k$  appearing in (12) are given by

$$A_k = \frac{1}{2k-1} \frac{(2k)!}{2^{2k}(k!)^2} \frac{1}{R^{2k-1}}, \quad k = 1, 2, \dots \quad (17)$$

The corresponding coefficients  $B_k$  defining the solution (13) for the penetration function  $\omega(a)$  are then obtained using (14).

### Frictionless indentation by a sphere

For the case of frictionless indentation by a flat cylindrical punch, it is known from Sneddon (1965) that the function  $\Psi$  is given by

$$\Psi(\rho) = \frac{2}{\pi} \sin^{-1} \frac{1}{\rho}, \quad \rho \geq 1. \quad (18)$$

On substituting (18) in (14) and making use of (17) it can be shown that

$$B_k = \frac{2^{2k}(k!)^2}{(2k)!} A_k = \frac{1}{2k-1} \frac{1}{R^{2k-1}}, \quad k = 1, 2, \dots \quad (19)$$

The substitution of (19) in (13) leads to the following expression for the penetration as a function of the contact radius  $a$

$$\omega(a) = R \sum_{k=1}^{\infty} \frac{1}{2k-1} \left(\frac{a}{R}\right)^{2k} = \frac{a}{2} \ln \frac{R+a}{R-a}, \quad a < R. \quad (20)$$

On substituting (2) and (20) into (7) it can be shown that the applied load as a function of contact radius is given by

$$L(a) = \frac{2Ea^2}{1-\nu^2} \sum_{k=1}^{\infty} \frac{2k}{(2k-1)(2k+1)} \left(\frac{a}{R}\right)^{2k-1} = \frac{E}{2(1-\nu^2)} \left[ (a^2 + R^2) \ln \frac{R+a}{R-a} - 2Ra \right], \quad a < R. \quad (21)$$

The result (20) for frictionless spherical contact is identical to that derived using a different method of analysis developed by Sneddon (1965) (see equation (6.13)). Note that Sneddon's equation (6.15) for the applied load has a misprint. The result (21) is the correct expression for the applied load and can be derived from Sneddon's equations (4.1), (6.13) and (6.14). The superposition technique used here is thus consistent with the classical method of solution.

It will now be shown that the analysis described above is consistent with the well known Hertz solution. When the contact radius  $a$  is small, it follows from (12-14) and (16) that

$$f(r) \cong \frac{r^2}{2R} , \quad \omega(a) \cong \frac{a^2}{R} . \quad (22)$$

It then follows from (2) that the applied load  $L$  is given by

$$L \cong \frac{4E}{3(1-\nu^2)} \frac{a^3}{R} , \quad (23)$$

and that the load-penetration relation is given by

$$L \cong \frac{4}{3} \frac{E R^{\frac{1}{2}}}{1-\nu^2} w^{\frac{3}{2}} . \quad (24)$$

### Numerical solution for coated half-spaces

On setting  $t = ru$  the integral equation (10) may be written in the form

$$f(r) = \omega(r) - \int_0^1 \omega'(ru) \Phi\left(\frac{1}{u}, \frac{t_c}{ru}\right) r du . \quad (25)$$

Solutions are now sought of the form

$$\omega(a) = \sum_{k=1}^n B_k a^{2k} . \quad (26)$$

On substituting (26) in (25) it can be shown that

$$\sum_{k=1}^n B_k r^{2k} [1 - \psi_k(r)] = f(r) , \quad r \geq 0 , \quad (27)$$

where

$$\psi_k(r) = 2k \int_0^1 u^{2k-1} \Phi\left(\frac{1}{u}, \frac{t_c}{ru}\right) du , \quad k = 1 \dots n . \quad (28)$$

A collocation technique is used to solve the equation (27) for the coefficients  $B_k$ ,  $k = 1 \dots n$ , by satisfying the equation at a discrete set of points  $r_1, r_2, \dots, r_n$  defined below. These coefficients are determined by solving the following set of linear algebraic equations using standard methods

$$\sum_{k=1}^n B_k r_p^{2k} [1 - \psi_k(r_p)] = f(r_p) \quad , \quad p = 1 \dots n \quad . \quad (29)$$

The quantities  $\psi_k(r_p)$  ,  $k, p = 1 \dots n$  , are estimated from (28) using the trapezoidal integration rule as follows

$$\psi_k(r_p) = k \sum_{i=1}^N h_i \left[ u_{i-1}^{2k-1} \Phi\left(\frac{1}{u_{i-1}}, \frac{t_c}{r_p u_{i-1}}\right) + u_i^{2k-1} \Phi\left(\frac{1}{u_i}, \frac{t_c}{r_p u_i}\right) \right] , \quad k, p = 1 \dots n \quad , \quad (30)$$

where  $u_i$ ,  $i = 0 \dots N$ , is a monotonic increasing sequence of values of  $u$  (set by the results of FEA) such that

$$u_0 = 0 , \quad u_N = 1 , \quad h_i = u_i - u_{i-1} , \quad i = 1 \dots N \quad . \quad (31)$$

It now only remains to calculate from (7) the corresponding values of the applied load. On substituting (26) in (7) it can be shown that

$$L(a) = \sum_{k=1}^n B_k a^{2k+1} g_k\left(\frac{t_c}{a}\right) , \quad (32)$$

where

$$g_k(x) = 2k \int_0^1 v^{2k} \alpha\left(\frac{x}{v}\right) dv \quad . \quad (33)$$

The functions  $g_k(x)$  ,  $k = 1 \dots n$  , are calculated from (33) using the trapezoidal integration rule as follows

$$g_k(x) = k \sum_{i=1}^M H_i \left[ v_{i-1}^{2k} \alpha\left(\frac{x}{v_{i-1}}\right) + v_i^{2k} \alpha\left(\frac{x}{v_i}\right) \right] , \quad k = 1 \dots n \quad , \quad (34)$$

where

$$v_i = \frac{i}{M} , \quad H_i = \frac{1}{M} , \quad i = 0 \dots M \quad . \quad (35)$$

The parameter  $M$  is selected to have the value 1000. The functions  $\Phi(r/a, t_c/a)$  and  $\alpha(t_c/a)$  are obtained from finite element analysis (using a flat cylindrical punch of radius 5  $\mu\text{m}$ ) and their values are known only at a number of discrete points. A linear interpolation technique is used to estimate the values of these functions at the points required by the relations (30) and (34). When providing values from FEA for the function  $\alpha(x)$  it is vital that the asymptotic limiting value of the function as  $x \rightarrow \infty$  is specified for two values of  $x$ , namely  $x = 20$  and a very large value of  $x$ . Failure to do this results in the discrete model being unable to predict the correct result when the coating thickness is very large.

### **Solution for a soft coating on a hard substrate**

Consider the soft coating having a Young's modulus of 70 GPa and a Poisson's ratio of 0.33 that is perfectly bonded to the hard substrate having a Young's modulus of 200 GPa and a Poisson's ratio of 0.3. The finite element results for a rigid flat cylindrical punch indenting a soft coating on a hard substrate for a variety of coating thicknesses are shown in Figs. 3 and 4.

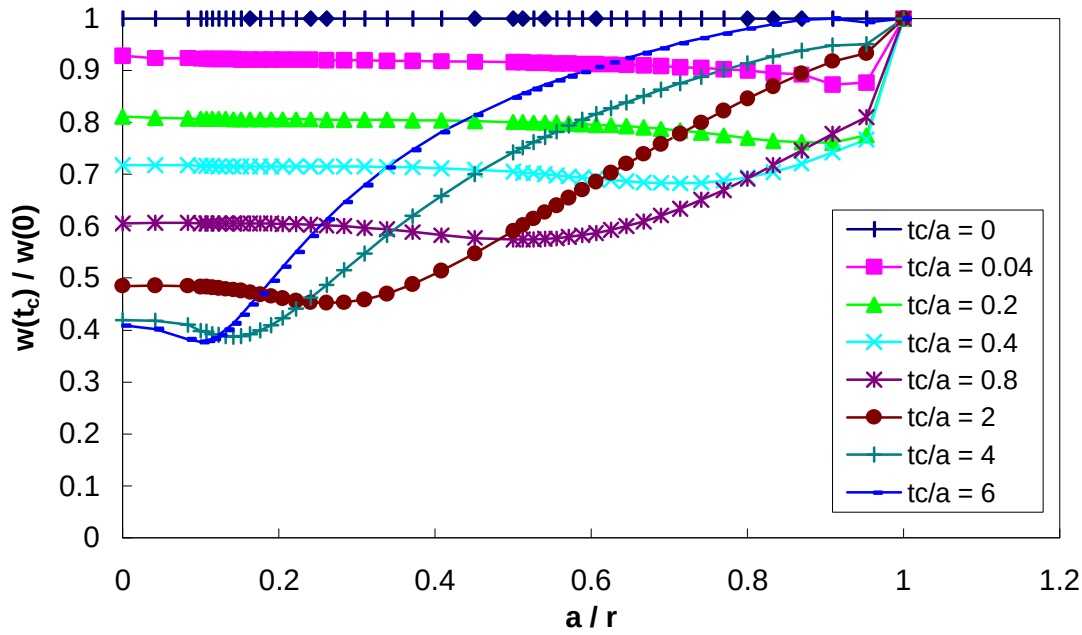


Fig.3: FEA results for the indentation of a soft coating bonded to a hard substrate by a rigid flat cylindrical punch.

In Fig.3 the normalised axial displacement, written  $w(t_c) / w(0)$ , is defined by  $\Phi(r/a, t_c/a) / \Phi(r/a, 0)$  and this ratio is plotted as a function of  $a/r$  rather than  $r/a$ . The quantity  $\Phi(r/a, t_c/a)$  is obtained from FEA for prescribed values of  $h/a$  while the quantity  $\Phi(r/a, 0)$  is calculated using the Sneddon (1965) solution  $\Phi = \Psi(r/a)$  given by (18). In Fig.4 the quantity  $\alpha(t_c/a) / \alpha(0)$  is plotted as a function of  $t_c/a$  where the quantity  $\alpha(t_c/a)$  defined by (1) is obtained from FEA while the value of  $\alpha(0)$  is calculated using the Sneddon result (2). It should be noted that the results in Fig.3 as  $a/r \rightarrow 0$  correspond to those in given in Fig.4 (see Appendix 2 for further discussion).

On using the data given in Figs. 3 and 4, and the relation (15) for a spherical indenter, in conjunction with the analysis given in this section, it can be shown that the resulting load-penetration curves for three layer thicknesses having the values 0.2, 0.8 and 2.0  $\mu\text{m}$  have the form given in Fig.5. When making the predictions the following values were used :

$$n = 15, \quad r_p = p/5, \quad p = 1 \dots n, \quad a_j = 0.002j, \quad j = 1 \dots 385.$$

Also in Fig.5 is shown the result of a corresponding finite element analysis for the indentation of the coated substrate by a rigid spherical sphere of radius 10 microns. It is seen that there is very good agreement of the FEA results with the predictions of the method used here.

It is worth noting that when  $t_c = 0.2 \mu\text{m}$  the load predicted by the analysis is less than that predicted by FEA. The opposite is found when  $t_c = 2 \mu\text{m}$ .

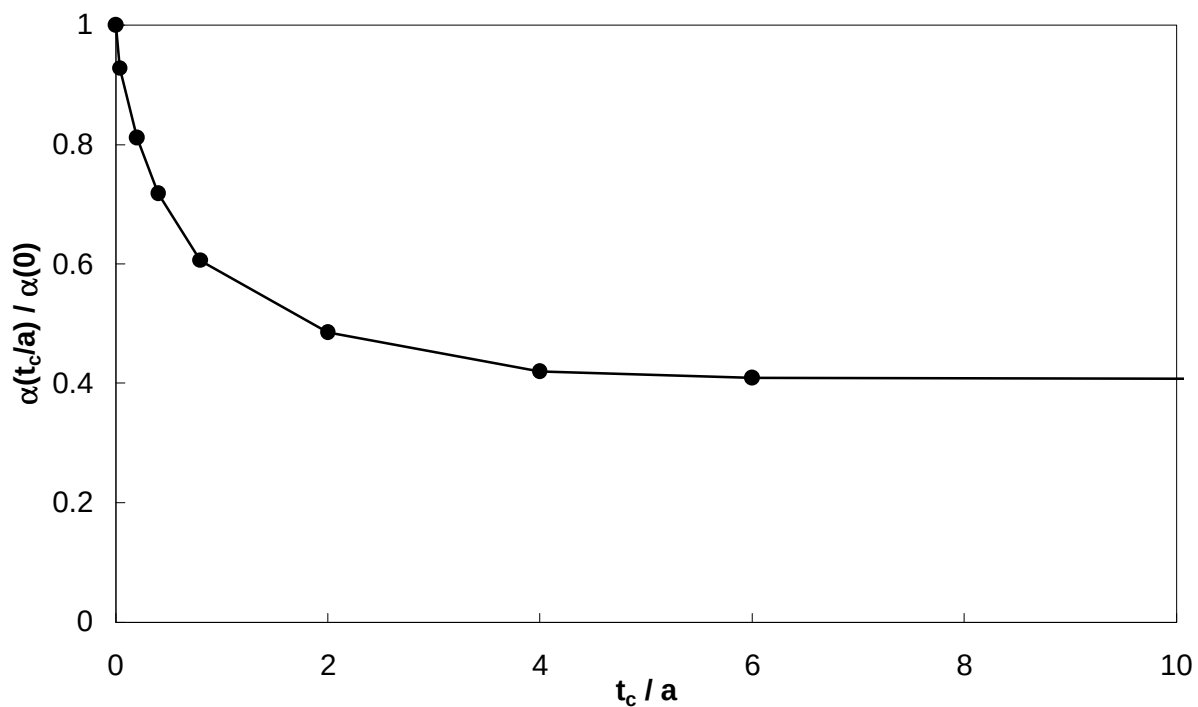


Fig.4: FEA results for applied load, as a function of  $t_c/a$ , for the function  $a$  relating to the indentation by a rigid flat cylindrical punch of radius 5 mm into a soft coating bonded to a hard substrate.

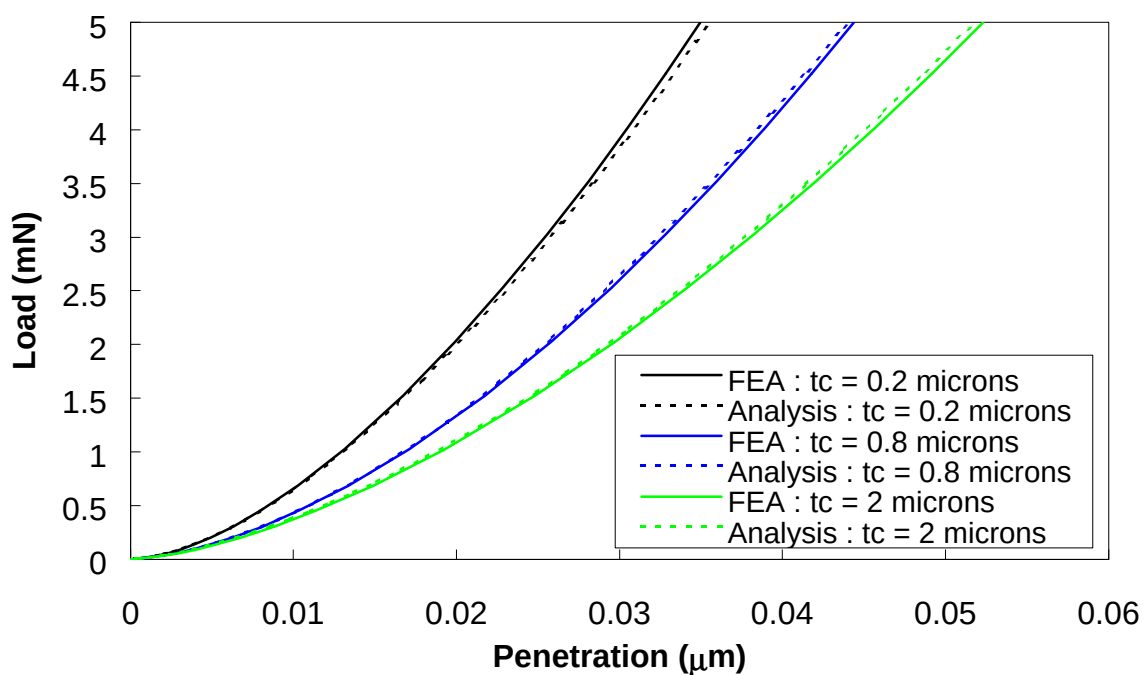


Fig.5: Comparison of FEA results with those of the analysis for the indentation, by a rigid sphere of radius 10 microns, of soft coatings having three different thicknesses bonded to a hard substrate.

### Solution for a hard coating on a soft substrate

Consider now the reversed situation where the hard coating having a Young's modulus of 200 GPa and a Poisson's ratio of 0.3 which is perfectly bonded to the soft substrate having a Young's modulus of 70 GPa and a Poisson's ratio of 0.33. The finite element results for a rigid flat punch indenting a hard coating on a soft substrate for a variety of thicknesses are shown in Figs. 6 and 7. In Fig.6 the normalised axial displacement defined by  $\Phi(r/a, t_c/a) / \Phi(r/a, 0)$  is plotted as a function of  $a/r$ . The quantity  $\Phi(r/a, t_c/a)$  is obtained from FEA while the quantity  $\Phi(r/a, 0)$  is calculated using the solution given by (18). In Fig.7 the quantity  $\alpha(t_c/a) / \alpha(0)$  is plotted as a function of  $h/a$  where the quantity  $\alpha(t_c/a)$  is obtained from FEA while the value of  $\alpha(0)$  is calculated using the result (2) applied to the substrate material. Again it should be noted that the results in Fig.6 as  $a/r \rightarrow 0$  correspond to those in given in Fig.7 (see Appendix 2 for further discussion).

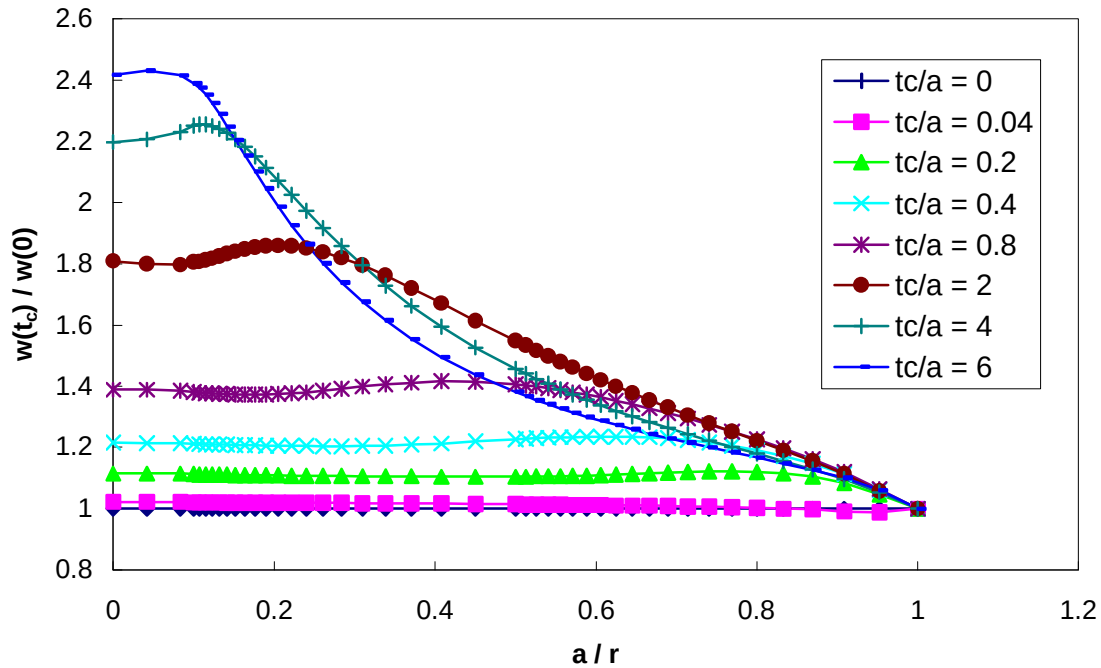


Fig.6 : FEA results for the indentation of a hard coating bonded to a soft substrate by a rigid flat cylindrical punch.

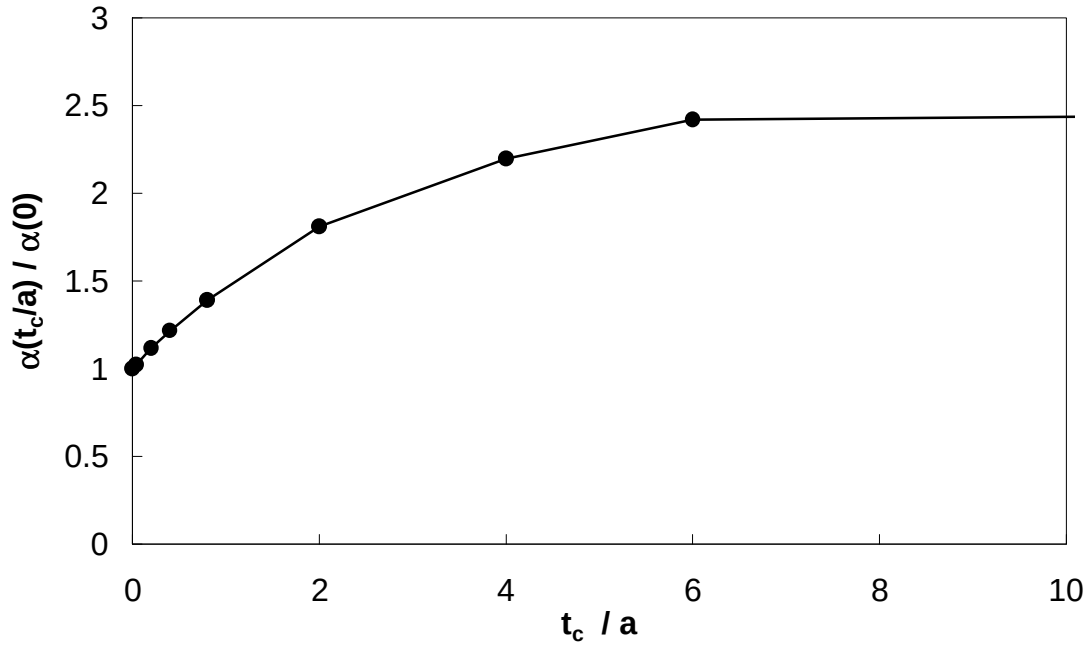


Fig.7: FEA results, as a function of  $t_c/a$ , for the function  $\alpha$  relating to the indentation by a rigid flat cylindrical punch of radius 5 mm into a hard coating bonded to a soft substrate.

On using the data given in Figs. 6 and 7, and the relation (15) for a spherical indenter, in conjunction with the analysis given in this section, it can be shown that the resulting load-penetration curves for three layer thicknesses having the values 0.2, 0.8 and 2.0  $\mu\text{m}$  have the form given in Fig.8. When making the prediction the following values were used:

$$n = 15, \quad r_p = p/5, \quad p = 1 \dots n, \quad a_j = 0.002j, \quad j = 1 \dots 350.$$

Also in Fig.8 is shown the result of a corresponding finite element analysis for the indentation of the coated substrate by a rigid spherical sphere of radius 10 microns. It is seen that there is very good agreement of the FEA results with the predictions of the method used here.

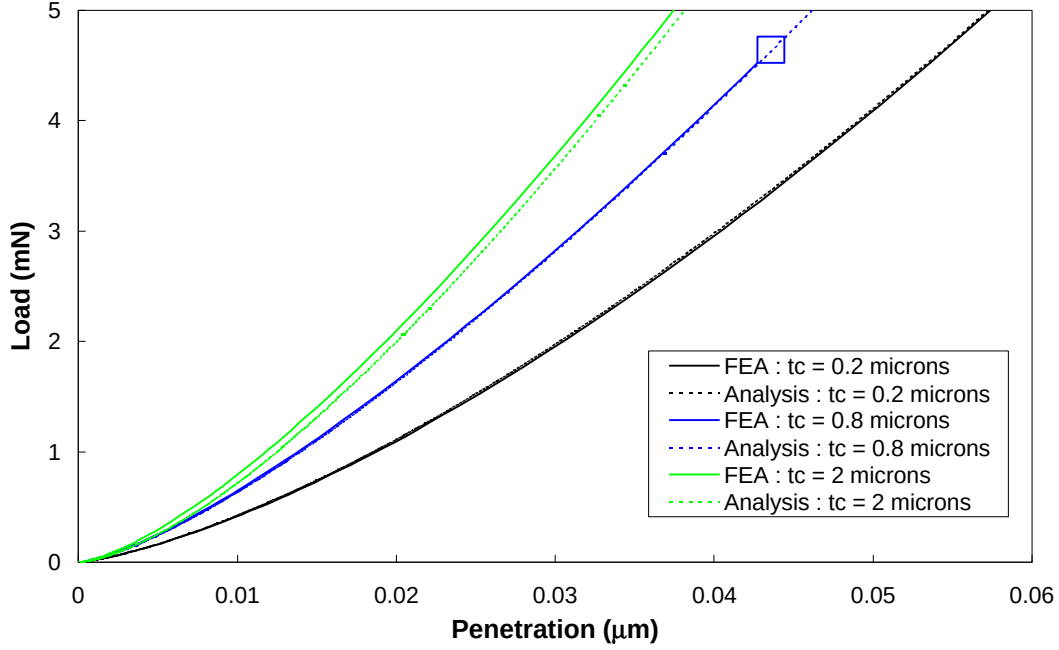


*Fig.8 : Comparison of FEA results with those of the analysis for the indentation, by a rigid sphere of radius 10 microns, of hard coatings having three different thicknesses bonded to a soft substrate.*

It is worth noting that when  $t_c = 0.2 \mu\text{m}$  the load predicted by the analysis is less than that predicted by FEA. When  $t_c = 0.8 \mu\text{m}$  and  $t_c = 2 \mu\text{m}$  the results of analysis and FEA are almost identical.

### Determination of the effective modulus of the coating

The principal objective of the analysis presented is to develop a procedure that enables load-penetration data for any rigid axi-symmetric indenter to be used to estimate the Young's modulus of a coating applied to a semi-infinite substrate. The results of the procedure must be independent of both the indenter tip curvature and the coating thickness  $t_c > 0$ . If such properties are not achieved then the results of the procedure will not be an estimate of the properties of the coating and substrate alone.

Before considering this aspect it is worth noting that the differentiation of the result (7) leads directly to the following well-known relation that defines the effective indentation modulus of the coated system

$$E_{IT} = \frac{1}{2} \alpha \left( \frac{t_c}{a} \right) = \frac{1}{2a \omega'(a)} \frac{dL}{da} \equiv \frac{1}{2} \sqrt{\frac{\pi}{A_p}} \frac{dL}{d\omega}, \quad (36)$$

where  $A_p = \pi a^2$  is the projected contact area.

It is of interest to investigate the properties of the load-penetration curve for small values of the contact area, hence for low values of the applied load. From (7) as  $a \rightarrow 0$  it follows that

$$L(a) \approx \alpha(\infty) \int_0^a t \omega'(t) dt . \quad (37)$$

On substituting (13) into (36) it is easily shown that as  $a \rightarrow 0$

$$L(a) \approx \frac{2}{3} \alpha(\infty) B_1 a^3 \quad \text{with} \quad \omega(a) \approx B_1 a^2 . \quad (38)$$

As the value of  $B_1$  is found as part of the solution of the linear equations (29), its value can be inserted into (38) followed by the elimination of the contact radius  $a$  to derive the following expression for the value of the effective modulus of the coating/substrate system defined by

$$E_{IT} = \frac{\sqrt{\pi}}{2} \frac{dL}{d\omega} \frac{1}{\sqrt{A_c}} = \frac{1}{2} \alpha(\infty) = \lim_{a \rightarrow 0} \frac{Lt}{4a\omega} = \lim_{\omega \rightarrow 0} \frac{3\sqrt{B_1} L(\omega)}{4\omega^{3/2}} , \quad (39)$$

where the applied load  $L$  is now regarded as a function of the penetration  $\omega$  and where  $A_c = \pi a^2$  is the contact area. When  $t_c = 0$  so that the coating is totally absent the value of  $E_{IT}$  is given by

$$E_{IT} = \frac{E_s}{1 - \nu_s^2} . \quad (40)$$

When  $t_c > 0$  is not too small the value of  $E_{IT}$  predicted by (39) is given by

$$E_{IT} = \frac{E_c}{1 - \nu_c^2} . \quad (41)$$

In general, the value of  $E_{IT}$  is independent of the coating thickness  $t_c$  (provided that  $t_c > 0$ ) and the indenter radius  $R$ , being only a function of the Young's moduli and Poisson's ratios of the coating and substrate. The value of  $E_{IT}$ , when  $t_c$  is large enough, could be the basis of a method for determining the value of the Young's modulus of a coating from depth sensing equipment assuming that Poisson's ratio for the coating is known.

## Conclusions

1. The method of superposition of indentation solutions for rigid flat cylindrical punches leads to a convenient technique for analysing indentation problems for any axisymmetric shape.
2. The superposition technique, when applied to an uncoated substrate, leads to results that are consistent with the classical results derived by Sneddon (1965).
3. The hybrid method, involving both finite element analysis for flat cylindrical punches and an analytical technique, for predicting the load-penetration behaviour of a coated semi-infinite substrate has proved to be highly successful.

4. The method provides an accurate method of estimating the effective modulus  $E_{IT}$  of the coated system. The method of determining  $E_{IT}$  vs  $t_c/a$  is such that its value is independent of the coating thickness, indenter radius and other geometrical variations, implying that the parameter  $E_{IT}$  is a characteristic only of the properties of the coating and substrate material.
5. A reliable method, for extracting from load-penetration data, values of the Young's modulus and Poisson's ratio for the coating, needs to be developed.

### Reference

Sneddon, I. N. 'The relation between load and penetration in the axisymmetric Boussinesq problem for a punch of arbitrary profile', Int. J. Engng. Sci. 3, (1965), 47-57.

## APPENDIX 2 : Asymptotic behaviour

An examination of Figs.3 and 4 (Appendix 1), which are results for the case of a soft coating bonded to a hard substrate indented by a rigid cylindrical flat punch, suggests that

$$\lim_{r \rightarrow \infty} \frac{\Phi(r/a, t_c/a)}{\Phi(r/a, 0)} = \frac{\alpha(t_c/a)}{\alpha(0)} . \quad (1)$$

The results given in Figs. 6 and 7 (Appendix 1) for the case of a hard coating bonded to a soft substrate also support the validity of (1) which is now written in the form

$$\frac{\Phi(r/a, t_c/a)}{\alpha(t_c/a)} \approx \frac{\Phi(r/a, 0)}{\alpha(0)} \quad \text{as } r \rightarrow \infty . \quad (2)$$

An analysis will be given that indicates why such a relationship might be valid.

From (Appendix 1, 18) it follows that for a rigid cylindrical flat punch

$$\Phi(r/a, 0) \approx \frac{2a}{\pi r} \quad \text{as } r \rightarrow \infty , \quad (3)$$

and from (Appendix 1, 2) it follows that

$$\alpha(0) = \frac{2E_s}{1 - \nu_s^2} . \quad (4)$$

Thus

$$\frac{\Phi(r/a, t_c/a)}{\alpha(t_c/a)} \approx \frac{1 - \nu_s^2}{\pi E_s} \frac{a}{r} \quad \text{as } r \rightarrow \infty . \quad (5)$$

Assume for the moment that at large distances on the free surface from the cylindrical flat punch for a coating of finite thickness, the surface profile is asymptotic to that which arises when a homogeneous sample of substrate material is indented by a point load of the same magnitude as that applied to the coated sample. It follows from the Boussinesq solution that when a load  $L$  is applied, resulting in a penetration  $\omega$  by a rigid flat circular punch, the surface profile has the form

$$w = \omega \Phi(r/a, t_c/a) \approx \frac{(1 - \nu_s^2)L}{\pi E_s} \frac{1}{r} \quad \text{as } r \rightarrow \infty . \quad (6)$$

From (Appendix 1, 1)

$$\omega = \frac{L}{a \alpha(t_c/a)} , \quad (7)$$

so that on substituting in (6)

$$\frac{\Phi(r/a, t_c/a)}{\alpha(t_c/a)} \approx \frac{1 - \nu_s^2}{\pi E_s} \frac{a}{r} \quad \text{as } r \rightarrow \infty , \quad (8)$$

which is consistent with the relation (5) derived from an observation of the results in Figs.3, 4, 6 and 7 of Appendix 1. It is concluded, therefore, that the assumption is valid that the surface profile for a coated sample at large distances from the indenter is asymptotic to that arising for a homogeneous sample of substrate material subject to indentation by a point force having the same magnitude as that applied to a coated sample.