

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

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ABSTRACT

The objective of this report is to describe progress that has been made with the development of a methodology to estimate, from nano-indentation tests involving elastic-plastic deformation, 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 performance of finite element analysis (FEA) predictions of indentation into coated systems is described. A key issue has been the selection of appropriate plastic properties for use in the FEA. It is shown that the use of yield and work hardening properties derived for macroscopic testing (tension and compression) does not lead to realistic load-penetration curves, including unloading. It is concluded that the use of conventional FEA will require plasticity values derived from indentation tests, thus introducing an undesirable circularity into the predictive methodology.

For elastic-plastic axisymmetric deformations, the load-penetration curves predicted in the INDICOAT project, by FEA modelling, are generally in agreement when steps are taken to ensure that identical problems are analysed by contributing partners, thus indicating that the FEA packages ABAQUS and ANSYS models are producing results that appear to be consistent with one another.

When analysing FEA results to estimate the indentation modulus of the system following the conventional Oliver and Pharr analysis, which involves extracting the effective modulus from the unloading part of experimental elastic-plastic load penetration curves, it is found that results depend significantly on the maximum load, and that predictions of the indentation modulus can lie outside the range defined by values for coating and substrate. Thus, for elastic-plastic FEA analyses, the Oliver and Pharr data reduction method does not lead to indentation modulus values that are a characteristic only of the coating/substrate properties and the coating thickness.

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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. These applications require 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, e.g. [1]. For cracking in layered materials, analytical models have been developed to predict the critical stress, crack spacing and progressive crack formation, e.g. [2]. 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, e.g. [3]. Steady state delamination has been analysed for conditions where yielding occurs in the film or in the substrate, e.g. [4]. In addition, spallation has been identified as a major mode of failure in thermal barrier coatings e.g. [5]. 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 summarise the results of a 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 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 INDICOAT project [6]. No attempt has been made to account for thermal residual stresses that can be large in coated systems, and are likely to have a significant effect on indentation behaviour.

2. Type of modelling considered

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-plastic deformations, the use of infinitesimal deformation theory was thought to be adequate.

As part of the INDICOAT project [6], three organisations (JRC (Italy), KTH (Sweden) and NPL (UK)) have carried out the FEA of the various coating problems. KTH have used ANSYS to predict their results. 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. Appendix 1 provides the results of a simple test case that demonstrates that ABAQUS and ANSYS predict identical results.

3. Constitutive relations

When modelling the elastic response of coatings and substrates, it is assumed that the materials are isotropic so that their elastic properties are fully characterised by values of the Young's modulus E and Poisson's ratio ν such that the elastic strain ϵ_{ij}^E is the following linear function of the stress tensor σ_{ij}

$$\epsilon_{ij}^E = \frac{1}{E} \left[(1 + \nu) \sigma_{ij} - \nu \sigma_{kk} \delta_{ij} \right]. \quad (1)$$

When modelling the inelastic response of a metallic phase, it is assumed that the accumulated effective plastic strain $\bar{\epsilon}_p$ during monotonic loading is related to the effective stress $\bar{\sigma}$ as follows:

$$\bar{\epsilon}_p = \epsilon_0 \left(\frac{\bar{\sigma}}{\sigma_Y} - 1 \right)^n \quad \text{for } \sigma \geq \sigma_Y \quad (2)$$

$$\text{otherwise } \bar{\epsilon}_p = 0,$$

where ϵ_0 and n are material constants. Here σ_Y denotes the uniaxial yield stress and the effective stress $\bar{\sigma}$, a scalar, is defined by

$$\bar{\sigma} = \sqrt{\frac{3}{2} \sigma_{ij} \sigma_{ij}}, \quad (3)$$

where σ_{ij} is the deviatoric stress tensor defined by

$$\sigma_{ij} = \sigma_{ij} - \frac{1}{3} \sigma_{kk} \delta_{ij}. \quad (4)$$

The quantity $\bar{\epsilon}_p$ is the accumulated effective plastic strain during monotonic loading calculated from the incremental relation

$$d\bar{\epsilon}_p = \sqrt{\frac{2}{3}} d\epsilon_{ij}^p d\epsilon_{ij}^p. \quad (5)$$

The relation (2) may also be written in the form

$$\bar{\sigma} = \sigma_Y + \sigma_0 \bar{\epsilon}_p^m \quad \text{for } \sigma \geq \sigma_Y, \quad (6)$$

provided that

$$\epsilon_0 = \frac{\sigma_Y}{\sigma_0}^{\frac{1}{m}} \quad \text{and} \quad n = \frac{1}{m}. \quad (7)$$

It is also assumed that the inelastic response is isotropic and results in associated flow. Each participating organisation could choose how the above formulae were to be implemented in

the finite element analysis, as this selection depended on the hardware and software facilities available to participants. Values for the plasticity parameters σ_Y , ϵ_0 , σ_0 , n and m are specified in the next section, but they are not all independent.

4. Material properties

The following material properties are best estimates based on the very limited data that are available regarding elastic properties of coatings, and the elastic-plastic properties of the metallic substrates/coatings. Values have been obtained by comparing FEA predictions for various elastic-plastic models with the results of indentation tests into uncoated samples, and some coated samples. This was necessary as properties based on tension data from macroscopic tests were wholly inadequate for application to nanoindentation (see the Appendix 2 for discussion of unplanned FEA that was essential to attempt to resolve the problem of plasticity property selection).

Substrates

M2 steel :

Elastic constants: $E = 220.6 \text{ GPa}$ $\nu = 0.29$

Plasticity parameters: Assume ideal elastic-plastic with $\sigma_Y = 4.0 \text{ GPa}$.

Nickel :

Elastic constants: $E = 220 \text{ GPa}$ $\nu = 0.31$

Plasticity parameters:

CASE I (based on using a reasonable macroscopic yield stress):

$$\sigma_Y = 200 \text{ MPa}$$

$$\sigma_0 = 850 \text{ MPa} \quad m = 0.1$$

and / or

CASE II (for an ideal elastic-perfectly plastic solid):

$$\sigma_Y = 800 \text{ MPa}$$

BK7 :

Elastic constants: $E = 81.9 \text{ GPa}$
 $\nu = 0.203$

Coatings

Pure aluminium :

Elastic constants: $E = 69.8 \text{ GPa}$ $\nu = 0.34$

Plasticity parameters:

$$\sigma_Y = 28 \text{ MPa} \quad \sigma_0 = 64.982 \text{ MPa} \quad m = 0.3333$$

Sensitivity check: Ideal elastic-plastic with $\sigma_Y = 0.4 \text{ GPa}$

Gold :

Elastic constants: $E = 60.0 \text{ GPa}$

$$\nu = 0.44$$

Plasticity parameters: Ideal elastic-plastic with $\sigma_Y = 0.3 \text{ GPa}$.

DLC :

Elastic constants: $E = 137 \text{ GPa}$

$$\nu = 0.2$$

Plasticity parameters: Assume ideal elastic-plastic $\sigma_Y = 6 \text{ GPa}$

Alumina :

Elastic constants: $E = 160 \text{ GPa} \quad \nu = 0.17$

5. Substrate Geometry

The coatings and substrates are divided into “hard” and “soft” materials. There are obviously four possibilities:

- case 1 hard on hard
- case 2 hard on soft
- case 3 soft on hard
- case 4 soft on soft

The following systems, representing the above cases, have been analysed by FEA:

DLC on M2 tool steel - thicknesses (μm): 0.28, 1.3, 2.1

Alumina on Nickel - thicknesses (μm): 0.32, 0.915

Aluminium on BK7 - thicknesses (μm): 0.31, 1.05, 3.4

Gold on Nickel - thicknesses (μm): 0.3, 1.19, 5.12

6. Indenter tip geometry

Spherical indenters have been considered having a radius of 10 μm . Where conical, Berkovich and other sharp indenters are investigated, it was suggested that the indenter tip could be taken as a sphere having a radius of 1 μm . Model predictions were, in the first instance, carried out for a rigid indenter. KTH modelled the indenter with a very high modulus material in order to simulate a rigid indenter. The properties assumed for the ‘rigid’ indenter are $E = 1.06 \times 10^7 \text{ GPa}$, $\nu = 0.07$.

7. Boundary loading conditions

For the present case, where only finite element solutions are considered, the boundary loading conditions may be expressed as follows:

1. The force driving the indenter into the substrate should be increased slowly from zero to maximum specified load (see Table 1) and then unloaded. In this context ‘slowly’ indicates that the solution required is quasi-static, i.e. it is free from all dynamic effects.
2. The reaction force necessary to maintain equilibrium is generated by requiring that the substrate boundary at infinity remains fixed as the indenter force increases. Clearly the finite element model cannot be infinite but the effect of fixing such a boundary at infinity can be reproduced, either by applying the results of an analytical solution on a finite boundary or by the use of ‘infinite’ elements. Any other method of applying this condition could be used by participants provided that it could be shown to be equivalent to the required condition.

Due to the changing contact area between the indenter and the substrate as the indenter force increases, the problem is always geometrically non-linear and a linear elastic perturbation solution does not exist. Nevertheless the solution within the elastic material may be obtained using finite or infinitesimal displacement theory.

All elastic/plastic solutions used finite deformation theory. When considering the elastic regime of deformation [7], some comparisons with infinitesimal theory were made. The effect was found close in magnitude to the effect of friction but opposite in sign so that the effects tend to cancel to some extent. It was concluded that the effect of finite deformations would be small in any loading regime where pure elastic deformation is likely.

8. Maximum loads

The maximum loads for each FEA simulation correspond to one of the maximum loads specified by the INDICOAT Experimental Matrix that are given in Table 1. For FEA simulations the loading stage was always followed by an unloading stage to zero load. The experiments include a hold time at maximum load. Such a hold is not relevant to the modelling as the materials models that participants have used do not have a time-dependence.

Table 1 : Maximum loads used for modelling and experiments

Maximum Load [mN]							No. Expts. per thickness
0.1	0.3	1.0	3.0	10.0	30.0	100.0	
(Soft)							2
	(Soft)	Soft		(All)			8
		All					4
		Soft	All	All		Hard	12
				All			4
		(Soft)		All	Hard	Hard	10
						Hard	2
		(Soft)		(All)		Hard	8

“Soft” = Soft coatings only

“Hard” = Hard coatings only

“All” = All coatings

9. Results

Comparison of load-penetration curves

In Figs. 1-11 are shown the results of comparing the FEA predictions of load-penetration (during loading and unloading to zero load) made by JRC, KTH and NPL for the coating systems specified in section 4 and the maximum loads specified in Table 1. Multiple loading plots are shown corresponding to the specified maximum loads. The results presented are intended to show either agreement between partners, when that is expected, and differences that can be ascribed to different assumptions of material properties so that sensitivity issues can be addressed.

In Figs.1-3 are shown the FEA predictions of load-penetration behaviour for aluminium coatings applied to a BK7 substrate (a soft-on-hard combination). The coating thicknesses considered are 0.31 μm , 1.05 μm and 3.4 μm . Fig.1 indicates that the predictions of JRC and NPL are in reasonable agreement as both partners used the same material properties. In particular, the yield stress used was 0.4 GPa that is very high for macroscopic yield behaviour, but thought to be reasonable for nano-indentation modelling. Differences between the JRC and NPL results can be ascribed to differences in the meshes used for the analysis. The loading part of the curve shows that, at loads in excess of 5 mN, the loading curve shows scatter associated with the locations chosen for mesh points. The KTH predictions assumed a yield stress of 28 MPa that is typical of bulk aluminium, and values $\sigma_0 = 64.982 \text{ MPa}$ and $m = 0.3333$ for the parameters appearing in the work hardening law specified by (6). Clearly the yield stress has a significant effect on load-penetration behaviour. This is not surprising as the aluminium is in direct contact with the indenter. Figs.2 and 3 show the results on the same system for coating thicknesses of 1.05 μm and 3.4 μm . These results indicate very good agreement between the results of JRC and NPL (to be expected), and the results of KTH again emphasise the critical importance of the value of the yield stress.

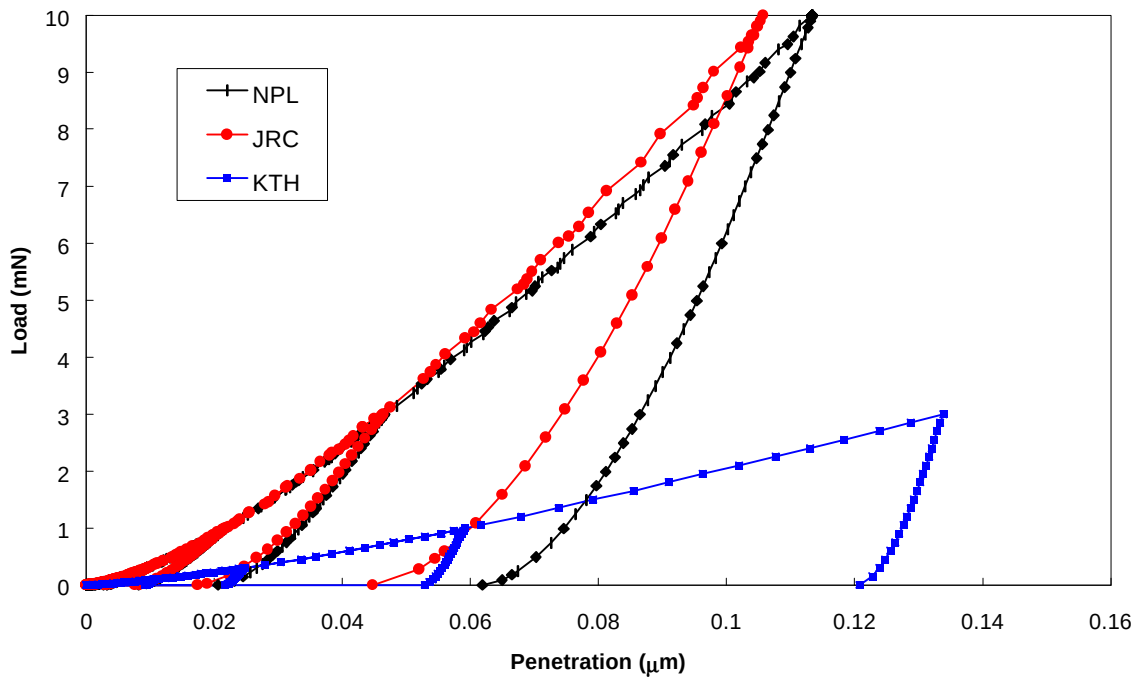


Fig.1 : 0.31 mm aluminium coating on a BK7 substrate.

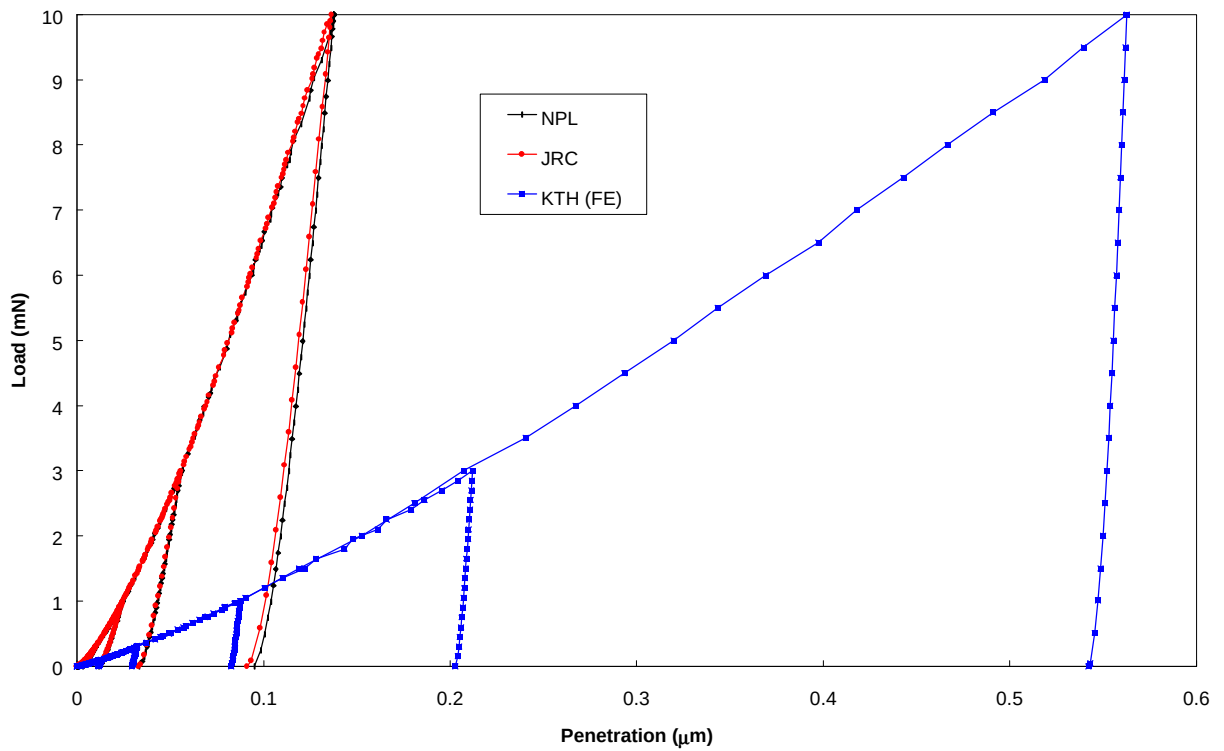


Fig.2 : 1.05 mm aluminium coating on a BK7 substrate.

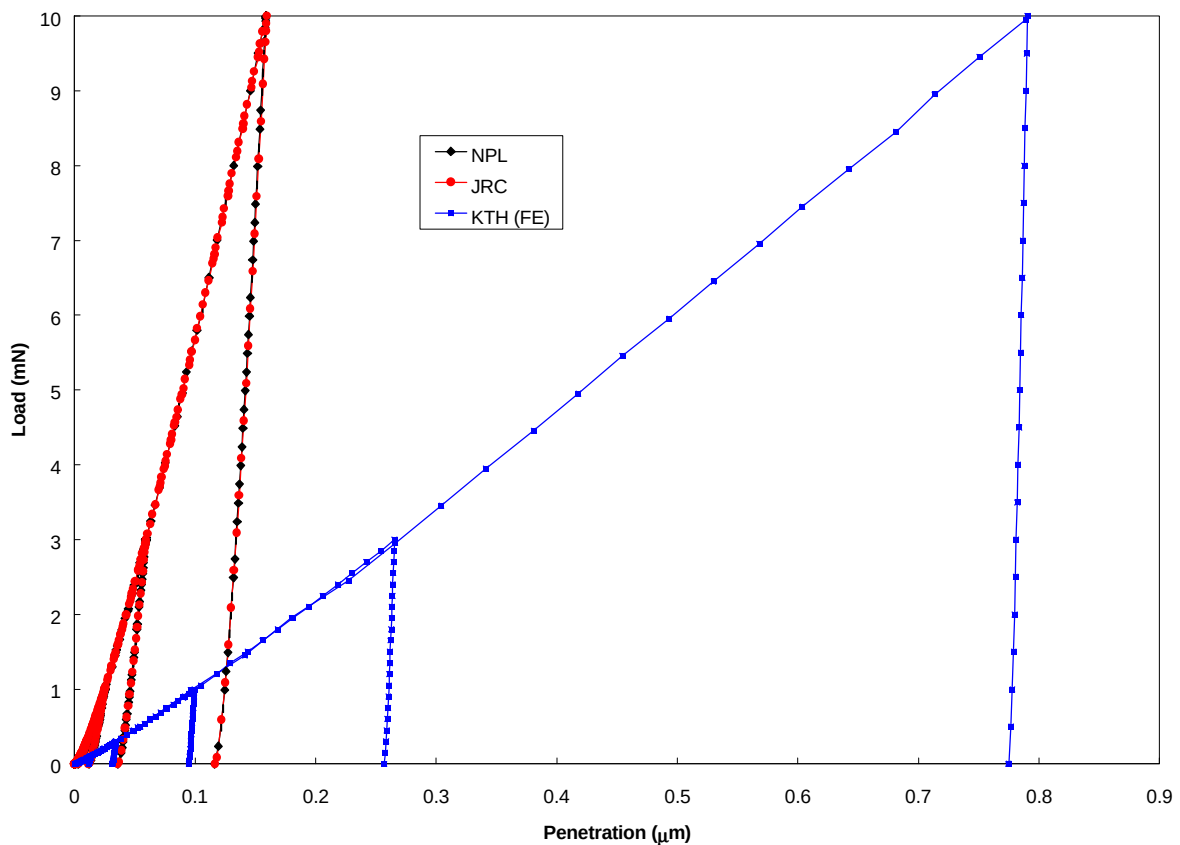


Fig.3 : 3.4 mm aluminium coating on a BK7 substrate.

In Figs. 4 and 5 are shown the FEA predictions of load-penetration behaviour for alumina coatings applied to a nickel substrate (a hard-on-soft combination). The coating thicknesses considered are $0.32\ \mu\text{m}$ and $0.915\ \mu\text{m}$. These results indicate that the predictions of JRC, KTH and NPL are in very good agreement as all partners used the same material properties. Differences between the JRC, NPL and KTH results are thought to arise from differences in the finite element meshes. It should be noted that there is much closer agreement between the results of JRC and NPL when the hard coating is thin (see Fig. 4).

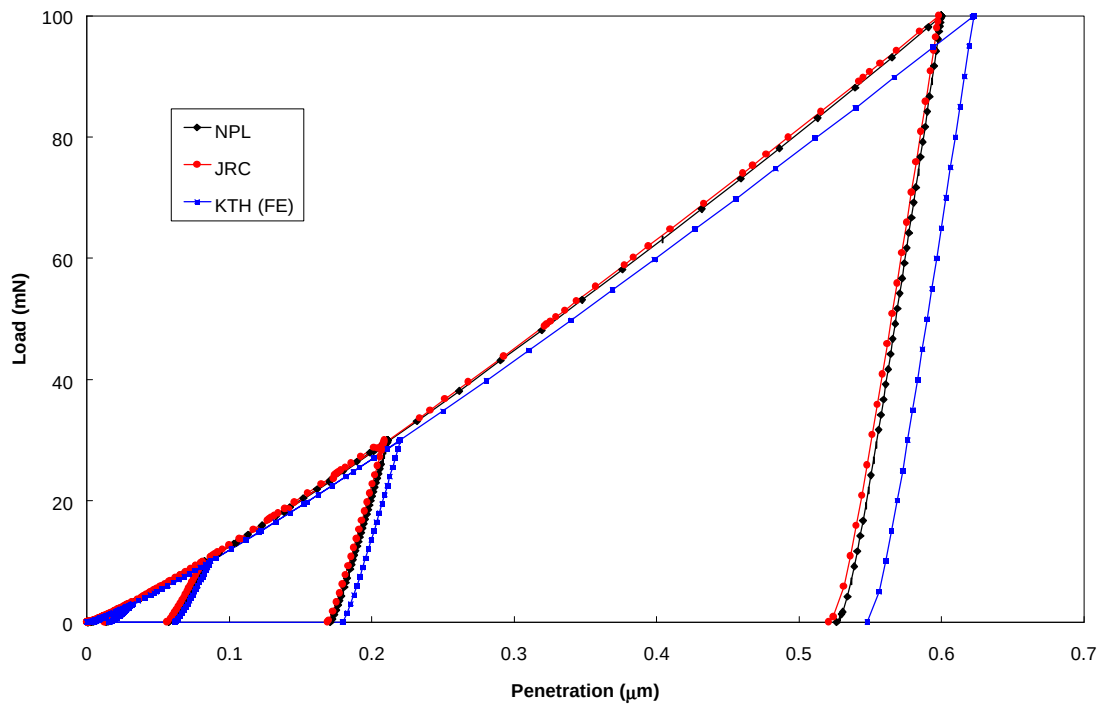


Fig.4 : 0.3 mm alumina coating on a nickel substrate.

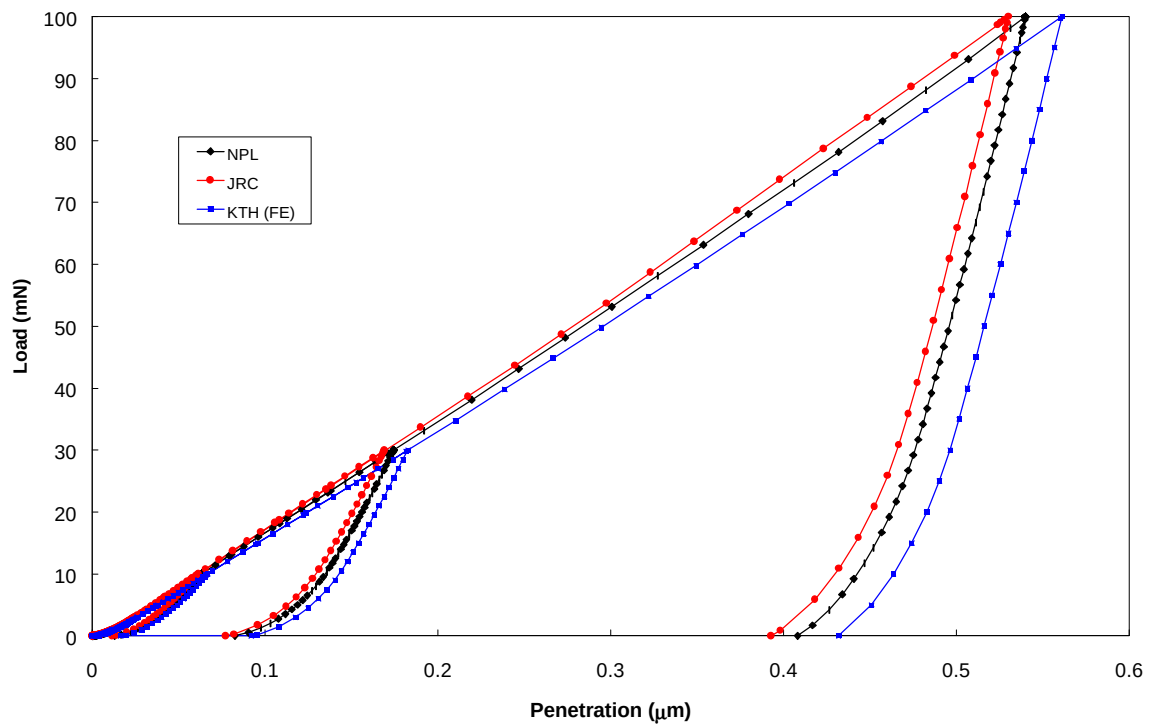


Fig.5 : 0.915 mm alumina coating on a nickel substrate.

In Figs. 6-8 are shown the FEA predictions of load-penetration behaviour for DLC coatings applied to a M2 steel substrate (a hard-on-hard combination). The coating thicknesses considered are $0.3\text{ }\mu\text{m}$, $1.19\text{ }\mu\text{m}$ and $5.12\text{ }\mu\text{m}$. These results indicate that the predictions of JRC, KTH and NPL are in good agreement for all coating thicknesses considered as all partners used the same material properties. Differences between the JRC, KTH and NPL results are again thought to arise from differences in the modelling of the finite element meshes. For the results shown in Fig.6, the JRC results show non-smooth load increases at loads above 3 mN probably arising from the use of a coarser mesh than those used by KTH and NPL.

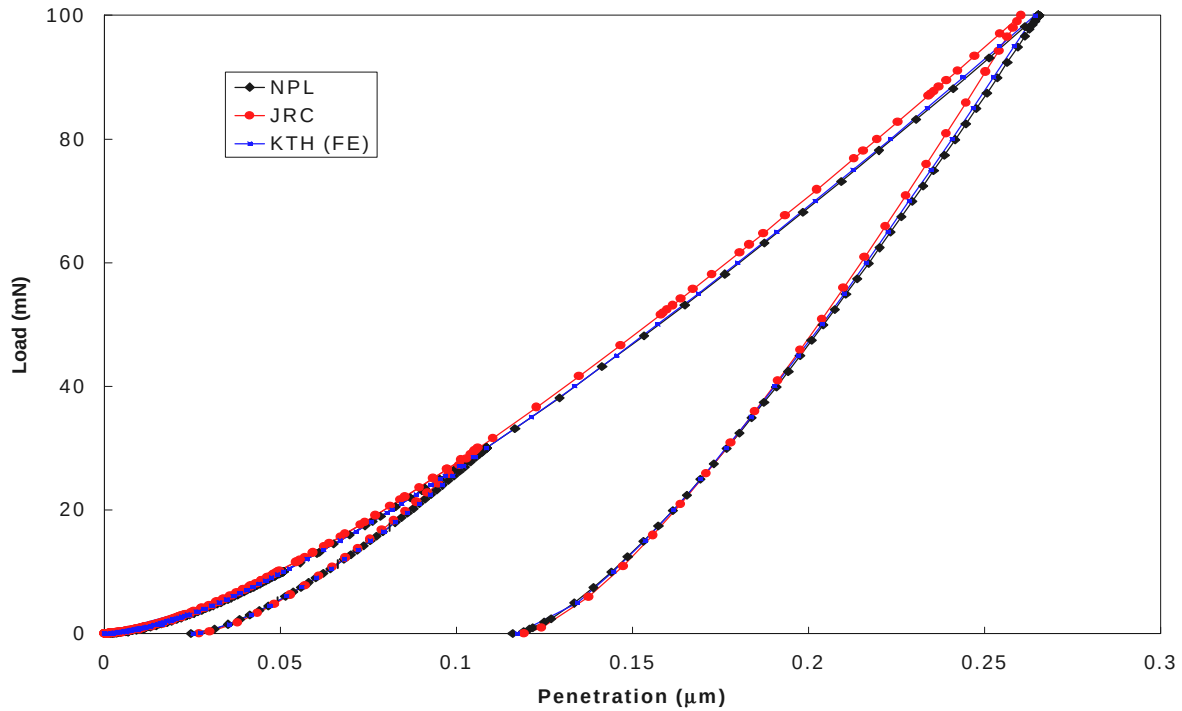


Fig.6 : 0.28 mm DLC on M2 toolsteel substrate.

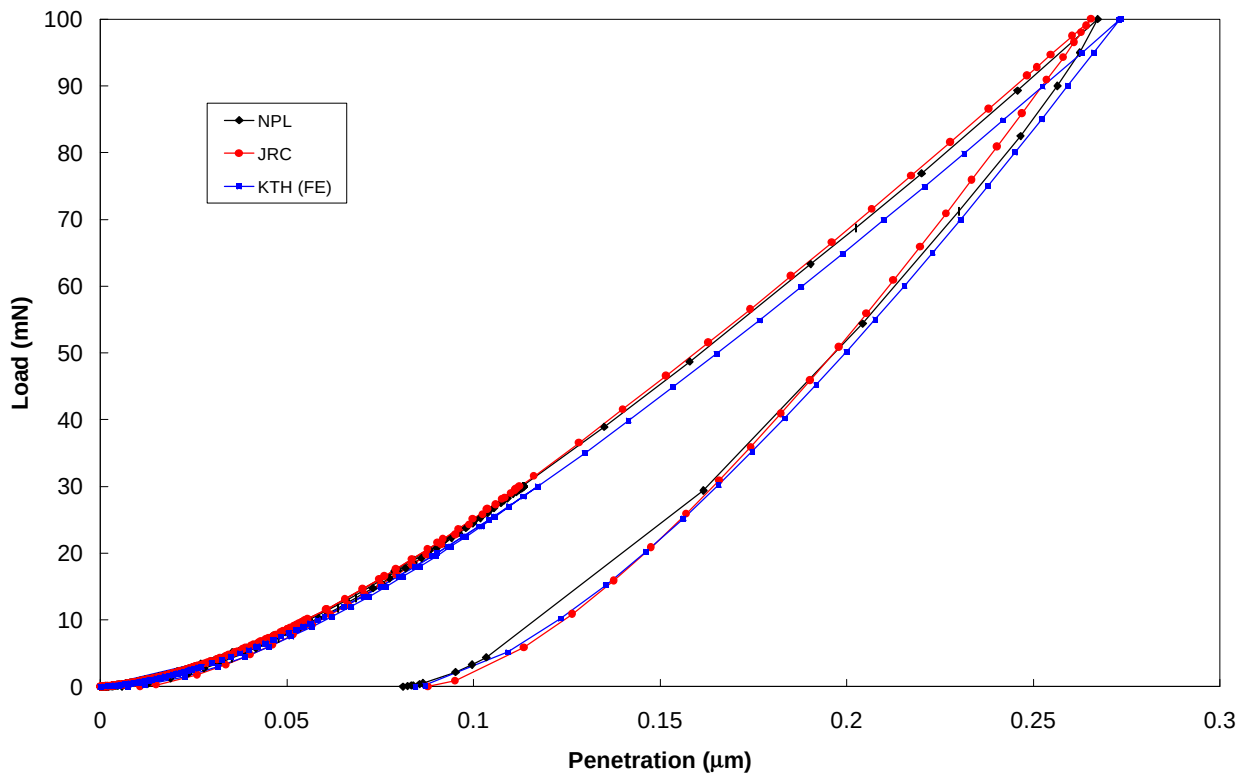


Fig.7 : 1.3 mm DLC on M2 Toolsteel substrate.

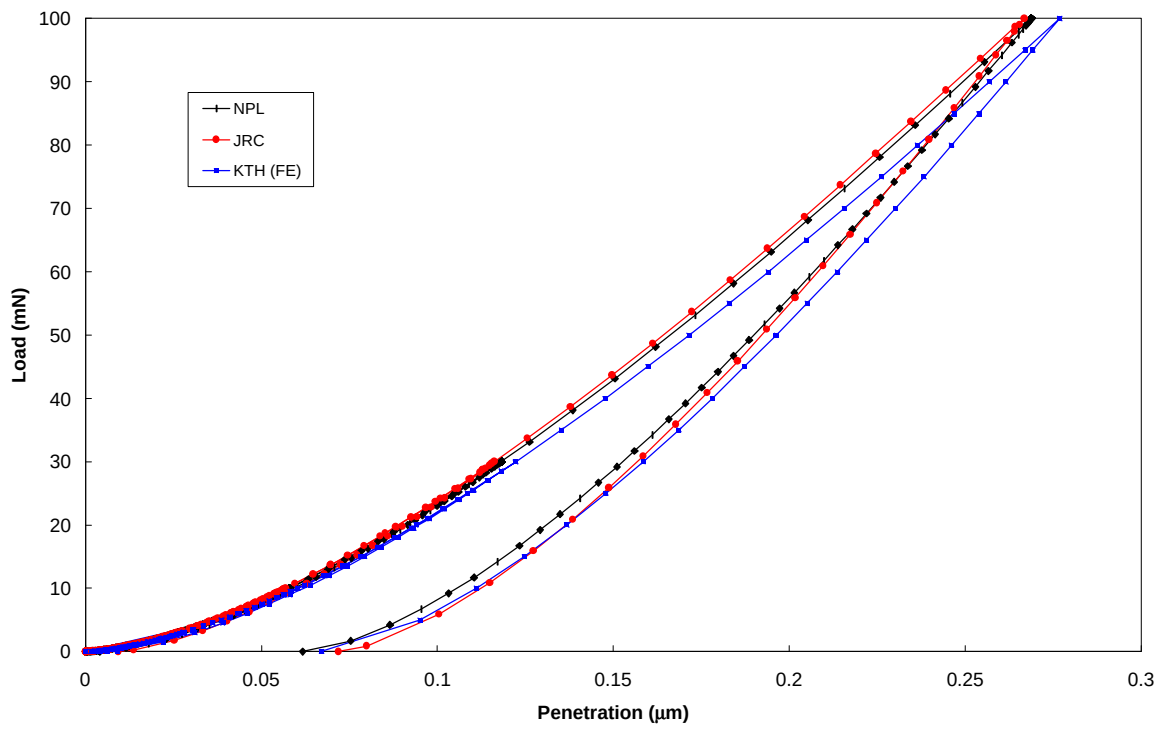


Fig.8 : 2.1 mm DLC on M2 Toolsteel substrate.

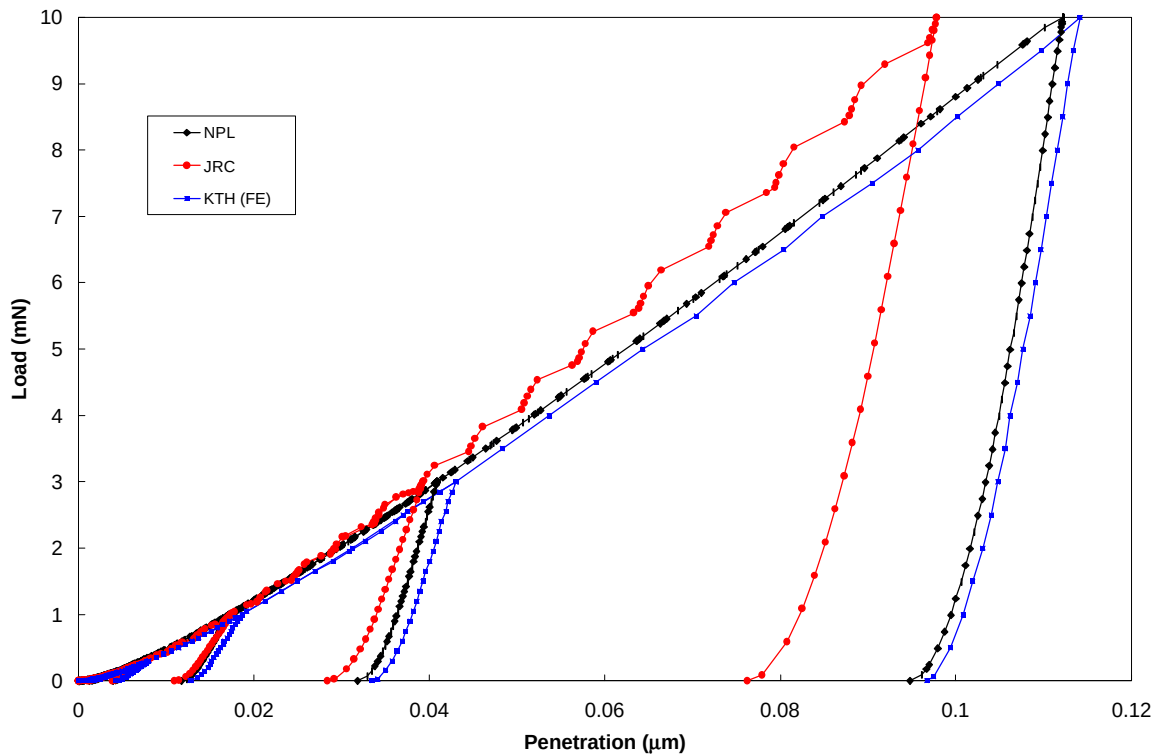


Fig.9 : 0.3 mm gold coating on a nickel substrate.

In Figs.9-11 are shown the FEA predictions of load-penetration behaviour for gold coatings applied to a nickel substrate (a soft-on-soft combination). The coating thicknesses considered are 0.3 μm , 1.19 μm and 5.12 μm . These results indicate that there are differences in the predictions of JRC, KTH and NPL even though all partners used the same material properties. In Fig.9 for the 0.3 μm coating there is very good agreement between KTH and NPL. The JRC results indicate a non-smooth loading curve that might indicate that a more refined mesh is needed for this case. In Fig.10 for the 1.19 μm coating the results are much more in agreement. The results of NPL and JRC show a similar non-smooth loading curve. For the thickest coating of 5.12 μm the results of JRC and KTH are in very good agreement, while the same non-smoothness of the load curve is apparent in the results of JRC and NPL. The soft-on-soft case is clearly leading to some unexpected variations in the predictions of the three partners. It would appear the softer the system the more variable the response. The next worst case is that of an aluminium coating. Also a very thick 5.12 μm coating can lead to meshing problems.

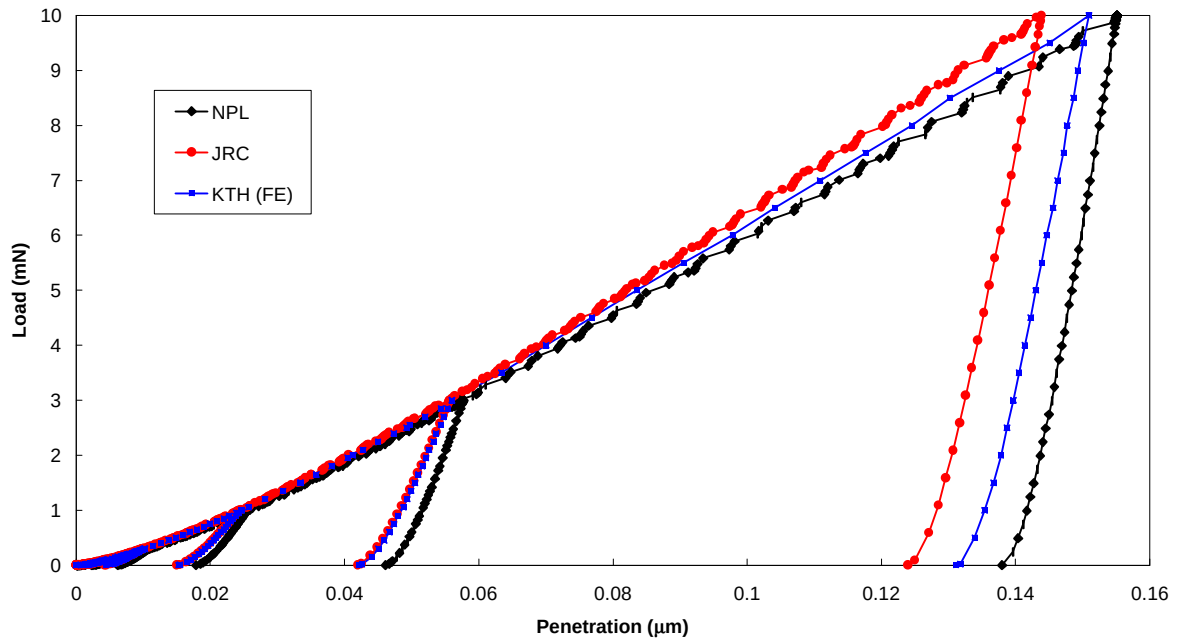


Fig.10 : 1.19 mm gold coating on a nickel substrate.

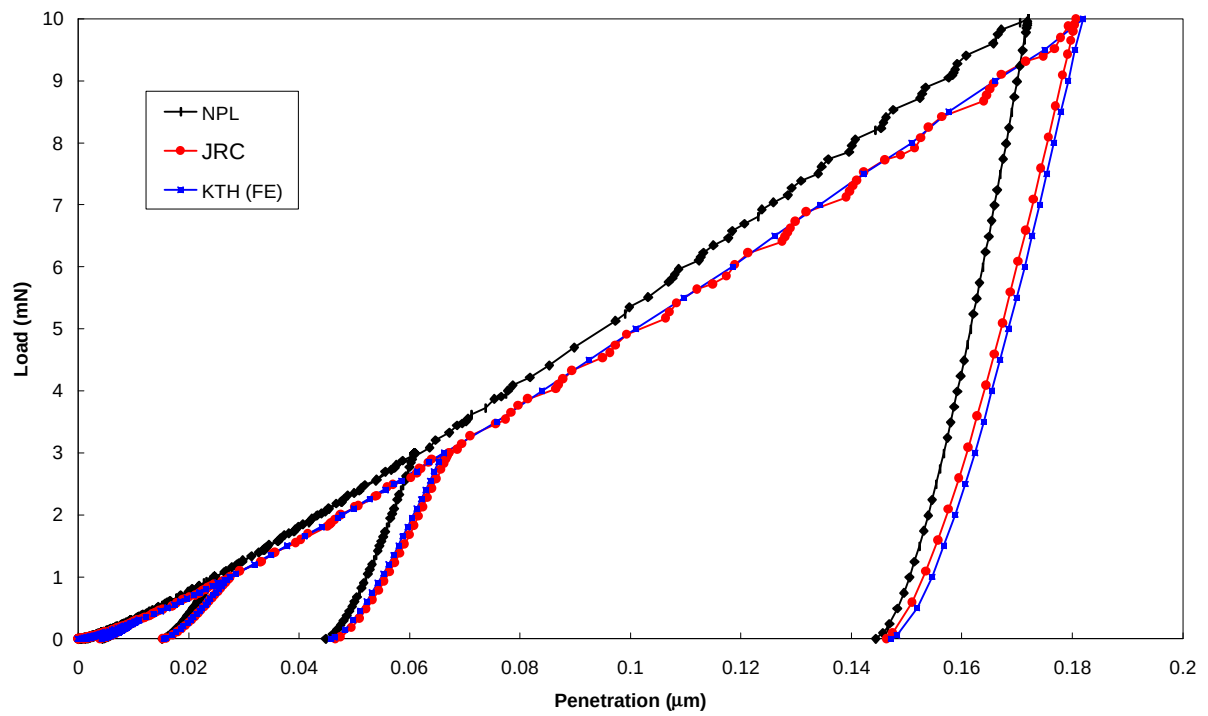


Fig.11 : 5.12 mm gold coating on a nickel substrate.

Estimation of indentation modulus

The load-penetration results of the elastic-plastic FEA predictions for various coating systems and maximum loads (including unloading to zero load) have been analysed in exactly the same way as would experimental data. This was achieved by modifying the software used at NPL to analyse experimental load-penetration data. The methodology for interpreting the results corresponds to that developed by Oliver and Pharr [8] providing an estimate of the reduced elastic modulus $E_{IT} = E / (1 - \nu^2)$. The fact that the Oliver and Pharr method involves estimating the gradient of the unloading part of the load-penetration data means that the unloading predictions by FEA should be as accurate as possible. As the NPL FEA results were readily available, they have been used as the basis of the following analysis. It is emphasised that when estimating the gradient of the unloading curve the procedure followed was to fit a curve to the simulation data and then to differentiate the fitted curve in order to estimate the gradient of the data.

Table 2 shows the results for two coating thicknesses where alumina has been deposited on a nickel substrate. The Oliver and Pharr method estimates of E_{IT}^* are seen to depend on the value of the maximum load. This means that the estimates for the indentation modulus are not just functions of the coating thickness, and properties of the coating and substrate. All the results in Table 2 lie between the values of E_{IT} for coating and substrate: 164.761 GPa and 242.728 GPa respectively. The result (in bold italics) for the case of a maximum load of 30 mN and coating thickness 0.915 μm does not follow the trend of the other results. The result has been checked and no explanation for this phenomenon has been found.

Table 2 : Alumina coating on a nickel substrate.

Coating thickness (microns)	Max. load (mN)	E_{IT} for coating (GPa)	E_{IT} Oliver & Pharr	Unloading Hardness (GPa)	Contact radius (microns)	Gao et al Modulus (GPa)	Gao et al Poisson's ratio
0.32	1	164.76	189.583	2.24	0.3772		
	3	164.76	191.088	2.30	0.6450		
	10	164.76	203.010	2.40	1.1506		
	30	164.76	222.562	2.61	1.9135		
	100	164.76	236.034	2.97	3.2750	-928.386	1.186225
0.915	1	164.76	179.514	2.60	0.3497		
	3	164.76	182.988	3.68	0.5093		
	10	164.76	175.045	4.07	0.8843		
	30	164.76	171.252	3.58	1.6333		
	100	164.76	193.665	3.42	3.0487	56.53433	0.167655

Coating : $E_c = 160 \text{ GPa}$, $\nu_c = 0.17$. Substrate : $E_s = 220 \text{ GPa}$, $\nu_s = 0.306$

Table 3 shows the results for three coating thicknesses where aluminium has been deposited on a BK7 substrate. It is seen again that the Oliver and Pharr method estimates of E_{IT}^* are dependent on the value of the maximum load. When the load is large enough it is seen (bold italics) that the Oliver and Pharr prediction of E_{IT} exceeds the value 85.42 GPa for the substrate, which is an unexpected unexplained result.

Table 3 : Aluminium coating on a BK7 substrate.

Coating thickness (microns)	Max. load (mN)	E_{IT} for coating (GPa)	E_{IT} Oliver & Pharr	Unloading Hardness (GPa)	Contact radius (microns)	Gao et al Modulus (GPa)	Gao et al Poisson's ratio
0.31	0.1	78.924	82.348	0.71	0.2113		
	0.3	78.924	87.204	0.86	0.3339		
	1	78.924	93.814	1.12	0.5334		
	3	78.924	100.941	1.45	0.8105		
	10	78.924	111.872	1.81	1.3244	-770.463	2.128153
1.05	0.1	78.924	79.433	0.67	0.2174		
	0.3	78.924	83.180	0.79	0.3488		
	1	78.924	87.768	0.93	0.5857		
	3	78.924	93.237	1.12	0.9219		
	10	78.924	103.169	1.41	1.5017	-25.4903	0.898999
3.4	0.1	78.924	80.820	0.67	0.2185		
	0.3	78.924	81.358	0.77	0.3518		
	1	78.924	83.748	0.91	0.5900		
	3	78.924	87.559	1.05	0.9539		
	10	78.924	93.041	1.20	1.6309	47.03558	0.618181

Coating : $E_c = 69.8$ GPa, $\nu_c = 0.34$. Substrate : $E_s = 81.9$ GPa, $\nu_s = 0.203$

Table 4 shows the results for three coating thicknesses where DLC has been deposited on an M2 steel substrate. It is seen again that the Oliver and Pharr method estimates of E_{IT}^* are dependent on the value of the maximum load. All the results in Table 4 lie between the values of E_{IT} for coating and substrate: 142.708 GPa and 240.856 GPa respectively. The result (bold italics) for the case of a maximum load of 3 mN and coating thickness 2.1 μm does not follow the trend of the other results. This result has been checked and no explanation for this phenomenon has been found.

Table 4 : DLC coating on a M2 steel substrate.

Coating thickness (microns)	Max. load (mN)	E _{IT} for coating (GPa)	E _{IT} Oliver & Pharr	Unloading Hardness (GPa)	Contact radius (microns)	Gao et al Modulus (GPa)	Gao et al Poisson's ratio
0.28	1	142.71	191.272	2.65	0.3463		
	3	142.71	201.342	3.99	0.4894		
	10	142.71	211.729	6.05	0.7256		
	30	142.71	218.619	7.68	1.1149		
	100	142.71	227.905	9.04	1.8763	-55.7142	0.524359
1.3	1	142.71	171.604	2.66	0.3374		
	3	142.71	174.712	3.75	0.5046		
	10	142.71	181.390	5.64	0.7516		
	30	142.71	192.366	8.05	1.0894		
	100	142.71	219.382	9.14	1.8664	49.87474	0.533885
2.1	1	142.71	169.534	2.55	0.3531		
	3	142.71	166.505	3.70	0.5083		
	10	142.71	170.535	5.49	0.7617		
	30	142.71	177.428	7.92	1.0978		
	100	142.71	190.983	10.16	1.7698	132.1255	0.281708

Coating : E_c = 137 GPa, ν_c = 0.2. Substrate : E_s = 220.6 GPa, ν_s = 0.29

Table 5 : Gold coating on a nickel substrate.

Coating thickness (microns)	Max. load (mN)	E _{IT} for coating (GPa)	E _{IT} Oliver & Pharr	Unloading Hardness (GPa)	Contact radius (microns)	Gao et al Modulus (GPa)	Gao et al Poisson's ratio
0.3	0.1	97.594	160.195	0.71	0.2122		
	0.3	97.594	188.227	0.89	0.3267		
	1	97.594	234.118	1.13	0.5311		
	3	97.594	269.196	1.33	0.8468		
	10	97.594	306.419	1.54	1.4363	-3188.23	2.294381
1.19	0.1	97.594	91.914	0.45	0.2669		
	0.3	97.594	109.190	0.58	0.4059		
	1	97.594	132.370	0.75	0.6511		
	3	97.594	170.259	0.93	1.0108		
	10	97.594	226.151	1.10	1.6987	-89.4991	0.759905
5.12	0.1	97.594	101.738	0.61	0.2278		
	0.3	97.594	106.495	0.72	0.3638		
	1	97.594	112.103	0.83	0.6199		
	3	97.594	121.453	0.92	1.0165		
	10	97.594	143.715	1.02	1.7663	65.49791	0.49626

Coating : E_c = 78.7 GPa, ν_c = 0.44. Substrate : E_s = 220 GPa, ν_s = 0.306

Table 5 shows the results for three coatings where gold has been deposited on a nickel substrate. It is again seen that the Oliver and Pharr method estimates of E_{IT}^* are dependent on the value of the maximum load. Most of the results in Table 4 lie between the values of E_{IT} for coating and substrate: 97.594 GPa and 242.728 GPa respectively. Values lying outside this range are shown in bold italics.

In order to check the Oliver and Pharr methodology for the case of indentation into a homogeneous material, predictions and analysis have been carried out for the case of nickel. Table 6 shows that the Oliver and Pharr predictions over-estimate the E_{IT} value, with the over-estimate increasing with the maximum load applied before unloading. This is the same trend that was observed for indentation into coated samples, and is thought to arise because of the significant effects of plastic deformation.

Table 6 : Homogeneous nickel substrate.

<i>Max. load (mN)</i>	E_{IT} for nickel (GPa)	E_{IT} Oliver & Pharr	Unloading Hardness (GPa)	Contact radius (microns)
1	243.38976	252.689	1.66	0.43841470
100	243.38976	299.225	2.80	3.37362870

Substrate : $E_s = 220$ GPa, $\nu_s = 0.306$

10. Estimating Young's modulus and Poisson's ratio

The indentation data discussed above provides estimates only of the effective reduced modulus of coated systems E_{IT} defined, in the case of homogeneous samples, by

$$E_{IT} = \frac{E}{1 - \nu^2} . \quad (8)$$

For coated samples the measured value of E_{IT} is a composite value involving the properties of both the coating and substrate, and the thickness of the coating. The objective now is to attempt to develop a method of estimating the Young's modulus and Poisson's ratio of the coating separately. The approach to be investigated involves the use of a theoretical model developed by Gao et al [9]. For this model a rigid circular flat punch is indented into a coated substrate and an approximate analytical method is given that results in the following relation that will be the basis of the method of estimating E and ν for the coating

$$E_{IT} = \frac{2[\mu_s + (\mu_c - \mu_s)I_0(t_c / a)]}{1 - \nu_s - (\nu_c - \nu_s)I_1(t_c / a)} , \quad (9)$$

where μ_s and μ_c are respectively the shear moduli of the substrate and coating, and where t_c is the thickness of the coating and a is the radius of the cross-section of the circular flat punch. The functions I_0 and I_1 are defined by

$$I_0(x) = \frac{2}{\pi} \arctan x + \frac{1}{2\pi(1-\nu_s)} (1-2\nu_s)x \ln \frac{1+x^2}{x^2} - \frac{x}{1+x^2}, \quad (10)$$

$$I_1(x) = \frac{2}{\pi} \arctan x + \frac{x}{\pi} \ln \frac{1+x^2}{x^2}. \quad (11)$$

It should be noted that the function I_0 depends on Poisson's ratio of the substrate. Gao et al [9] have compared the values of I_0 for the values 0.2 and 0.4 of Poisson's ratio and shown that the value of I_0 is affected very little by its value. In (10) it has been assumed that the value of Poisson's ratio is that of the substrate.

The approach to be made here notes that the relation (9) can be rewritten in the form

$$2I_0(t_c/a)\mu_c + I_1(t_c/a)E^*v_c = [1-\nu_s + \nu_s I_1(t_c/a)] E^* - 2\mu_s[1-I_0(t_c/a)], \quad (12)$$

that is linear in the parameters μ_c and v_c . By applying the relation to two cases having different values of E_{IT} corresponding to two different sets of values of the coating thickness and/or indenter radius the following simultaneous linear equations are obtained

$$2I_0(t_c^1/a_1)\mu_c + I_1(t_c^1/a_1)E_{IT}^1v_c = [1-\nu_s + \nu_s I_1(t_c^1/a_1)] E_{IT}^1 - 2\mu_s[1-I_0(t_c^1/a_1)], \quad (13)$$

$$2I_0(t_c^2/a_2)\mu_c + I_1(t_c^2/a_2)E_{IT}^2v_c = [1-\nu_s + \nu_s I_1(t_c^2/a_2)] E_{IT}^2 - 2\mu_s[1-I_0(t_c^2/a_2)]. \quad (14)$$

Given values of t_c^1 , a_1 , E_{IT}^1 and t_c^2 , a_2 , E_{IT}^2 arising from two different cases it is possible to solve the equations (13) and (14) simultaneously to provide estimates of the shear modulus μ_c and Poisson's ratio v_c for the coating. The software used to derive the elastic properties of the coating has been tested by using (9) to derive input values of E_{IT}^1 and E_{IT}^2 and then using (13) and (14) to recover the input properties of the coating. The software has also been applied to the E_{IT} values given in Tables 2-5 for two different loads (30 and 100 mN). The basis of this approach is that during the initial stages of the elastic response during unloading the contact radius is fixed at its maximum value, and the elastic response can be approximated by the Gao et al analysis. In reality, plastic deformation will have occurred during loading forming a permanent indentation that is not taken into account by the Gao et al model.

In order to avoid introducing errors arising from the use of different FEA meshes, the relations (13) and (14) have been applied to a coated sample having the same thickness t_c but with two different maximum loads (leading to two different maximum contact radii a_1 and a_2 when indenting with a spherical indenter). The results of applying this methodology to the coated substrates analysed by FEA are given in Tables 1-4. It is seen that unsatisfactory predictions arise, which might be because the values of E_{IT} resulting from the FEA are too inaccurate, as already demonstrated. The methodology for extracting values of Young's modulus and Poisson's ratio for the coating from the value of E_{IT} might still have some merit.

Another reason for the unsatisfactory predictions for Young' modulus and Poisson's ratio could be the fact that the approximate Gao et al analysis is not accurate enough for the purpose intended. To investigate this Gao et al predictions for the case of a rigid circular punch (radius $5\text{ }\mu\text{m}$) penetrating coated substrates have been compared to previous FEA predictions used when developing the hybrid technique for modelling indentation in the elastic regime of deformations [7]. For the case of penetration by unit displacement into a soft coating ($E = 70\text{ GPa}$, $\nu = 0.33$) attached to a hard substrate ($E = 200\text{ GPa}$, $\nu = 0.3$) the results of the comparison are given in Fig.12.

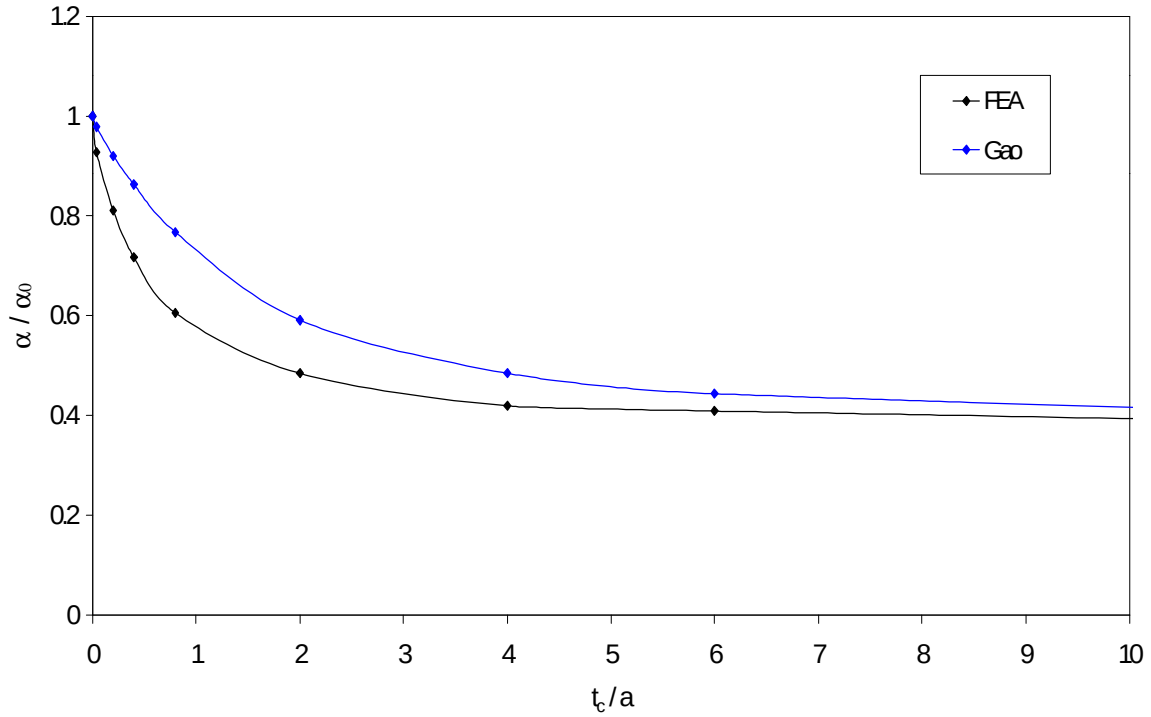


Fig.12 : Comparison of Gao et al predictions with FEA results for the case of indentation into a soft coating on a hard substrate

The parameter α plotted in Fig.12 denotes the load applied to a rigid circular punch divided by the product of the punch radius and the penetration. When indenting a homogeneous sample $\alpha = 2E_{IT}$. The parameter α_0 is the value of α when the coating thickness is zero. Fig.13 shows the corresponding comparison for penetration into a hard coating on a soft substrate.

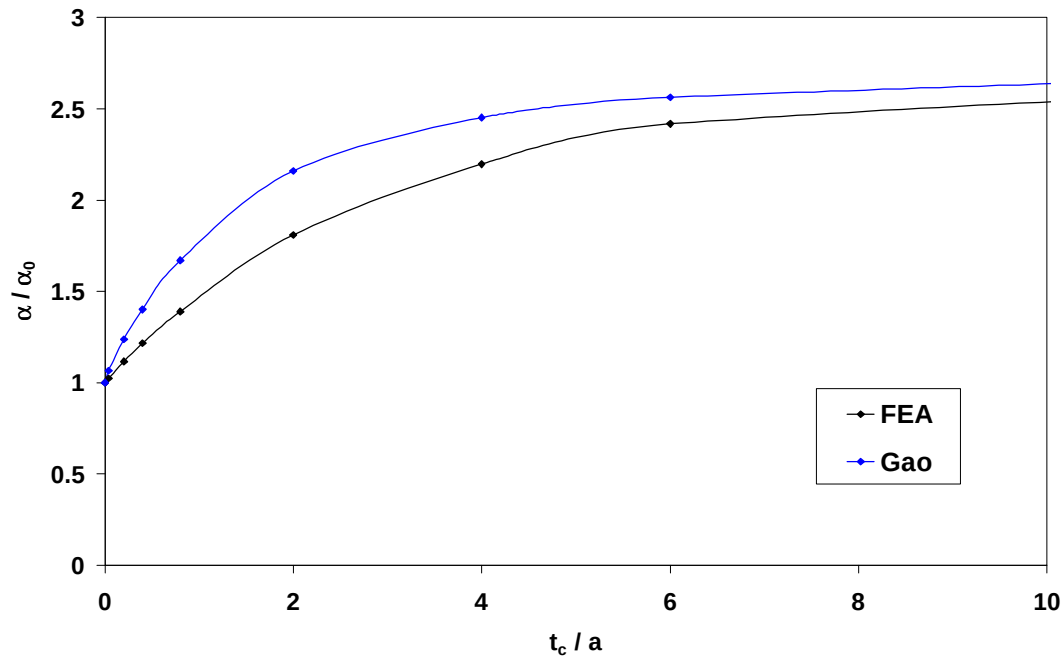


Fig.13 : Comparison of Gao et al predictions with FEA results for the case of indentation into a hard coating on a soft substrate.

It is seen from Figs.12 and 13 that the predictions of the Gao et al model do not agree particularly well with those obtained by FEA when developing the hybrid model for elastic deformations. This might explain the poor results in Tables 2-5 obtained using the methodology based on the Gao et al model to estimate coating properties. To investigate this in more detail, two cases have been selected for *elastic* analysis where the coating thicknesses are $1\mu\text{m}$ and $10\mu\text{m}$ and the radius of a flat circular rigid cylinder is $5\mu\text{m}$. For the case of a soft coating ($E = 70\text{ GPa}$, $\nu = 0.33$) on a hard substrate ($E = 200\text{ GPa}$, $\nu = 0.3$) the corresponding FEA estimates of E_{IT} for a rigid flat circular punch are 178.27676 GPa and 106.55117 GPa respectively. On using the methodology described above based on the Gao et al model to estimate coating properties it is found that the predicted coating Young's modulus is 74.06929 GPa rather than 70 GPa , and that the predicted coating Poisson's ratio is 5.45×10^{-4} rather than 0.33 . Thus the errors of the Gao et al model have led to a reasonable, but not particularly accurate modulus, and an inaccurate value of Poisson's ratio. For the case of a hard coating ($E=200\text{ GPa}$, $\nu=0.3$) on a hard substrate ($E = 70\text{ GPa}$, $\nu = 0.33$) the FEA estimates of E_{IT} for a rigid flat circular punch are 87.64085 GPa (when coating thickness is $1\mu\text{m}$) and 142.1548 GPa (when coating thickness is $10\mu\text{m}$). On using the methodology described above based on the Gao et al model to estimate coating properties it is found that the predicted coating Young's modulus is 168.68475 GPa rather than 200 GPa , and that the predicted coating Poisson's ratio is -0.26512 rather than 0.3 . Thus the errors of the Gao model have led to an inaccurate modulus, and an unreasonable negative value of Poisson's ratio. It should be noted that, when using values of E_{IT} generated by the Gao model (rather than by FEA) for rigid flat circular punches, the procedure to estimate the elastic properties of the coating has been shown to lead to the correct results, confirming that the method of analysis is consistent.

It is concluded that the methodology based on the Gao et al analysis for estimating, in the case of elastic deformations, the elastic properties of the coating is not satisfactory due to inaccuracies of the Gao et al model. It is, therefore, not surprising that when the methodology was applied to unloading data predicted by elastic-plastic FEA, unsatisfactory predictions for the elastic properties of coatings were obtained, as seen in Tables 2-5.

11. Conclusions regarding estimation of elastic coating properties

1. When using the Oliver and Pharr method to estimate the value of E_{IT} for the coating from unloading load-penetration data generated by FEA, the resulting values are inaccurate.
2. More accurate predictions might arise when using a method of analysis that can be applied to purely elastic deformations during penetration. This would imply that large radius spherical indenters should be used when obtaining experimental data.
3. The Gao et al analysis for the elastic penetration of a coated substrate by a flat cylindrical indenter has been shown to be too inaccurate for use in the procedure to extract values of Young's modulus and Poisson's ratio from two values of E_{IT} obtained from the same coating/substrate system in the presence of plastic deformation.
4. Further work is needed to develop a reliable method of extracting the elastic properties coatings from load penetration data.

12. Hardness results

In order to have any meaning as a 'materials property', hardness should really have a value independent of the applied load for any homogeneous material. Equally, the computed hardness of an elastic/plastic model substance should ideally be independent of the applied load. It is seen from Table 6 that this does not appear to be true for the hardness computed from the FEA results for nickel. Hardness is defined by:

$$H_{IT} = P / A_p \quad (15)$$

where P is the applied load and A_p is the contact area. As we are working from load/penetration data the value of P is not in any doubt and thus any error in H_{IT} is due to an error in estimating A_p .

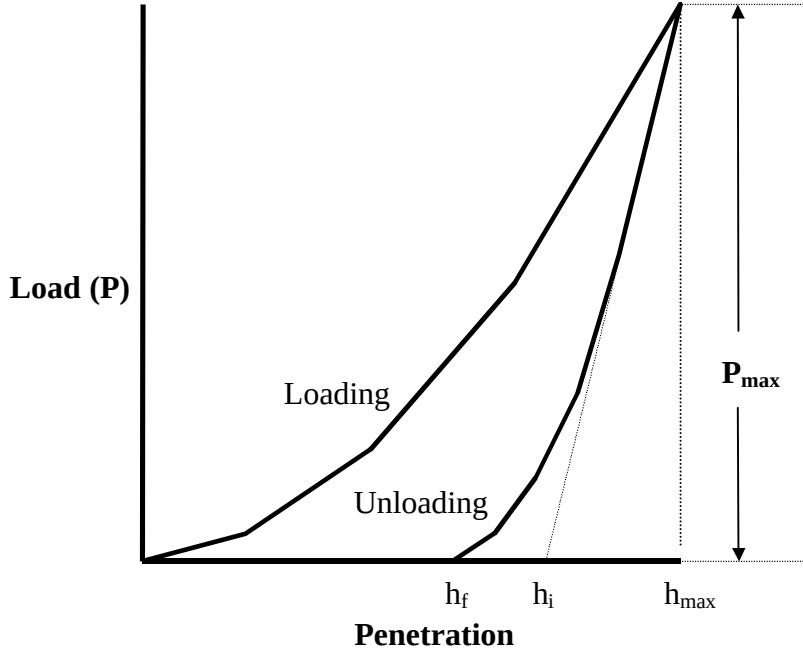


Fig.14 : Schematic diagram of a typical load/penetration curve.

It has been part of the philosophy of the present work to estimate E_{IT} and H_{IT} from the load/penetration data obtained from a finite element analysis using the same procedures as would have been used to analyse the corresponding experimental data. Thus A_p is estimated from a subroutine describing the indenter profile in the form:

$$A_p = A_p(h_c) \quad (16)$$

where h_c is the contact depth estimated from

$$h_c = h_f + \chi(h_{max} - h_i) \quad (17)$$

where χ is a dimensionless constant, h_f is the final penetration at zero load, h_{max} is the penetration at maximum load and h_i is the intercept of the tangent to the upper part of the unloading curve with the $P = 0$ axis (see Fig.14).

In the analysis program the upper 80% of the unloading curve is fitted by a polynomial. This makes it possible to find the gradient dP/dh at h_{max} and so extrapolate to find h_i the intercept of that gradient with the h axis. The corrected intercept h_c is then found from (17).

The parameter χ is a constant taken as 0.75. This correction factor is intended to remove the effects of purely elastic response. Using the Hertz solution for the purely elastic case without a coating where $h_f = 0$, it may be shown that the contact area A_p at maximum load is correctly given by $A(h_c)$ where h_c is defined by equation (17) only if ϵ is 0.75. Conversely in the case of perfect plasticity where $h_f = h_i = h_{max}$ any value of χ gives the correct value of A_p . Thus if the elastic/plastic case is thought of as a superposition of the elastic and plastic contributions to h_c

then it might be concluded that setting χ equal to 0.75 would give accurate values of A_p , although there is no rigorous justification for this assumption. As A_p is used to estimate both E_{IT} and H_{IT} it is clearly vital to compute it correctly. The variability of the estimates of H for pure nickel suggests that the evaluation of A_p should be improved.

In the Tables 2-6 the hardness generally increases with maximum load indicating either a systematic error in the estimation of A_p or that H_{IT} cannot be regarded as a constant material property for elastic/plastic coated systems. The only exception to this rule are the results for the 0.915 micron coating of alumina on a nickel substrate which also give anomalous results for E_{IT} (see bold italic results in Table 2). Apart from this trend the hardness of different coatings varies as expected: DLC is harder than alumina and both of them are harder than aluminium or gold.

13. Intercomparison of elastic-plastic results

Initially this study used only results obtained at NPL but, in view of the problems of interpreting the results, it was decided to compare these results with those based on FEA analyses carried out by JRC and KTH for a single case to ensure that the findings were not a peculiarity of the NPL analyses. The case taken for the comparative study was the 100 mN loading of a 0.28 μm thick coating of DLC on an M2 steel substrate. This case has the merit that a good set of results from each institution was available and the Gao et al analysis gives an unphysical negative modulus.

Table 7 shows that the FEA data from the three institutions at a given load agree better than the results from one institution (NPL) at different loads (shown in Table 4).

Table 7: Comparison of results from different institutions.

	E_{IT} Oliver & Pharr (GPa)	Contact Hardness (GPa)	Contact Radius (μm)
JRC	232.544	9.03	1.8775904
KTH	220.220	9.07	1.8733728
NPL	227.905	9.04	1.8763193

It has been described how, in the Oliver & Pharr analysis of the unloading curve, the contact area A_p is estimated from $A_p = A_p(h_c)$. In the finite element solution A_p may be estimated directly. As this area affects the estimation of both E_{IT} and H_{IT} it is prudent to check that these estimates of contact area agree. The system chosen for this check was gold on nickel.

As FEA estimates of A_p were too uncertain at loads of 0.1 mN, loads of 1 mN and 10 mN were considered. The results of the analysis are shown in Table 8.

Table 8: Results using estimated and computed values for A_p in the gold on nickel system.

Coating Thickness (μm)	Max. Load (mN)	A_p Oliver & Pharr (μm^2)	A_p Finite Element (μm^2)	E_{IT} Oliver & Pharr (GPa)	E_{IT} Finite Element (GPa)	H_{IT} Oliver & Pharr (GPa)	H_{IT} Finite Element (GPa)
0.30	1.0	0.886	1.203	234.12	200.94	1.13	0.83
0.30	10.0	6.4806	10.230	306.42	243.88	1.54	0.98
1.19	1.0	1.3318	1.615	132.37	120.21	0.75	0.62
1.19	10.0	9.0655	12.140	226.15	195.42	1.10	0.82
5.12	1.0	1.2074	1.355	112.10	105.84	0.83	0.74
5.12	10.0	9.8012	12.350	143.72	128.04	1.02	0.81

It should be noted that an accurate estimate for contact area is very difficult to achieve by FEA analysis, as the output values tend to be rather 'noisy' unless a very refined mesh is used. Nevertheless it is certain that the finite element values are always greater than those estimated by Oliver and Pharr and this discrepancy, which is larger than expected FEA errors, becomes larger with increasing maximum load. It is also seen from Table 8 that this difference in A_p leads to a considerable difference in the estimates of E_{IT} and H_{IT} .

It is concluded that the unsatisfactory predictions of E_{IT} by the Oliver and Pharr method cannot be attributed to inaccurate stress analysis using FEA. The most likely reason is that the methodology is invalid.

In order to investigate further the integrity of the FEA results, when attempting to predict values for the effective modulus E_{IT} and the hardness H , the results from NPL and KTH have been compared. The results for E_{IT} and H are plotted against penetration depth/coating thickness. It is seen from the results shown in Figs.15-22 that the NPL results are on the whole numerically larger than the KTH results, and this is in part due to differing definitions of the contact area A_c used by the two institutions. NPL has used the same methodology as would be used by the Oliver & Pharr method of analysing experimental data, as this approach was thought to be essential if good comparisons between model predictions and experimental results were to be achieved. KTH have used the contact area predicted directly by the FEA. The issue of contact area is discussed further in Section 14. It should be noted that the discrepancy shown in Fig. 16 and Fig. 20 for the case of an aluminium coating on a BK7 substrate is due to KTH using a very different set of material properties, as has been noted in Section 9 when the load/displacement results were compared.

The results of KTH and NPL have been plotted as a function of the ratio of penetration depth and coating thickness. It is interesting to note that the KTH modulus results for the various coating thicknesses tend to collapse on a single master curve. The NPL results show a less marked trend towards this type of behaviour. In Fig. 15, showing data for an alumina coating

on BK7, the KTH and NPL results are in reasonable agreement. The results in Fig. 16, for aluminium on BK7, show a distinct difference between the predictions of KTH and NPL; the NPL results showing higher values of the effective modulus E_{IT} . This type of trend is also observed in Figs. 17 and 18 for a gold coating on nickel, and for DLC on M2 toolsteel. The reasons for this trend are explored further in Section 14 where the differences are explained to some extent by differences used by KTH and NPL when estimating the contact area.

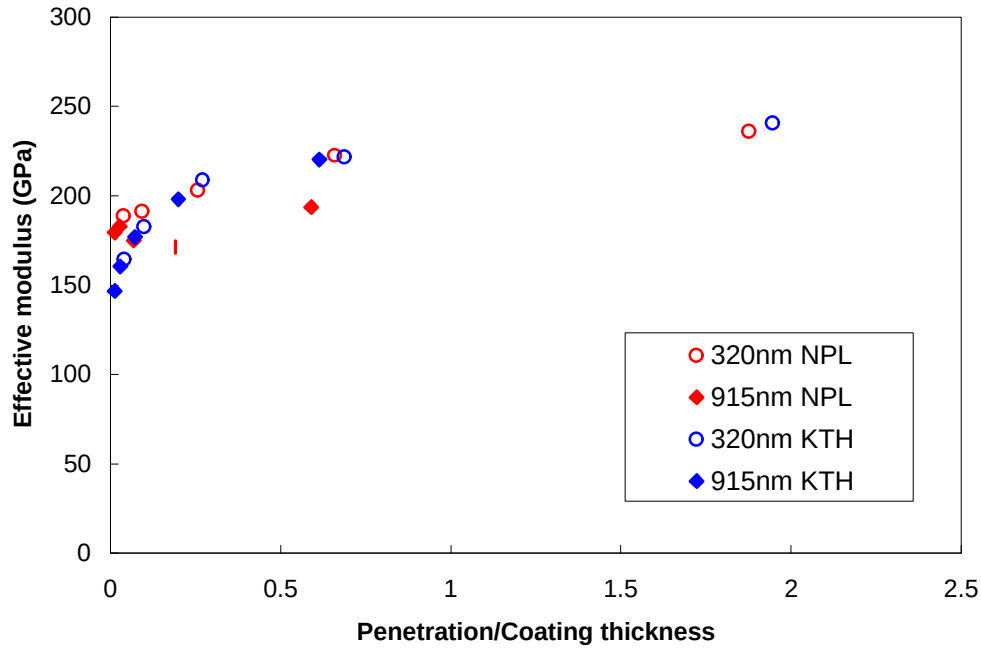


Fig.15 : NPL and KTH results E_{IT} for the alumina on nickel system.

$$E_{IT}^c = 164.76 \text{ GPa} , E_{IT}^s = 242.73 \text{ GPa} .$$

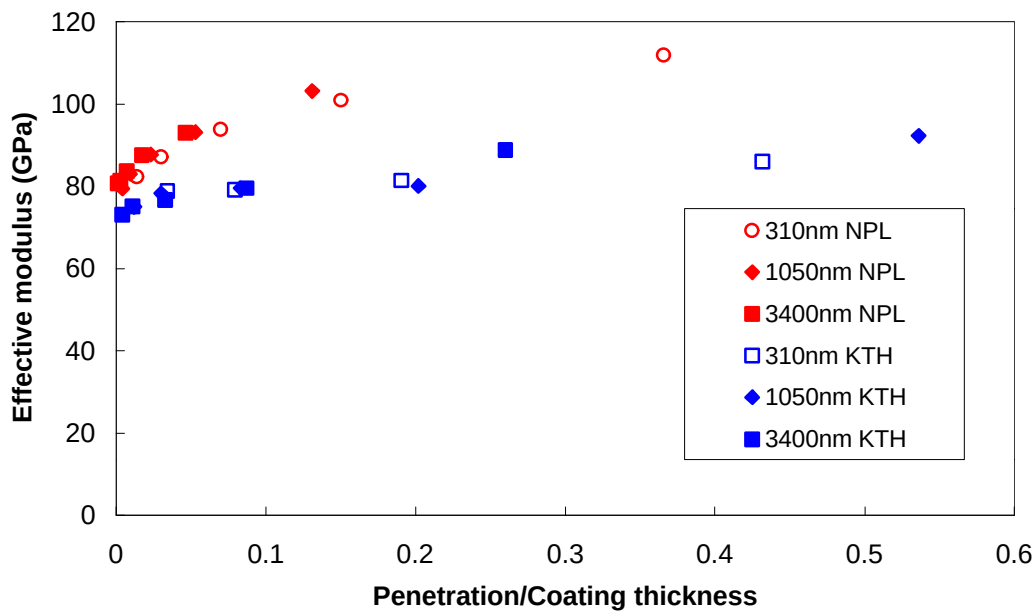


Fig.16: NPL and KTH results for E_{IT} for the aluminium on BK7 system.

$$E_{IT}^c = 78.92 \text{ GPa} , E_{IT}^s = 85.42 \text{ GPa}$$

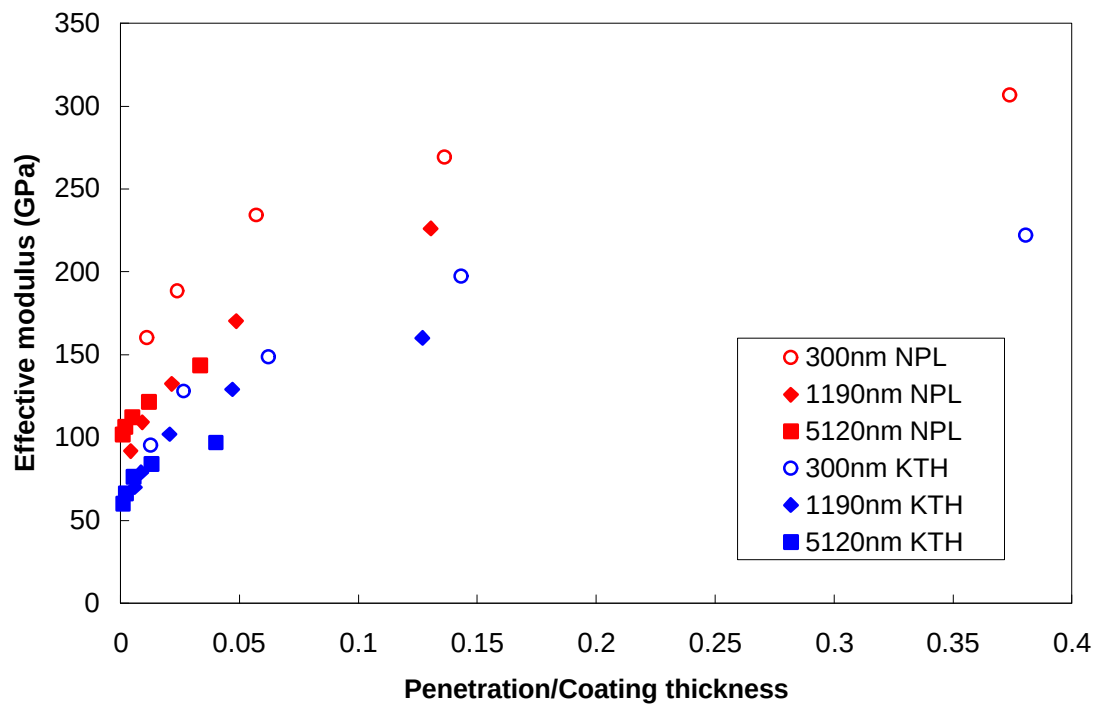


Fig.17: NPL and KTH results for E_{IT} for the gold on nickel system.

$$E_{IT}^c = 97.59 \text{ GPa} , E_{IT}^s = 242.73 \text{ GPa}$$

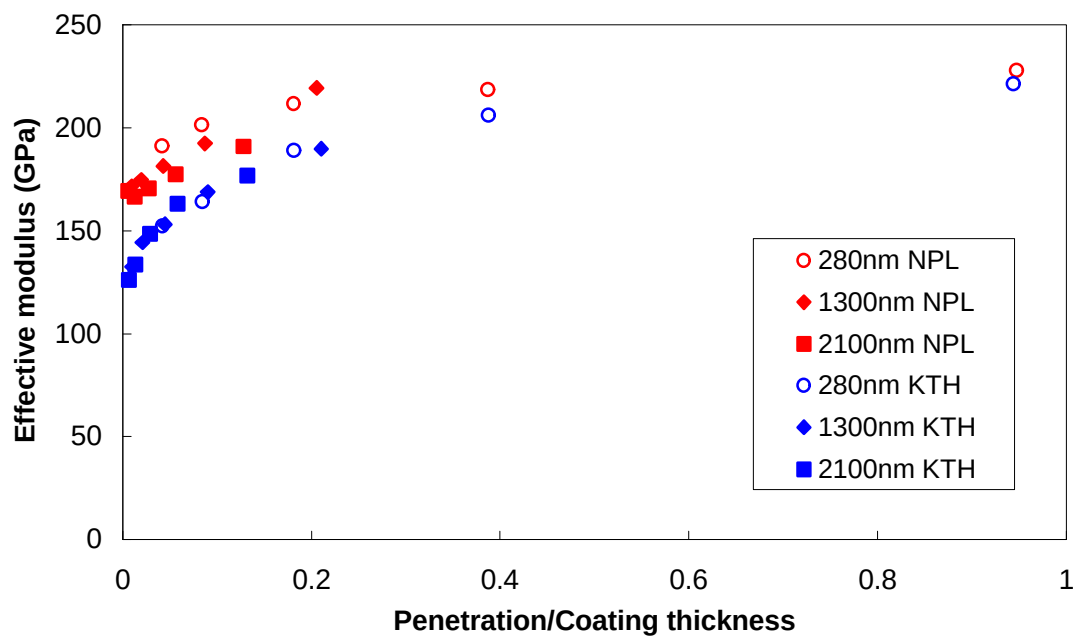


Fig.18: NPL and KTH results for E_{IT} for the DLC on M2 steel system.

$$E_{IT}^c = 142.71 \text{ GPa} , E_{IT}^s = 240.86 \text{ GPa}$$

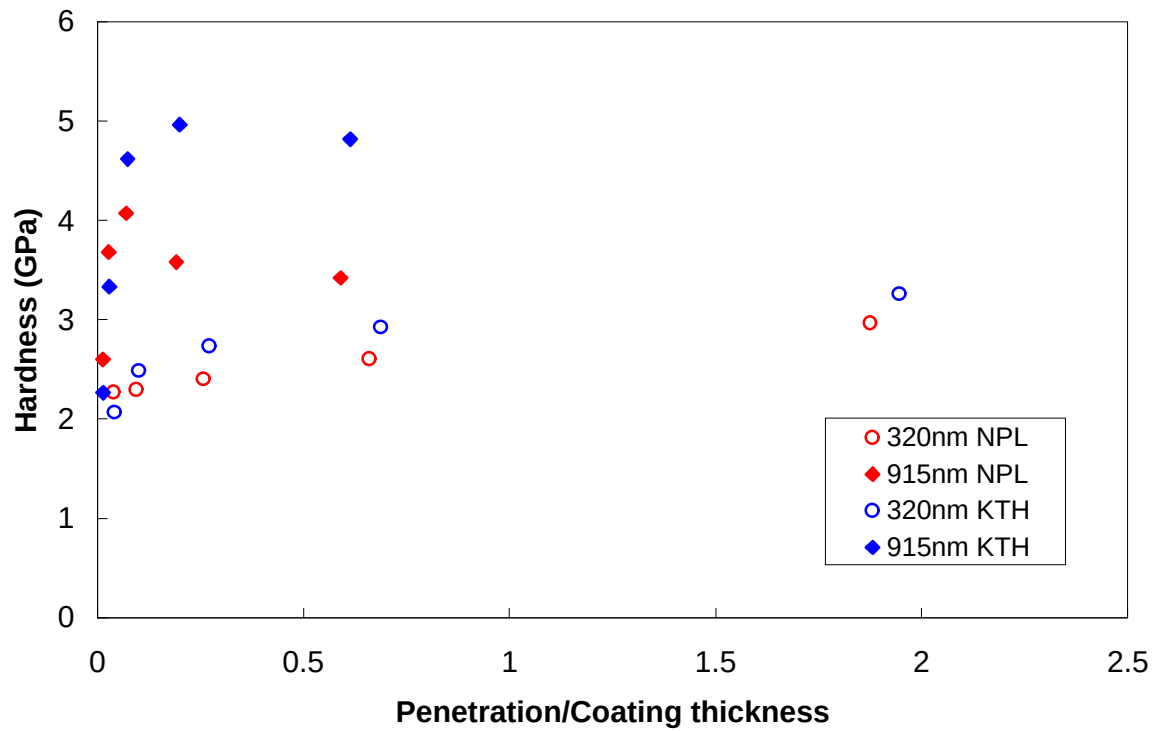


Fig.19 : NPL and KTH results for hardness of the alumina on nickel system.

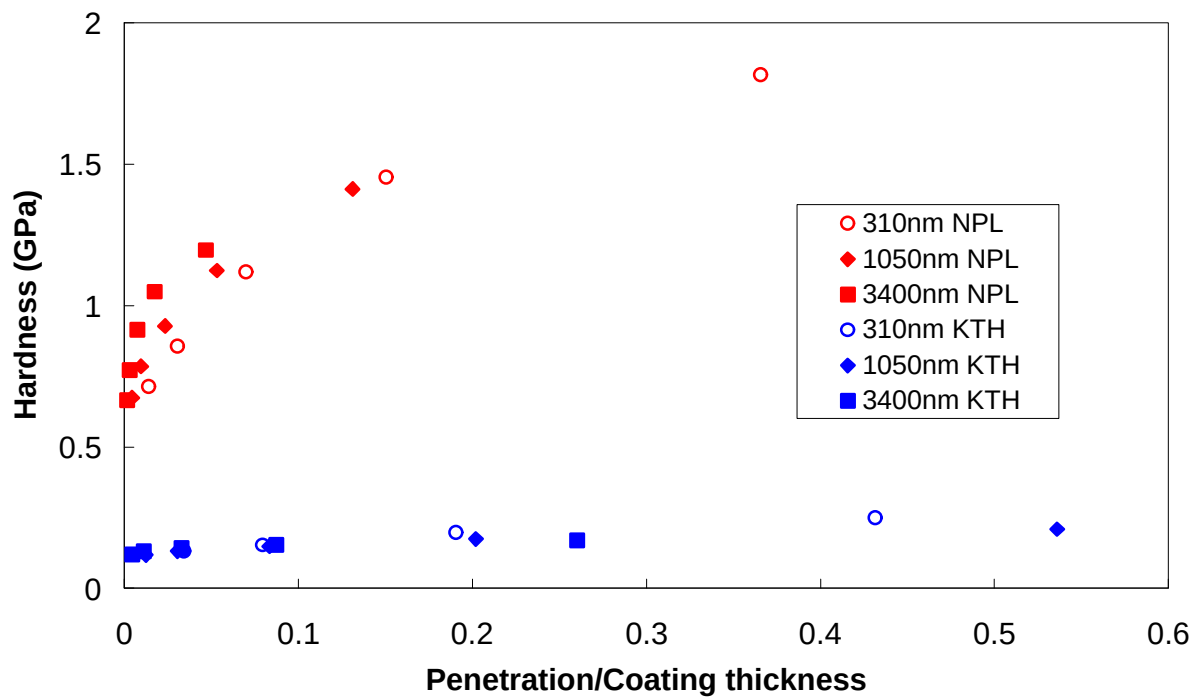


Fig.20 : NPL and KTH results for hardness of the aluminium on BK7 system.

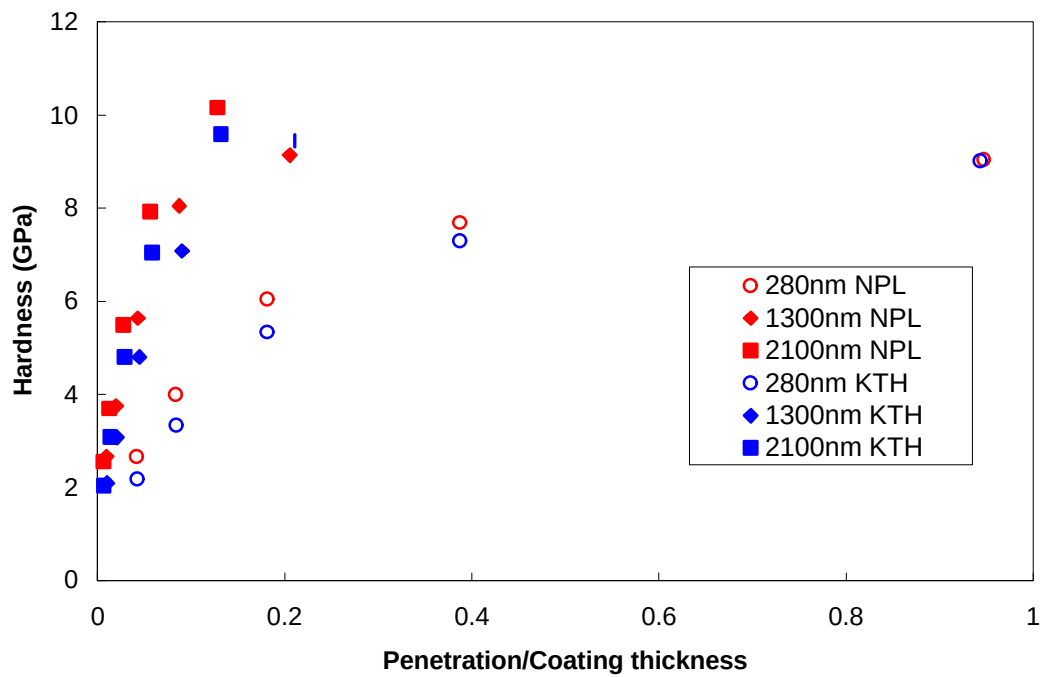


Fig.21: NPL and KTH results for hardness of the DLC on M2 steel system.

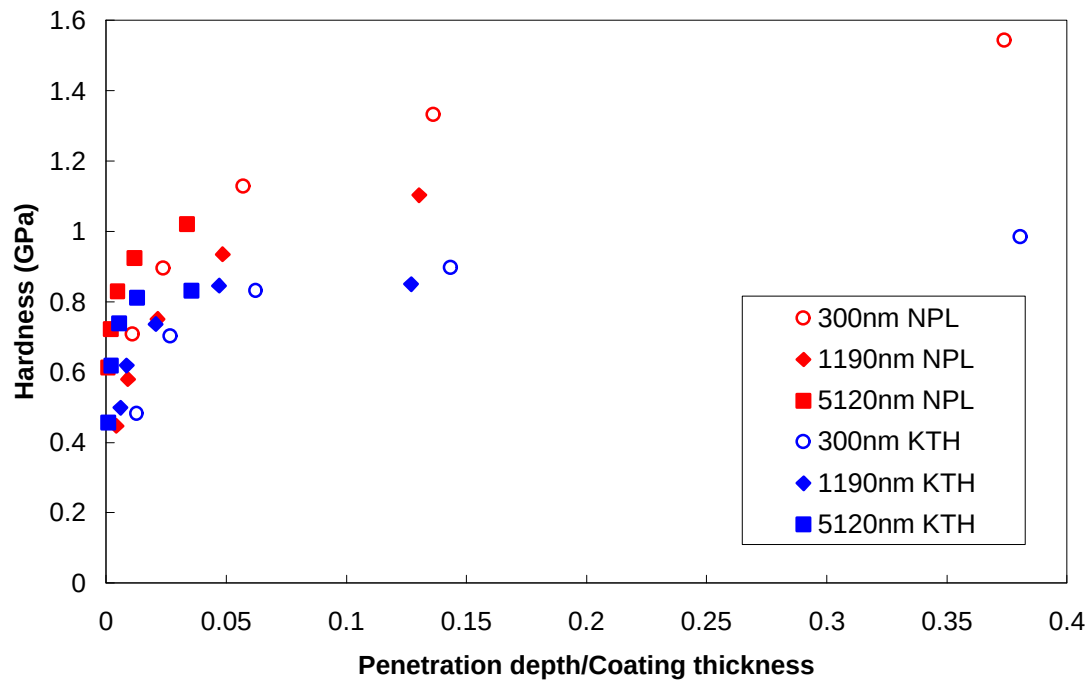


Fig.22 : NPL and KTH results for the hardness of the gold on nickel system.

14. The contact area effect

As indicated in Table 8 and the associated text above, the contact area A_p affects the calculation of both the effective Young's modulus E_{IT} and the hardness H , where H is proportional to the reciprocal of A_p and E_{IT} is proportional to the square root of this reciprocal. It turns out that there are at least four definitions of the contact area A_p that might be used when applying the results of FEA (see Fig. 23a,b).

The definitions of contact area are specified by :

1. After unloading, the projected area of that part of the deformed free surface lying below the level of the undeformed surface remote from the indentation $A_p = \pi(R_1)^2$.
2. The projected area $A_p = A_p(h_c)$ where h_c is defined by Oliver and Pharr (see equation (17)).
3. After unloading, the projected area of the deformed free surface lying within the local maximum of the piled up rim of the indentation $A_p = \pi(R_3)^2$.
4. At maximum load, the projected area of contact computed by FEA $A_p = \pi(R_4)^2$.

Of these, the areas defined by 1 and 3 are the closest to the values used in traditional hardness testing where the measurement is invariably done after the test is complete. The area 2 seems to be the most usual to use in nano-indentations obtained using machines that record both force and displacement. The area 4, although closest to the usual theory that applies to the unloading part of the load/penetration curve, cannot be measured in normal test conditions. In the bulk of the work carried out at NPL the area defined by 2 has been used as this corresponds to the way experimental data is used to estimate E_{IT} and H_{IT} . KTH have used the definition 4. It is often said that these definitions are equivalent, but the results of FEA indicate that this is not the case as seen from Table 9.

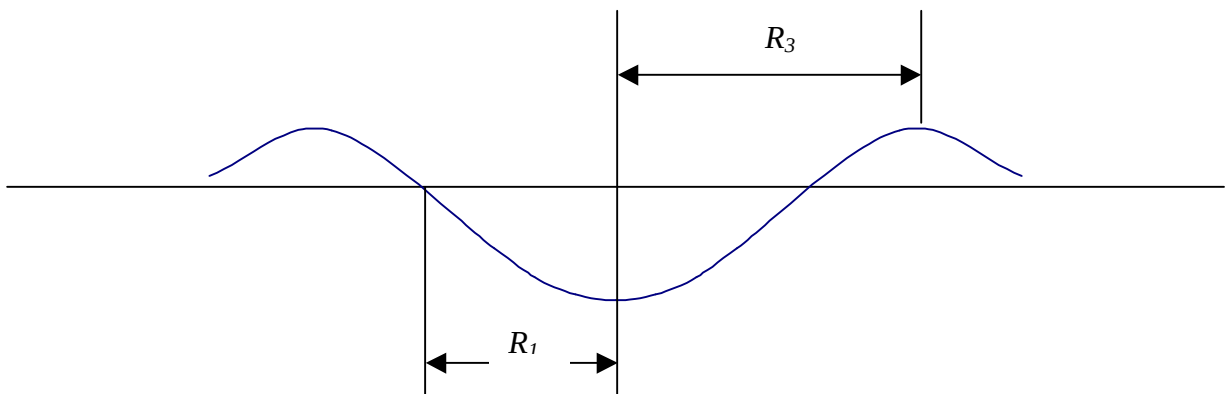


Fig.23a : The definitions of R_1 and R_3 in the unloaded state.

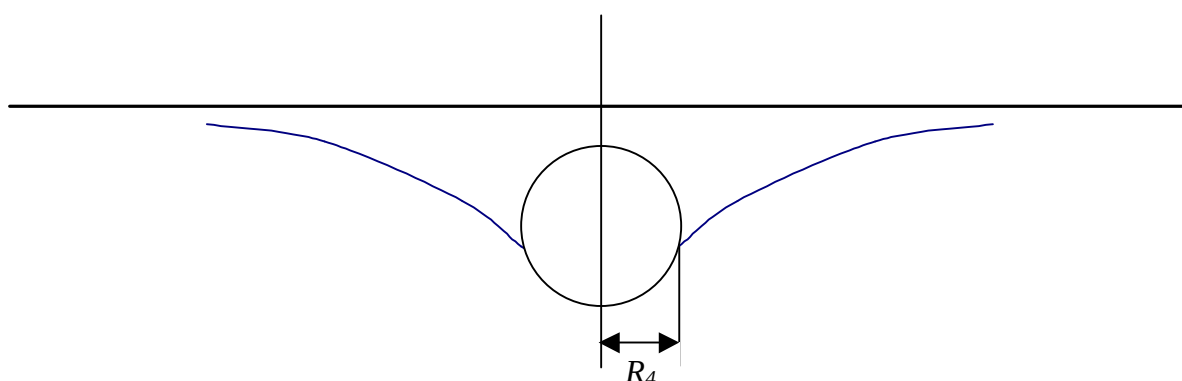


Fig.23b : The definition of R_4 at maximum load.

Table 9 : Results based on the four definitions of contact area for the case of a gold coating on a nickel substrate (see Appendix3 for further discussion).

Coating thickness (microns)	Max. Load (mN)	A_p (unloaded) Zero crossing (μm^2)	A_p Oliver & Pharr (μm^2)	A_p (unloaded) Crater rim (μm^2)	A_p (at max. load) ABAQUS Standard (μm^2)
Method		(1)	(2)	(3)	(4)
0.3	1	0.83	0.886	1.1469	1.203
1.19	1	1.0663	1.3318	1.3706	1.615
5.12	1	1.1325	1.2074	1.3981	1.355
0.3	10	5.1655	6.4806	9.566	10.23
1.19	10	8.3469	9.0655	11.9337	12.14
5.12	10	9.6211	9.8012	12.1181	12.35

There are significant differences in the values of the contact area produced by the four specified methods, especially when the coating thickness is small. Clearly the choice of method to estimate the contact area will have a dramatic effect on the corresponding estimates of effective modulus and hardness. The ideal definition (method 4) cannot unfortunately be realised in practice. This is the definition used by KTH when estimating the effective moduli and hardnesses given in Figs.15-22, and led to data (for various coating thicknesses) tending to fall on a single curve when plotting against the ratio of penetration to coating thickness.

15. Effect of the contact area on the comparison

It has been noted in Section 14 that NPL has used a definition of the contact area following the methodology of Oliver and Pharr whereas KTH have used the value at maximum load as computed by the finite element analysis i.e. definitions 2 and 4. Using the data in Table 9 the effect of these choices of contact area on the values of E_{IT} and H has been investigated. In particular, the NPL predictions can be 'corrected' to bring them closer to the KTH values. As all the values of the contact area used were calculated at NPL, the 'correction' is not perfect

otherwise the hardness plot, which depends only on the contact area and the applied load, would show the values coming into exact coincidence.

The correction procedure has been applied to the effective modulus and hardness results for a gold coating on a nickel substrate. The effect of modifying the contact area on hardness is shown in Fig. 24 where it is seen that the KTH and NPL results cluster together after the correction has been made. It should be noted that the corrected points on the far left of Fig. 24 are virtually coincident.

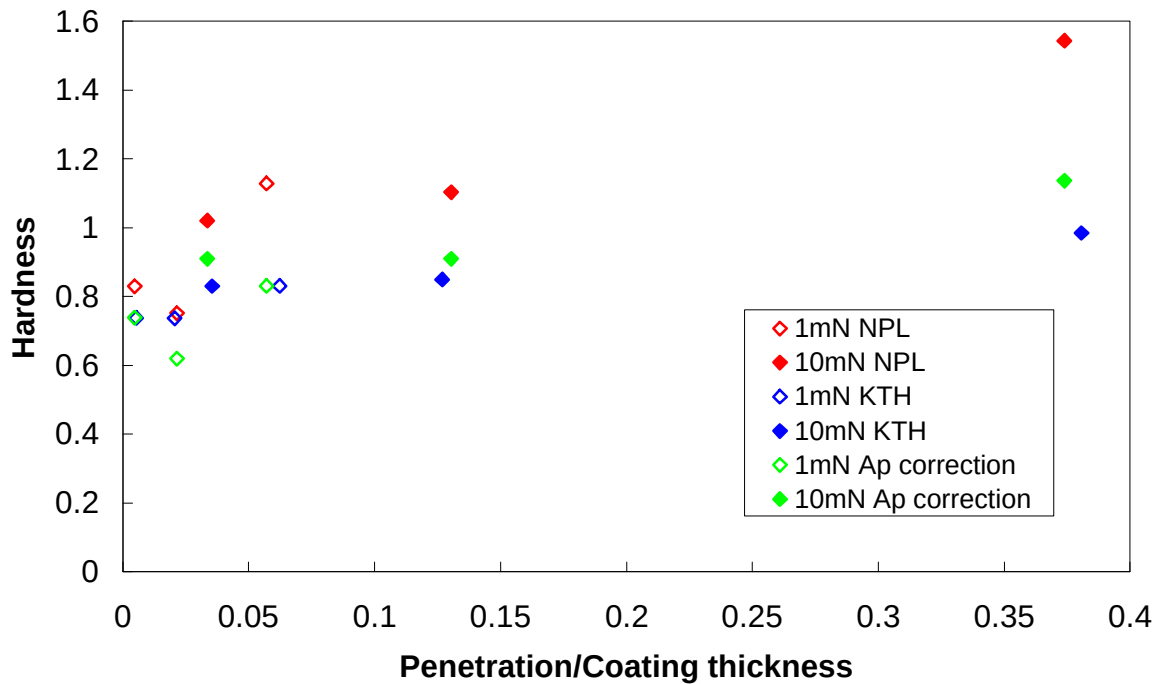


Fig.24 : Predicted values of H for the gold on nickel system by NPL and KTH, and the 'corrected' NPL values.

The corresponding correction to the effective modulus is shown in Fig. 25 where again it is seen that there is much better agreement between the KTH and NPL results when using the same definition of contact area. The computation of the effective modulus E_{IT} using the Oliver and Pharr technique requires the slope of the unloading curve at or near maximum load so the effect of the definition of the contact area alone is not expected to bring the values into agreement.

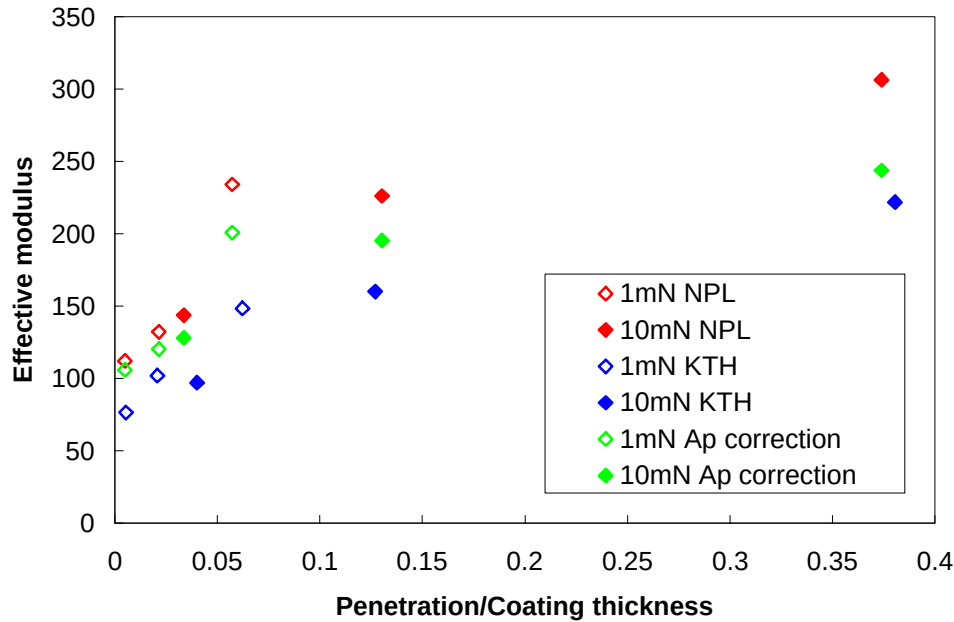


Fig.25 : Predicted values of E_{IT} for the gold on nickel system by NPL and KTH, and the 'corrected' NPL values. $E_{IT}^c = 97.59 \text{ GPa}$, $E_{IT}^s = 242.73 \text{ GPa}$

The major conclusion from this assessment, of the role of contact area on the predictions of hardness and effective modulus, is that the Oliver and Pharr estimate does not correspond to that which actually occurs during the elastic-plastic indentation of coated samples. The second conclusion is that the FEA carried out by KTH and NPL does lead to similar predictions of hardness and effective modulus when using the same method of estimating contact area. It should be noted that for the gold on nickel system KTH and NPL have used differing plasticity properties, and KTH have modelled the elasticity of the indenter while NPL modelled the indenter as a rigid solid.

It is also observed that some of the values predicted for the effective modulus lie outside range defined by the values for homogeneous coating and substrate material.

16. Analysis of loading curve

It is seen from the results presented in Section 10 that there are difficulties associated with the usual methods of evaluating the Young's modulus E and Poisson's ratio ν from the unloading curve. As both E and ν are elastic quantities they are best evaluated from the only truly elastic part of the load/penetration curve i.e. the beginning of the loading curve. Possible methods for achieving this are now discussed.

An alternative way of estimating the elastic constants is to use the initial part of the loading curve where the initial response is expected to be elastic. Elasticity theory provides the exact result:

$$E_{IT} = \frac{1}{2} \sqrt{\frac{\pi}{A_p}} \frac{dP}{dh} . \quad (18)$$

For the Hertz solution, provided the contact radius is very much smaller than the indenter radius R , where

$$A_p = \pi R h , \quad (19)$$

it follows that

$$E_{IT} = \frac{3P}{4h} \sqrt{\frac{\pi}{A_p}} = \frac{3P}{4h\sqrt{Rh}} . \quad (20)$$

It should be noted that, although equation (18) is identical to that used by Oliver and Pharr [8], it is used in a different context. Here A_p is the variable area of contact during initial loading. Whereas, for Oliver and Pharr, A_p is the constant area of contact at the start of the unloading curve largely formed by plastic deformation during loading.

It is emphasised that in (19) h is the penetration depth measured from the location of the free surface before loading. Equations (18) and (20) each give a formula for E_{IT} that can be used with data from the initial part of the loading curve. Using the data directly, or fitting it with a polynomial, in an attempt to reduce the effects of scatter on the numerical results, doubles the number of possibilities. Thus there are four possible estimates of E_{IT} based on one set of load penetration data $\{P_i, h_i\}$.

Let $\bar{h}_i = (h_i + h_{i+1}) / 2$, $\Delta h_i = h_{i+1} - h_i$ and $\Delta P_i = P_{i+1} - P_i$,

then from (18) and (19) :

$$E_{IT}^1(i) = \frac{1}{2\sqrt{R\bar{h}_i}} \frac{\Delta P_i}{\Delta h_i} \quad (21)$$

and from (19) and (20) :

$$E_{IT}^2(i) = \frac{3P_i}{4h_i\sqrt{Rh_i}} . \quad (22)$$

It is expected that the initial part of the loading curve would follow the Hertzian form with P proportional to $h^{3/2}$. This dependence can be accounted for before fitting and the $\{P_i, h_i\}$ data fitted using the relation:

$$P(h) = h^{3/2} f(h, n) , \quad (23)$$

where $f(h, n)$ is a polynomial in h of degree n . If we reserve $P(h)$ to refer to the value of P obtained from the polynomial fit, the remaining estimates of E_{IT} may be written from (21) and (22) as:

$$E_{IT}^3(h) = \frac{1}{2\sqrt{Rh}} \frac{dP(h)}{dh} , \quad (24)$$

and

$$E_{IT}^4(h) = \frac{3P(h)}{4h\sqrt{Rh}} . \quad (25)$$

Despite E_{IT}^3 and E_{IT}^4 being formally functions of the continuous variable h they were only evaluated at the measured values $h = h_i$.

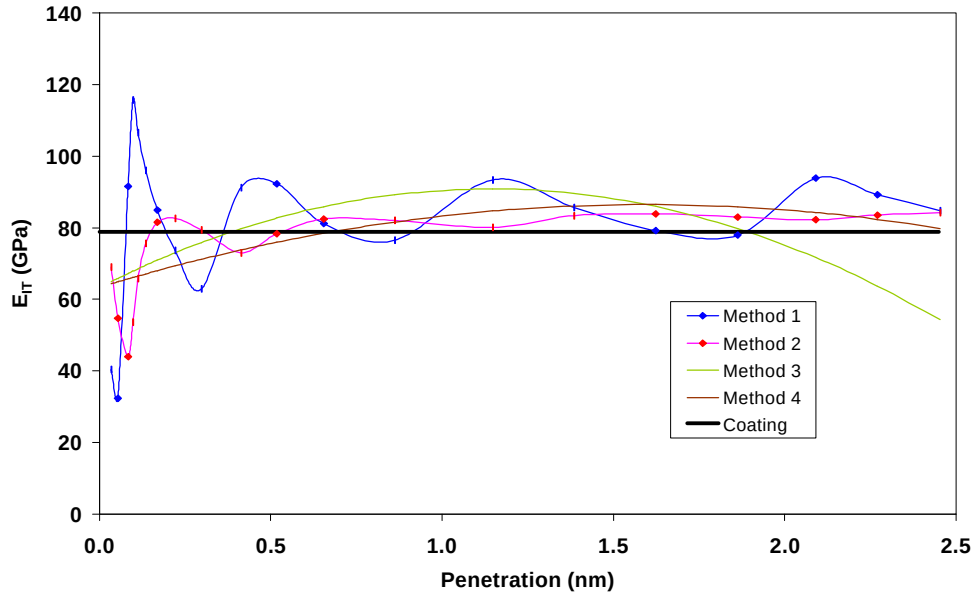


Fig.26 : Estimates of E_{IT} using equations (21), (22), (24), and (25) for a 0.31mm layer of Aluminium on BK7.

These estimates of E_{IT} were first computed for the Aluminium on BK7 system and the results are shown in Fig.26. In the figure E_{IT}^1 is shown as 'Method 1', E_{IT}^2 as 'Method 2' etc. The line marked 'coating' represents the input value of $E_{IT} = 78.92356$ GPa used in the finite element analysis. It is seen that the values obtained are not incompatible with the input value of E_{IT} for the coating. The two estimates E_{IT}^2 and E_{IT}^4 which do not estimate the gradient (dP/dh) give the least noisy and more accurate answers. Unfortunately, in this system, the input value of E_{IT} for the coating and for the substrate are similar to one another. It is difficult to determine whether the coating or the substrate is dominant.

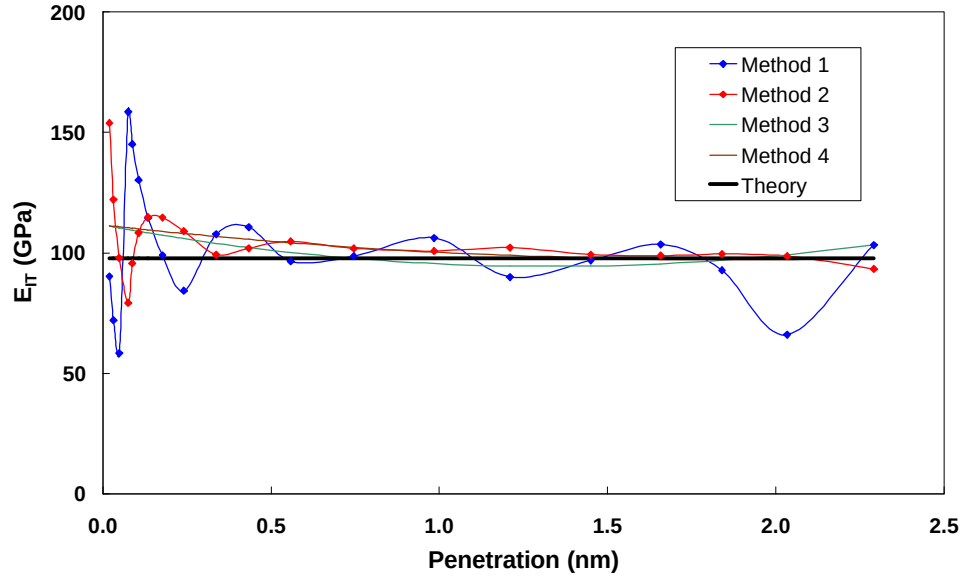


Fig.27 : Estimates of E_{IT} using equations (21), (22), (24), and (25) for a 5.12mm layer of gold on nickel.

The gold on nickel system has also been used in this study as it exhibits the largest difference between E_{IT} for the coating and the substrate, and the results have been used to observe to what extent coating or substrate properties were obtained. In this case E_{IT} is 97.594 GPa for the coating and 242.728 GPa for the substrate. In Fig. 27 a thick (5.12 μ m) coating is subjected to a comparatively shallow indent and it is clear that the estimates of E_{IT} are close to the value expected for the coating. Again estimates E_{IT}^2 and E_{IT}^4 give the best estimates of E_{IT} . When the layer is considerably thinner it is expected that the estimates of E_{IT} should converge towards an intermediate value between that expected for the substrate and that expected for the coating.

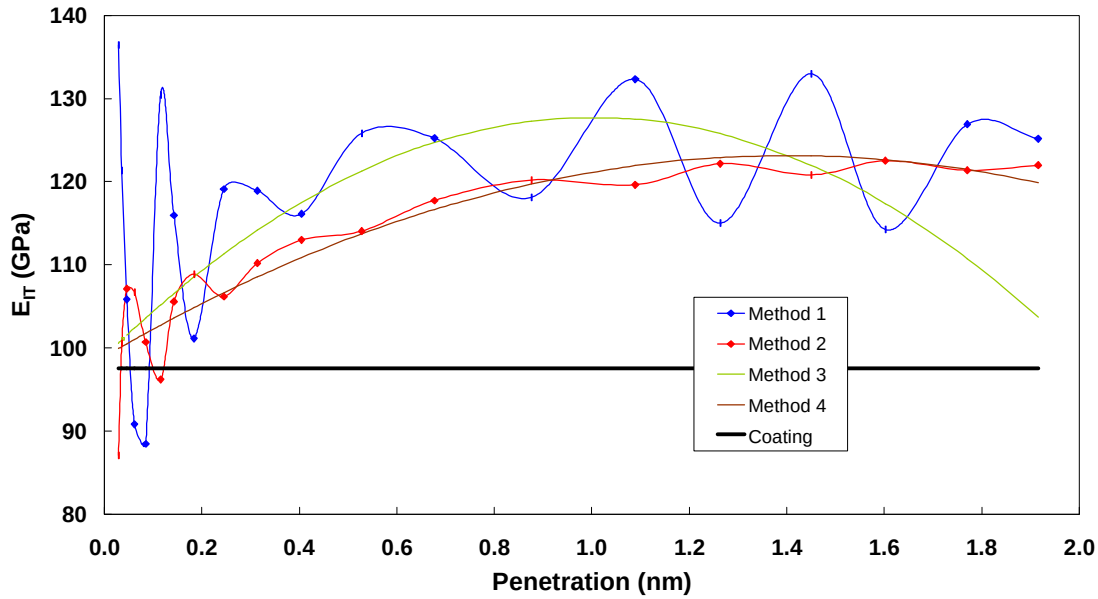


Figure 28 : Estimates of E_{IT} using equations (21), (22), (24), and (25) for a 0.3mm layer of gold on nickel.

It is seen from Fig. 28 that the estimates of E_{IT} are substantially larger than that of the coating alone which is consistent with our expectation. At larger penetrations where one might expect to regain the properties of the substrate the estimates are dominated by the plastic flow of the material and decrease continuously with applied load.

There is a need to extend the range of penetrations shown in Figs. 26-28 as the maximum penetration shown determined by the FEA results available is extremely small. The key question is to what penetration is the asymptotic value of E_{IT} observed in the elastic regime of deformation. If the asymptotic value of E_{IT} can be exhibited over reasonably large range of penetrations then it may be possible to develop a measurement method for E_{IT} based on the elastic loading part of load-penetration curves. The FEA results did not allow the required extension of the penetration range as more mesh refinement is needed to deal with the relatively low loads that would be applied when extending the data.

17. An alternative analysis method ?

For the case of a $2.1\ \mu\text{m}$ DLC on a substrate of M2 toolsteel, the load and unloading data obtained from elastic-plastic FEA modelling has been analysed in a particularly simple manner. Fig. 29 shows the plots obtained of load versus penetration h raised to the power $3/2$ predicted by elastic-plastic FEA carried out by NPL, JRC and KTH. This type of plot is expected to be linear when applying Hertz theory for the case of the elastic behaviour of homogeneous materials. The remarkable observation is that the plots remain almost linear for the case of elastic-plastic modelling of coated systems. It is seen that the results of NPL and JRC (who use infinite elements in the FEA) are close together whereas the KTH result slightly differs. These results are probably indicating that the load penetration behaviour is governed principally by the far field which deforms elastically.

The results in Fig. 29 have been used in conjunction with (21) and (22) to predict approximately E_{IT} (using method 2). The results are shown in Fig. 30 where it is seen that the three FEA predictions are showing more marked differences. All predictions show a local maximum when $t_c \cong 2$ which is a direct result of elastic-plastic deformation. This conclusion is reached on plotting the corresponding results (obtained using Elastica with 'rigid' indenter) for purely elastic deformations that are shown as continuous lines in Fig. 30. The continuous line is the accurate value of E_{IT} obtained using (21) while the broken line is the approximate prediction based on (24) and (25) (method 2). It is seen that the elastic predictions are monotonic decreasing functions that will tend for a given contact radius a to 240.86 GPa as $t_c \rightarrow 0$ and to 142.71 GPa as $t_c \rightarrow \infty$ thus indicating that the local maximum value shown by the FEA must be a result of elastic-plastic deformation. It is also observed that as $t_c / a \rightarrow 0$ the elastic-plastic results appear to tend towards the value $E_{IT}^c = 142.71\ \text{GPa}$ for the coating. No reason for this behaviour is at all apparent as one would expect the substrate to dominate in this limit. The KTH results show the most consistency with the purely elastic results in the region $t_c / a \geq 2$ while both the NPL and JRC results appear to over-estimate E_{IT} significantly. Both NPL and JRC used ABAQUS and infinite elements, while KTH used ANSYS and a finite sample for analysis. Further investigation is needed to resolve this significant discrepancy.

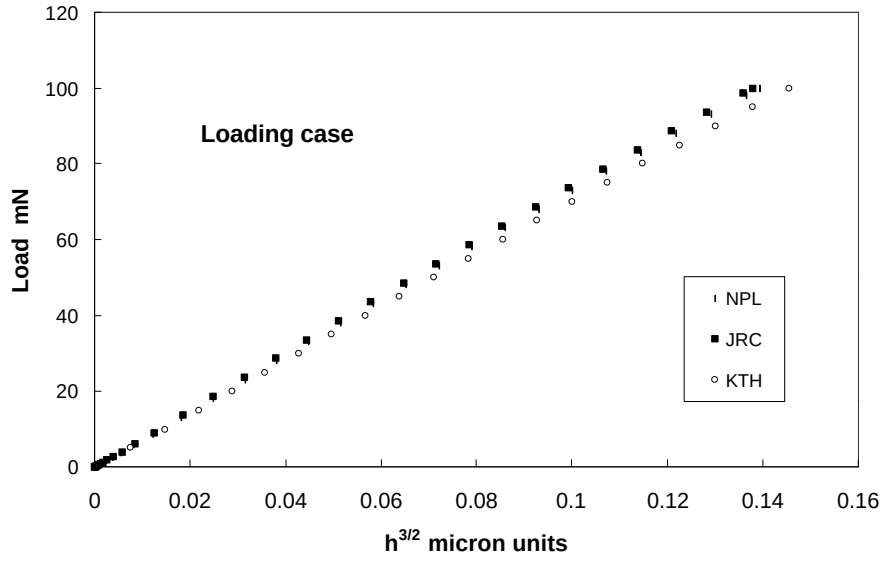


Fig.29 : FEA elastic-plastic results of load plotted as a function of $h^{3/2}$ for a 2.1 mm DLC coating applied to M2 toolsteel. $E_{IT}^c = 142.71 \text{ GPa}$, $E_{IT}^s = 240.86 \text{ GPa}$.

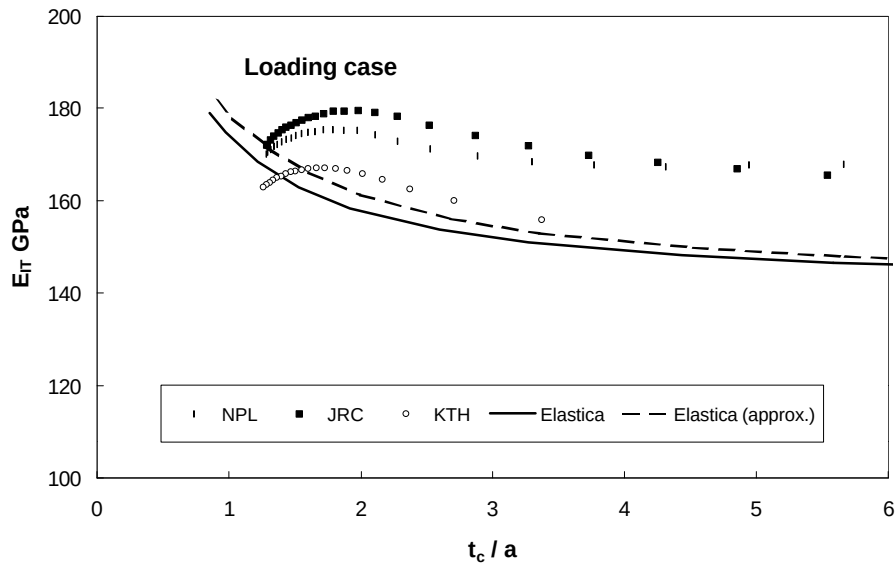


Fig.30 : FEA elastic-plastic results of E_{IT} as a function of t_c/a for a 2.1 mm DLC coating applied to M2 toolsteel. $E_{IT}^c = 142.71 \text{ GPa}$, $E_{IT}^s = 240.86 \text{ GPa}$

Fig. 31 shows the plots during unloading obtained of load versus penetration $h - h_0$ raised to the power $3/2$ predicted by elastic-plastic FEA carried out by NPL, JRC and KTH, where h_0 is the predicted value of the penetration when the load has been reduced to zero. The value of h_0 would be zero for purely elastic deformations. This type of plot might be expected to be linear when applying Hertz theory for the case of elastic behaviour during unloading as the contact is between a spherical indenter and a spherical indentation created by plasticity during the loading stage. The remarkable observation is again that the plots remain almost linear for

the case of elastic-plastic modelling. It is seen that for the case of unloading the results of NPL and JRC are almost identical while those of KTH differ slightly.

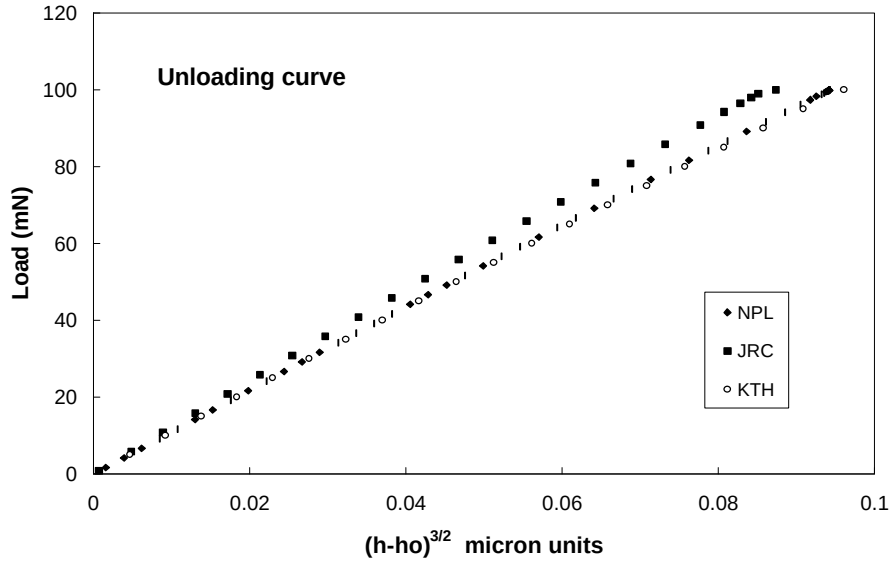


Fig.31 : FEA elastic-plastic results of unload plotted as a function of $(h-h_0)^{3/2}$ for a 2.1 mm DLC coating applied to M2 toolsteel. $E_{IT}^c = 142.71$ GPa , $E_{IT}^s = 240.86$ GPa .

On using the results shown in Fig. 31 to predict approximate values of E_{IT} using method 2 the predictions shown in Fig. 32 are obtained. For this case the local maximum values are not quite as prominent as for the loading case, and more importantly the E_{IT} values are seen to be much higher being of the same order as that for the substrate.

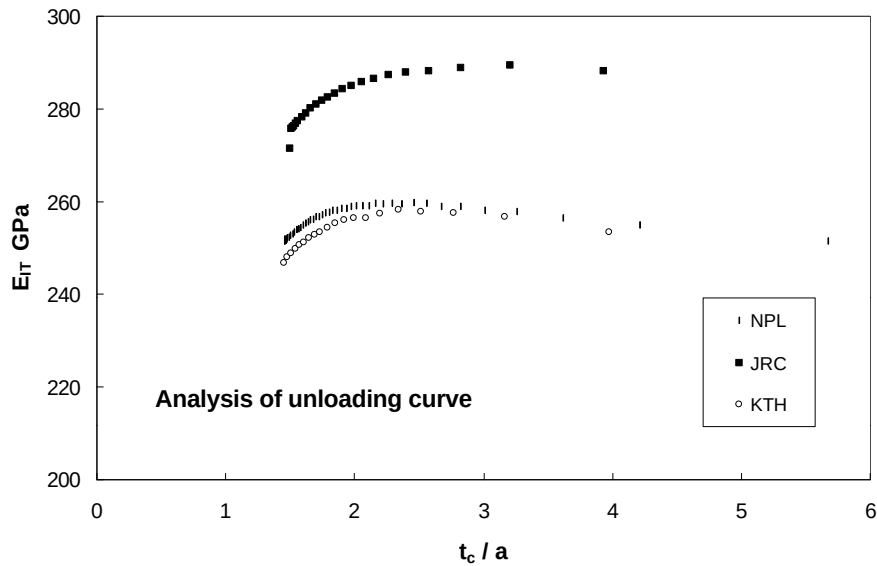


Fig.32 : FEA elastic-plastic results of E_{IT} from the unloading curve as a function of t_c/a for a 2.1 mm DLC coating applied to M2 toolsteel. $E_{IT}^c = 142.71$ GPa , $E_{IT}^s = 240.86$ GPa .

The shape of the curves shown in Fig. 32 has been shown to be highly sensitive to the selection of h_0 . By varying h_0 for each set of FEA results the curves shown in Fig. 33 result. The value of h_0 has been selected so that the initial gradient is as close to zero as possible,

which is equivalent to shifting the origin for the data given in Fig. 31 so that the linear behaviour at large indentations is linear and if extrapolated will pass through the origin. It is seen that the NPL and KTH results have the same value of E_{IT} at large indentations, and that value corresponds almost exactly to the substrate value $E_{IT}^s = 240.86 \text{ GPa}$. The KTH value tends to a value of 223.7 GPa that is close to the value of the substrate modulus. It is concluded that at large penetrations the elastic-plastic response during unloading is almost exactly Hertzian provided that h_0 is selected appropriately. This Hertzian response gives exactly the properties of the substrate when using the Hertz method of estimating E_{IT} .

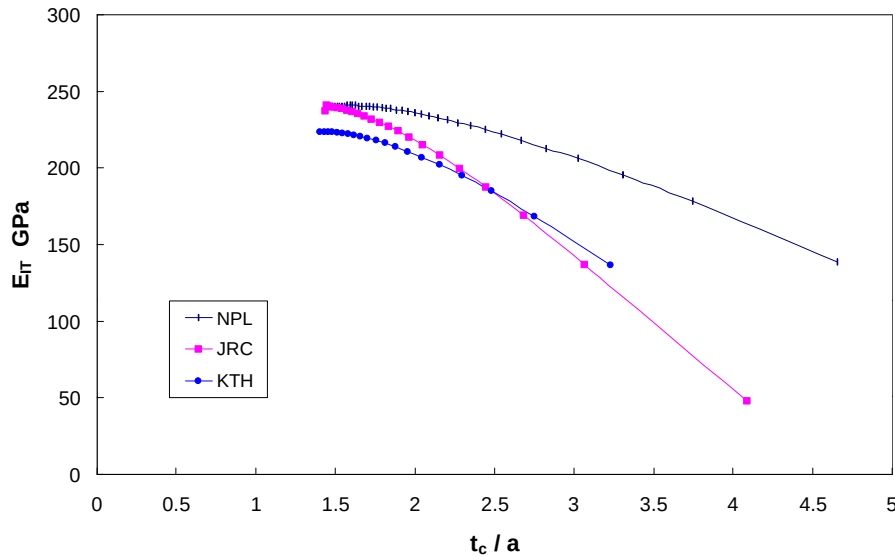


Fig.33 : FEA elastic-plastic results for E_{IT} from the unloading curves as a function of t_c/a for a 2.1 mm DLC coating applied to M2 toolsteel. Data in Fig.32 re-plotted by varying h_0 for each data set.

18. Conclusions regarding elastic-plastic modelling

1. When using conventional FEA to predict load-penetration behaviour during nanoindentation, the use of yield and plasticity parameters derived from macroscopic testing lead to unrealistic predictions. More suitable values can be obtained by fitting indentation results to predictions, thus introducing an undesirable circularity into the procedure.
2. When using the Oliver and Pharr method to estimate values of E_{IT} from the unloading portions of load-penetration data generated by FEA, the resulting values are found to depend significantly on the maximum load, and that predictions of the indentation modulus can lie outside the range defined by values for coating and substrate. Thus, for elastic-plastic FEA analyses, the Oliver and Pharr data reduction method does not lead to indentation modulus values that are a characteristic only of the coating/substrate properties and the coating thickness.

3. More reliable predictions of indentation modulus, that are independent of the maximum applied load, might arise when using a method of analysis that can be applied to purely elastic deformations during penetration. This would imply that large radius spherical indenters should be used when obtaining experimental data.
4. The Gao et al analysis for the elastic penetration of a coated substrate by a flat cylindrical indenter has been shown to be too inaccurate for use in the procedure to extract values of Young's modulus and Poisson's ratio from two values of E_{IT} obtained from the same coating/substrate system.
5. The elastic-plastic results of load-penetration during loading and unloading for spherical indenters, analysed on the basis of Hertz theory, lead to remarkable linear plots when load is plotted as a function of $h^{3/2}$ and $(h-h_0)^{3/2}$. The approximate method of analysis to predict E_{IT} (method 2) has been shown to be very sensitive technique for indicating significant differences in FEA predictions which appear to be very close when observing load penetration behaviour.
6. Reliable predictions of 2D FEA models are possible only if the appropriate elastic-plastic material properties are known. The identification of this data relevant to nanoindentation is exceedingly difficult to achieve and it is concluded that FEA modelling is unlikely in the near future to be the basis of any standardised method of measuring coating properties. The irony is that a methodology is being developed to measure elastic properties (Young's modulus and Poisson's ratio) of coatings using sharp indenters that induce elastic-plastic behaviour that can be modelled only if the elastic properties are known. If the objective is to measure these elastic properties, then the major conclusion of the modelling activities, is that large radius spherical indenters should be used at relatively low loads. Sharp indenters can be used when measuring the hardness of the coating system. There seems little prospect of attributing a hardness to the coating material alone, principally because hardness is not a material property – values depend upon the geometry of indenter used, and the thickness of the coating.

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Appendix 1 : Test case comparing ABAQUS and ANSYS

As there are several differences in the possible input to ANSYS and ABAQUS due to the differing forms of the input files it was decided to test the solvers using a simplified input which could be used unmodified except for syntax in both systems. This meant that infinite elements could not be used as they do not exist in ANSYS for elastic problems and the rigid indenter of previous work had to be replaced by one with a very large modulus for similar reasons. Plasticity is represented differently in the two systems so it was decided to compare them using a purely elastic problem.

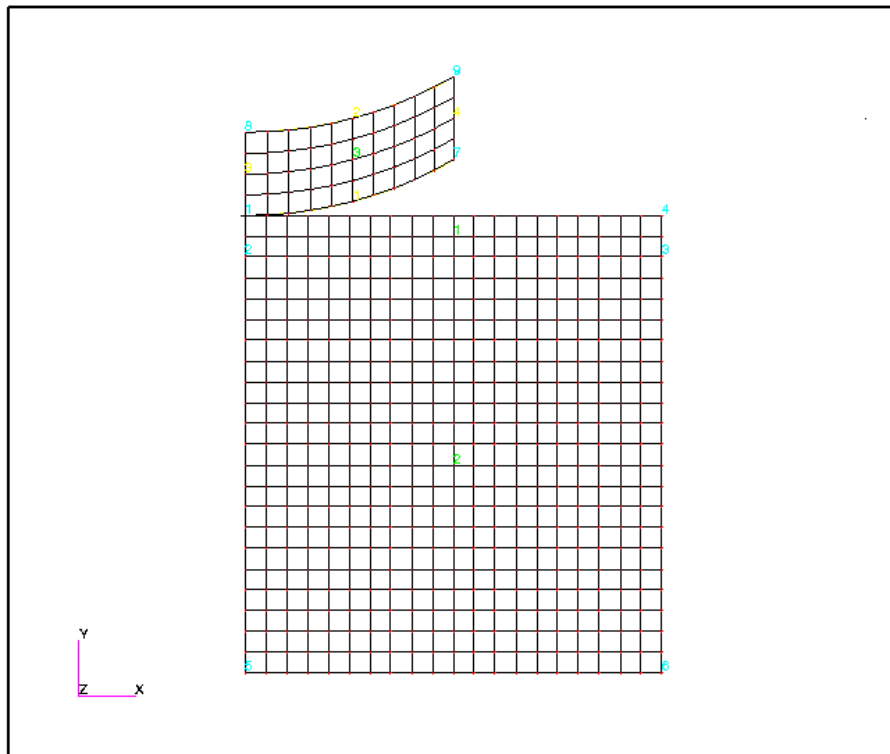


Fig. A1.1: Simple mesh used to compare ANSYS and ABAQUS solvers.

The axisymmetric mesh shown in Fig. A1.1 was used where the hemispherical indenter was given a Young's modulus E of 10000 GPa and a Poisson's ratio $\nu = 0.3$. The next two rows of elements were regarded as the coating, have properties $E = 10$ GPa and $\nu = 0.3$, and the remainder of the elements, regarded as the substrate, were given $E = 100$ GPa with $\nu = 0.3$. The lower edge of the substrate was not allowed to move downwards (y direction) and the left edge, regarded as the axis of symmetry was not allowed to move in the radial direction (x direction). The system was then stressed by loading the top edge of the indenter downwards along the axis of symmetry.

It is seen from Fig. A1.2 that that the agreement between the two solvers is very good despite the widely differing step length used in the two cases. The noticeable errors such as the lack

of smoothness in the curve are due to the relatively crude model and is reproduced by both solvers. We also have evidence from elsewhere that this agreement may be extended to far more realistic models provided they are large enough for the lack of infinite elements in ANSYS to be insignificant. Similarly, plasticity can also be reconciled in the two systems by arranging to have different but compatible hardening data in the two systems. In the present case we set out to show the two solvers give the same answer to the same problem and this has been done.

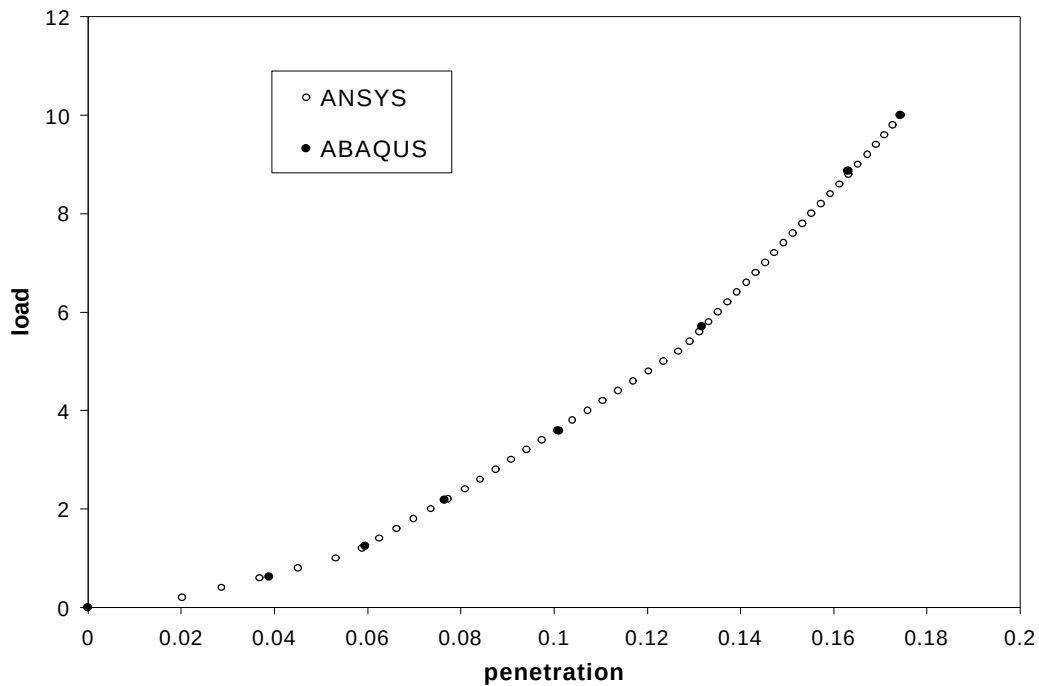


Fig. A1.2: Comparison of ANSYS and ABAQUS results for the load penetration curve for the model in Fig.1.

Appendix 2 : Plasticity values for Nickel, DLC, Gold and Aluminium

Nickel

The original intention was that the modelling tasks for INDICOAT should use bulk properties for the plasticity parameters - the only reliable source of data for homogeneous states of stress required by FEA. The result of this approach is that the yield stress (assuming von Mises) would have to be of the order of 200 MPa (a value also suggested by QMWC on the basis of bulk compression tests). The stress-strain data provided by VTT for nickel have a stress at 0.2% strain of 234 MPa (near the compression yield value), and the UTS is a mere 414 MPa at a failure strain of about 20%. The elastic properties for nickel, assuming that it is isotropic, are: Young's modulus 220 GPa and Poisson's ratio 0.31. QMWC have provided indentation data for bulk nickel based on spherical indentation, and a radius of 8 microns has been suggested for use in the FEA study. The objective here is to examine how well FEA predictions of indentation (using a rigid spherical indenter) agree with the QMWC experiments on bulk nickel. The radius of 8 microns suggested by QMWC is a compromise value as the undeformed radius of the indenter used in the experiments is 9 microns, reducing to 8.7 microns at the onset of initial observable yield, and thereafter reducing to 6.5 microns.

FEA predictions for bulk nickel were carried out at NPL using a bulk value for the von Mises yield stress of 200 MPa, and a variety of stress-strain models have been investigated. When dealing with elastic-plastic modelling, it was proposed that the work hardening behaviour of the elastic-plastic materials should be assumed to be of the form (2) or (6) in main text. When using (2) or (6) to model uniaxial data, $\bar{\sigma}$ and $\bar{\epsilon}_p$ are taken respectively as the uniaxial stress and plastic strain, and σ_Y is the uniaxial yield stress. With these interpretations the relations (2) or (6) together with the triaxial definitions (3) and (5) for $\bar{\sigma}$ and $d\bar{\epsilon}_p$ respectively may be used in FEA for multi-axial stress states encountered during indentation. The form of the work hardening relations (2) and (6) are such that the uniaxial stress vs total uniaxial strain curves have a smooth transition from the elastic to elastic-plastic regimes of deformation, as exhibited by most materials.

The following discussion is based on the form (6) involving the plasticity parameters σ_Y , σ_0 and m . The values of these parameters selected for investigative FEA on uncoated samples are as follows :

FEA run	σ_Y	σ_0	m
wh1	200	200	0.1
wh2	200	200	0.33
wh3	200	400	0.1
wh4	200	400	0.33
wh5	200	600	0.1
wh6	200	700	0.1
wh7	200	800	0.1
wh8	a bi-linear equivalent of wh5		

The stress-plastic strain relations predicted by (6) using the above values are given in Fig.A2.1. The bi-linear form wh8 (for which $\sigma_Y = 730$ MPa) has been fitted to the case wh5.

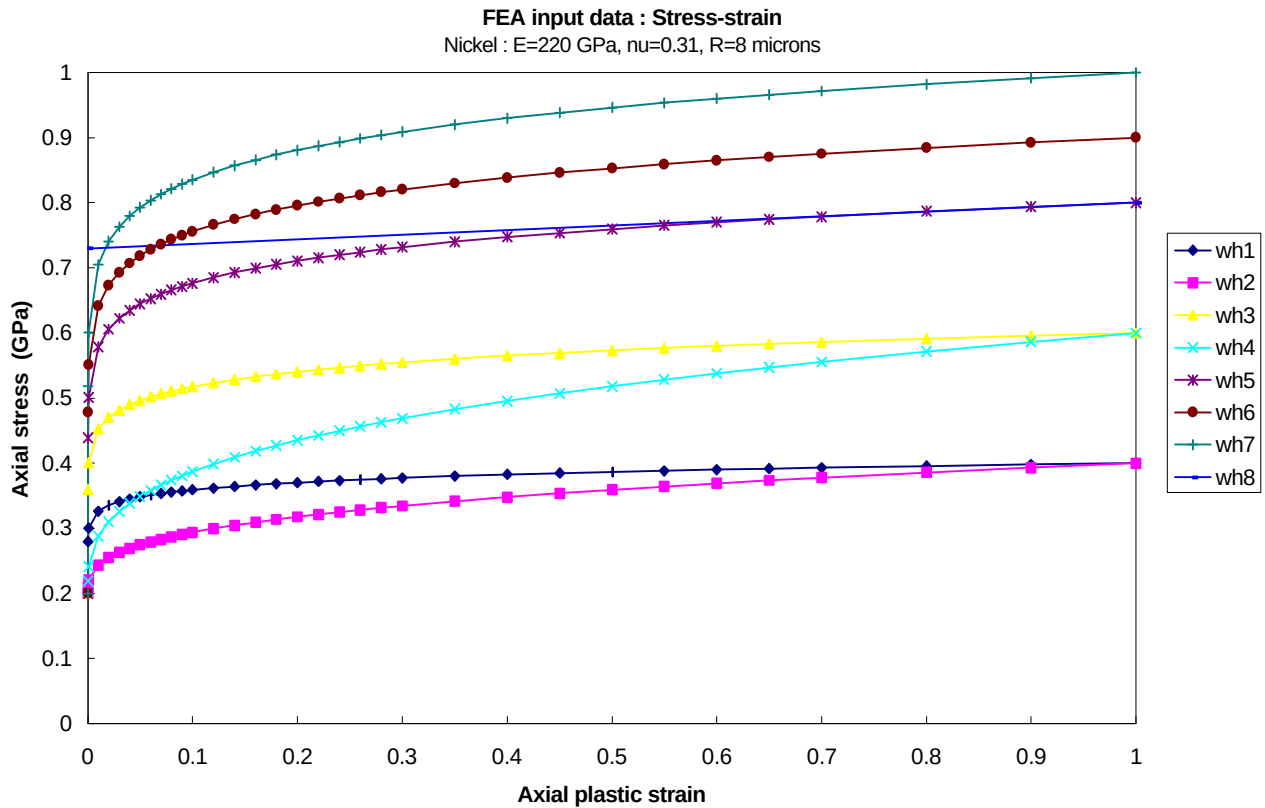


Figure A2.1 : Various stress-strain relations investigated by FEA of indentation

FEA predictions of load-penetration data have been made and compared to experimental data provided by QMWC. Fig.A2.2 shows the comparison for the cases which assume that $m = 0.1$; a value suggested by QMWC based on their analysis of indentation data using Meyer's relation. The oscillatory behaviour at penetrations beyond 75 nm arises from the use of a relatively coarse mesh remote from indenter axis that has been used to speed up calculations. It is seen that the bi-linear form (having a yield stress $\sigma_Y = 730$ MPa) agrees best with the data and yet the bulk stress-strain data for nickel (provided by VTT) is by no means bilinear in form and the compression yield stress (suggested by QMWC) is around 200 MPa. As the first approach is to use the best available bulk properties for FEA it follows from Fig.A2.2 that the results wh7 ($\sigma_Y = 200$ MPa, $\sigma_0 = 800$ MPa, $m = 0.1$) provide the best representation to the experimental data for this case.

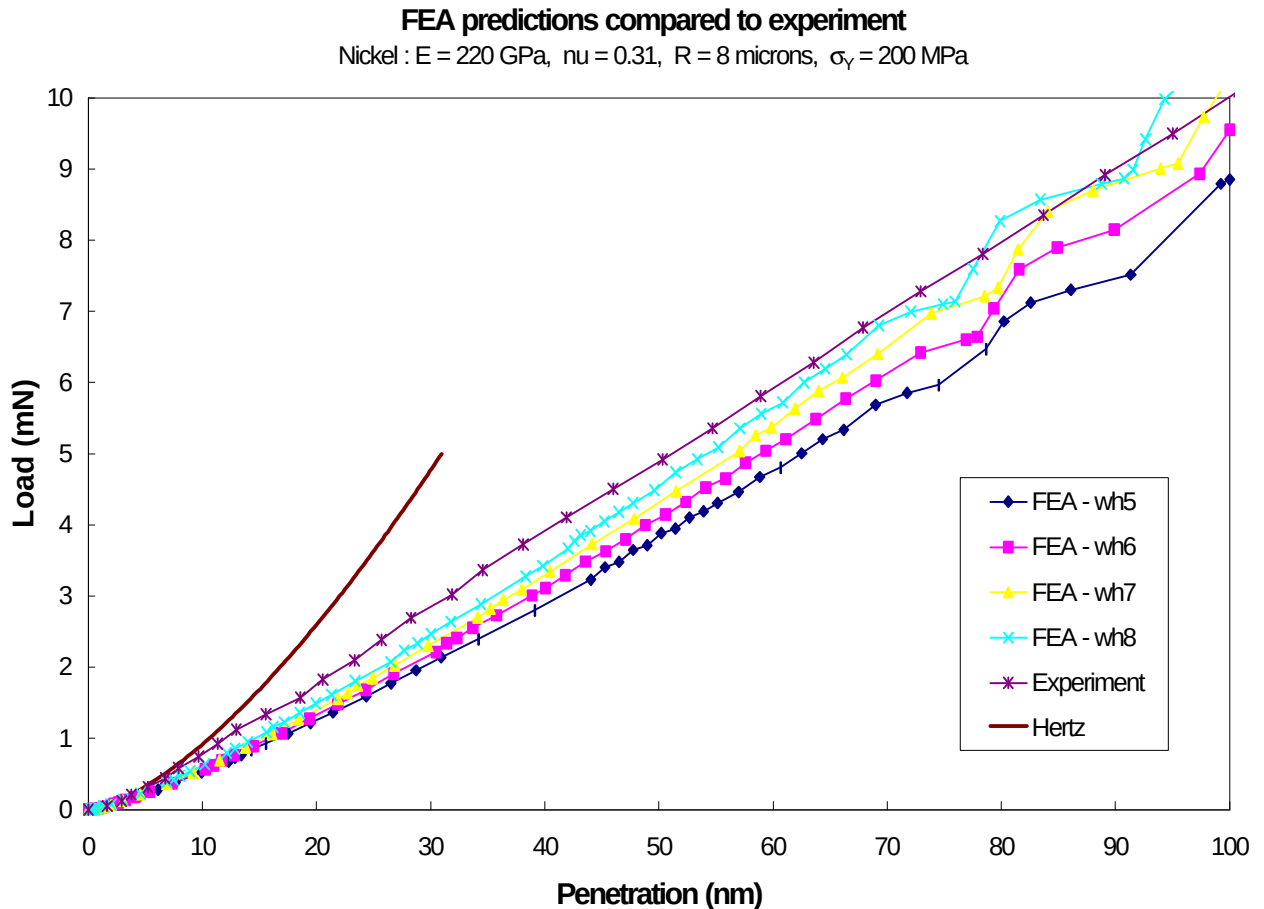


Fig. A2.2 : Comparison of predictions with experiment when $s_Y = 200 \text{ MPa}$

The experimental data given in Fig. A2.2 show an almost linear response in contrast to all FEA solutions. The experimental data have been adjusted so that the initial point passes through the origin by subtracting 0.0475 mN from each value of the load. The first three points then closely match the Hertz solution that is also shown in Fig. A2.2. Fig. A2.2 does show a lack of agreement in the penetration range 0-20 nm where all the FEA results more or less agree with one another and show much more curvature than the experimental data that are almost linear for penetrations greater than 10 nm. One interesting result is that the bi-linear form (wh8) having a yield stress of 730 MPa is very similar to wh7 having a yield stress of 200 MPa with significant work hardening.

The very recent data for nickel provided by TUC estimating initial yield using the cyclic load-unload technique for large indenter radii (57 and 138 microns) indicate that the von Mises initial yield stress has a large value (1.17 and 1.13 GPa respectively). QMWC have plotted on linear scales the experimental data in the form of mean indentation pressure vs a/R where a is the projected contact radius and R the radius of the indenter. The results show a sharp knee at a mean stress of about 2.2 GPa. This stress value is thought to relate to a yield stress somewhere in the range $1.1\sigma_Y$ to $3\sigma_Y$, i.e. between 730 MPa and 2000 MPa.

These results suggest that the yield stress being investigated (200 MPa) because of its relevance to macroscopic measurements for homogeneous stress states is much too low when modelling indentation. Thus plasticity associated with indentation could be very different to that which applies for macroscopic deformation, as suggested by QMWC. It is almost impossible to arrive quickly at reliable plasticity properties for indentation due a lack of methods to extract all the required work hardening properties from indentation data. The modellers have, therefore, faced a fundamental problem of deciding which of the following strategies should be followed:

- CASE I: assume a macroscopic yield stress of about 200 MPa and use a work hardening model of the type described. This strategy adheres to the important principle that input data for FEA should be based on measured data obtained from test methods for which the stress states are homogeneous.
- CASE II: assume an indentation yield modulus which is significantly greater than the UTS of bulk material and the bulk compressive yield stress - but without any information on work hardening as it is not possible to decide quickly which value of σ_0 to use. The best approach is probably to assume that indentation plasticity is ideal elastic-perfectly plastic characterised by the single plasticity parameter σ_Y . The second strategy introduces an element of circularity into the predictive method, i.e. we need to carry out indentation tests to obtain meaningful data for predictions. For this case the best that can be achieved is to assess whether data obtained from indentation into uncoated materials provides reliable results when the same data are used in predictions for coated samples.

As we need to assess uncertainties inherent in the modelling, it is highly desirable that both strategies are adopted and compared wherever possible.

To assist with the assessment of the second strategy three FEA solutions have been obtained for indentation into nickel using a plasticity law that is ideal elastic-perfectly plastic. For this case the plasticity behaviour is determined only by the von Mises yield stress for which three widely varying values have been chosen: $\sigma_Y = 200, 730, 2000$ MPa. For these values Elastica predicts that the mean pressure at the onset of yield is very close to 1.1 times the yield stress, consistent with the classical Tabor relation (the actual factor for nickel is 1.083).

Fig. A2.3 shows the comparison of the FEA results (for an ideal elastic-plastic model) with the Hertz solution and the QMWC experimental data for nickel. It is seen that the FEA prediction for $\sigma_Y = 730$ MPa is closest to the experimental data, and furthermore that its gradient is also closest to the experimental gradient, although the FEA predictions do exhibit a slight curvature. Increasing the value of $\sigma_Y = 730$ MPa is not really desirable as it will increase the gradient of the load-penetration curve. It is concluded that if using an ideal elastic-perfectly plastic deformation law then, of the three values investigated using FEA, $\sigma_Y = 730$ MPa is the best value to use on the basis of the results in Fig. A3. The use of an ideal elastic-perfectly plastic law avoids having to guess values for its work hardening behaviour. Meyer theory could be used to estimate the parameter m appearing in (2) but there is no means of estimating σ_0 from indentation measurements. The only way of estimating σ_0 is by using FEA to fit predictions to experiment for uncoated systems, as mentioned above.

Fig. A2.4 compares the experimental results with the FEA predictions for an ideal elastic-perfectly plastic material having a yield stress of 730 MPa with the bilinear work hardening model (wh8) having the same yield stress. The effect of the work hardening is shown to be negligible for the load range considered.

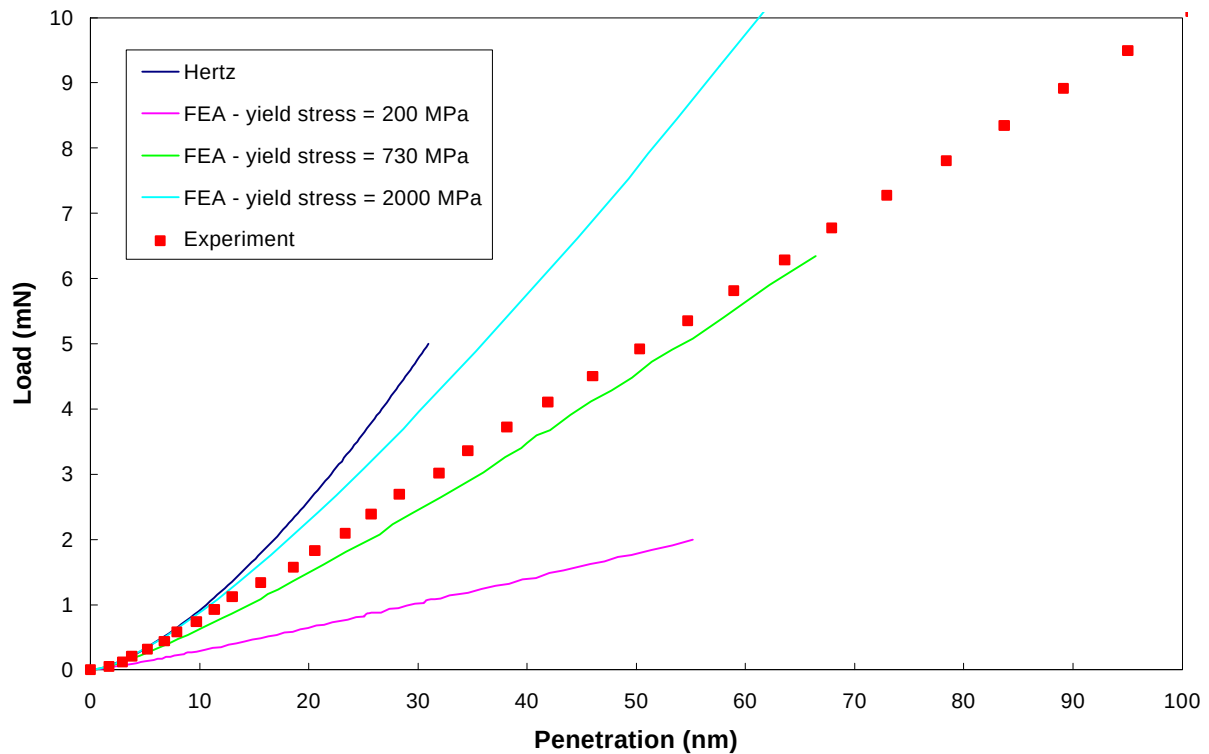


Fig. A2.3 : Comparison of ideal elastic-perfectly plastic solutions with Hertz solution and experiment.

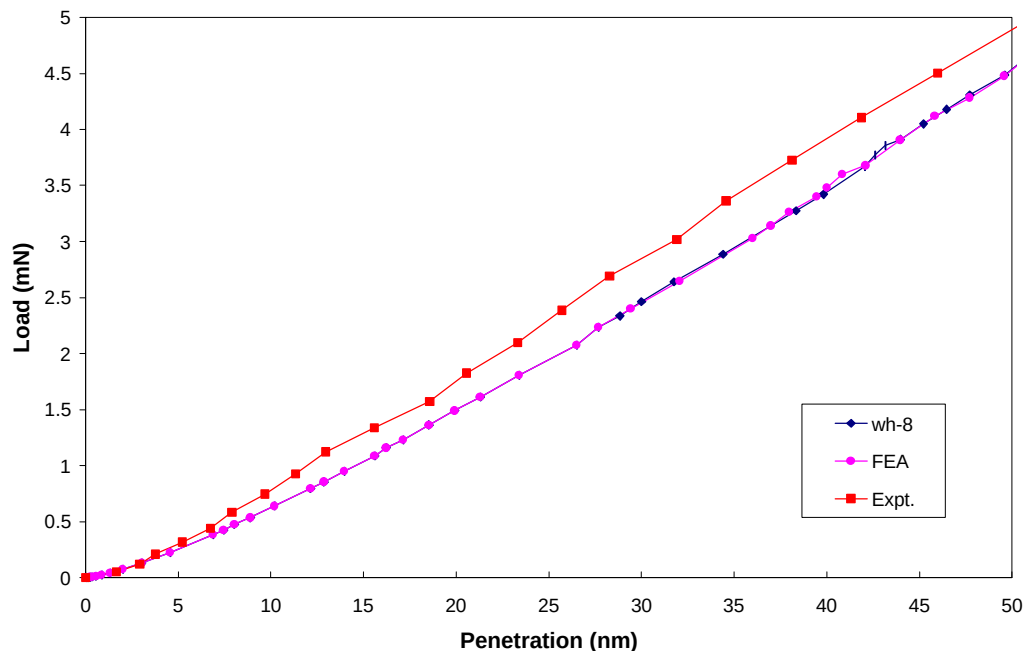


Fig. A2.4 : Comparison of ideal elastic-perfectly plastic FEA results for $s_Y = 730$ MPa with the bi-linear work hardening law and experiment.

As the value $\sigma_Y = 730$ MPa for the yield stress suggested by FEA is significantly greater than the UTS of nickel that is in the region of 414 MPa (VTT data) and the preliminary compression yield stress of 200 MPa obtained by QMWC, it has to be concluded that indentation plasticity is significantly different in nature to bulk plasticity when plasticity is modelled using von Mises methodology and homogeneous properties are assumed. Either surface phenomena are important (oxide films, etc) or new plasticity models for indentation are needed. With regard to surface phenomena TUC have reported evidence that nickel exhibits unexpected behaviour at the surface (as well as steel). The TUC data for Berkovich and Vickers measurements are given in the mid-term report. TUC have concluded that the major problem when comparing the results of FEA and experiment is that the mechanical properties (especially the work-hardening) are not uniform near the surfaces of nickel (or steel). TUC suggest that for FEA, values are used that are relevant to a surface region having a depth of $0.5 \mu\text{m}$. TUC propose that for nickel $\sigma_Y = 230$ MPa (the 0.2% value given on VTT data) and $\sigma_0 = 850$ MPa for Case I. This selection will bring the FEA predictions shown in Fig.A2.2 closer to the experimental data. For Case II the value of $\sigma_Y = 800$ MPa is suggested which will lead to the FEA results being closer to the experiments for the load range shown in Fig.A2.3 but with more deviation at larger loads.

The property values selected for nickel when carrying out FEA to assess the impact of using bulk properties obtained from conventional testing methods are:

CASE I (based on using a reasonable macroscopic yield stress):

$$E = 220 \text{ GPa} \quad \nu = 0.31$$

$$\sigma_Y = 200 \text{ MPa (less than the VTT 0.2% strain value)} \quad \sigma_0 = 850 \text{ MPa} \quad m = 0.1$$

and/or

CASE II (for an ideal elastic-perfectly plastic solid):

$$E = 220 \text{ GPa} \quad \nu = 0.31$$

$$\sigma_Y = 800 \text{ MPa.}$$

M2 Toolsteel

Using the elastic parameters for toolsteel given in the main text, and three different values of the yield stress, a finite element analysis of indentation by a rigid $4 \mu\text{m}$ radius indenter into a monolithic sample of toolsteel was carried out at NPL. The plasticity law assumed was one for an ideal elastic-plastic solid. The $4 \mu\text{m}$ radius data has been selected for the comparison (in preference to data for larger diameters) as the 3D modelling of the Berkovich indenter in the INDICOAT project assumed a tip radius of $1 \mu\text{m}$. The results of the comparison are given in Fig.A2.5 together with the Hertz elastic prediction and experimental data provided by TUC when carrying out load-unload tests. It is seen that a yield stress $\sigma_Y = 4.0$ GPa should fit the experimental data very well and this value is chosen for FEA modelling.

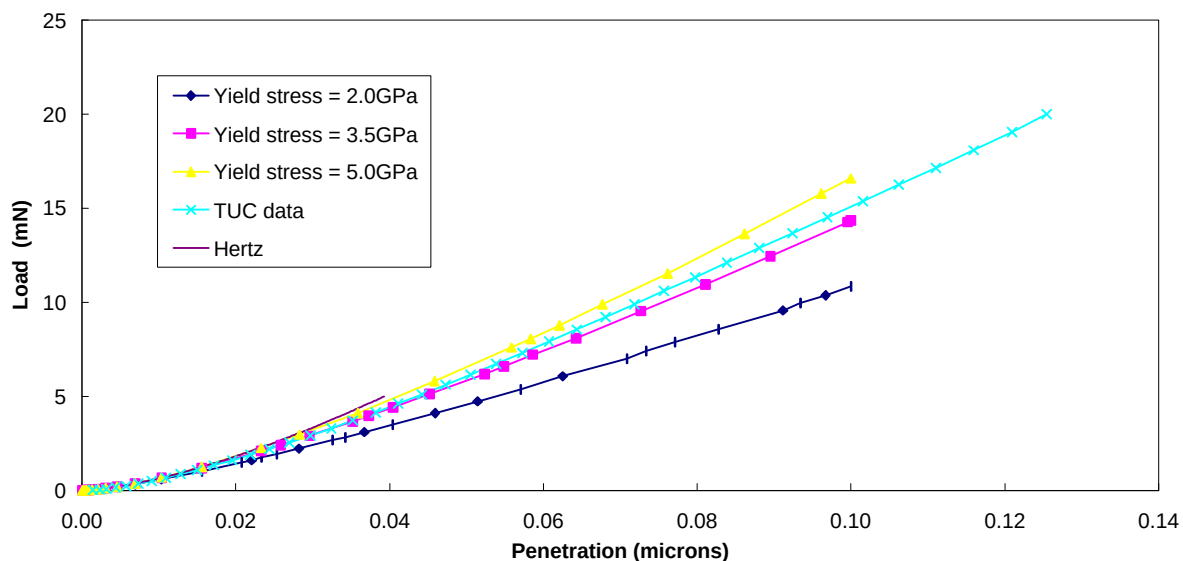


Figure A2.5 : Comparison of ideal elastic-perfectly plastic solutions at various yield stresses with the Hertz solution and experiment (TUC data) for the case of indentation into a monolithic sample of M2 toolsteel.

DLC

As no data will exist for indentation into monolithic DLC, we have no alternative but to examine the indentation behaviour into a DLC coating. Fortunately such data have been provided by TUC for the case of DLC applied to a steel substrate. The results of FEA for 3 values of the yield stress are given in Fig. A2.6 and are compared to the experimental data. The rigid indenter assumption when carrying out FEA probably explains the difference between the FEA and experimental data at low loads. The results suggest that the yield stress for DLC is at least 6 GPa which is the value selected for FEA.

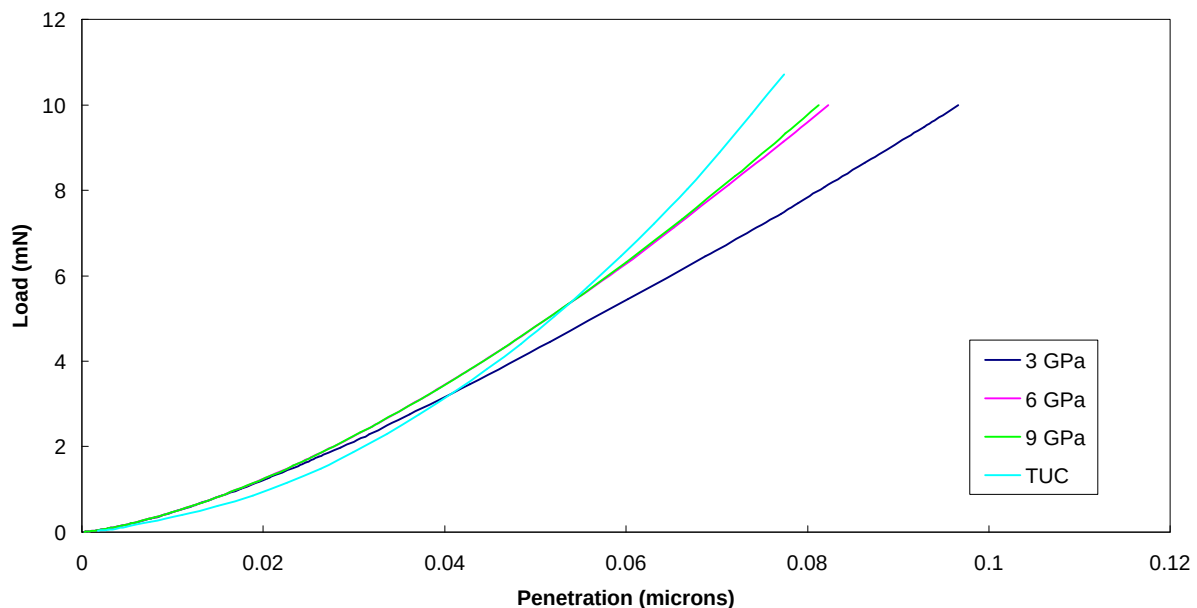


Fig. A2.6 : Comparison of ideal elastic-perfectly plastic solutions at various yield stresses with experiment (TUC data) for the case of indentation into a sample of M2 toolsteel coated with 2.1 mm of DLC.

Gold and Aluminium

For the gold and aluminium coatings it was not possible to identify any indentation data that could easily be used to compare with FEA predictions. On the basis of an examination of the experimental results for the coated systems it was decided that the best estimates of yield stress for gold and aluminium could be obtained by identifying the yield stress with about one third of the mean pressure at the onset of the knee in the mean pressure vs a/R plots. While for bulk nickel this knee is well defined, for gold and aluminium coatings the knee was less well defined. This is not a satisfactory situation, but the problem cannot be overcome within the limited resources of the project.

Acknowledgement

The valuable contributions/discussion during the preparation of this Appendix from QMWC (Andy Bushby) and TUC (Thomas Chudoba) are gratefully acknowledged.

Appendix 3 : Estimation of areas given by ABAQUS and used in Table 9

INDICOAT partners have expressed some doubt concerning the numerical values of contact area shown in Table 9. In particular the values given in the last column, which were obtained directly from the finite element analysis (ABAQUS), are larger than those in the penultimate column representing the 'rim' of the unloaded deformation. The view was expressed that this is both physically and theoretically unlikely. Additionally there is often some confusion as to the true interpretation of the 'CAREA' output variable in the ABAQUS software. In order to test this, an elastic model was set up with a mesh identical to one used in a layered specimen analysis except that the material used for both the layer and the substrate was an elastic model of nickel ($E = 220 \text{ GPa}$ and $\nu = 0.31$) and the indenter was taken as a perfectly rigid sphere of radius $10 \mu\text{m}$.

For relatively shallow indentations with the load up to 100 mN the area of contact should be given very accurately by Hertzian theory for which the contact area is given by

$$A_p = \pi \left(\frac{3P(1-\nu^2)R}{4E} \right)^{2/3},$$

where P is the load and R is the radius of the spherical indenter.

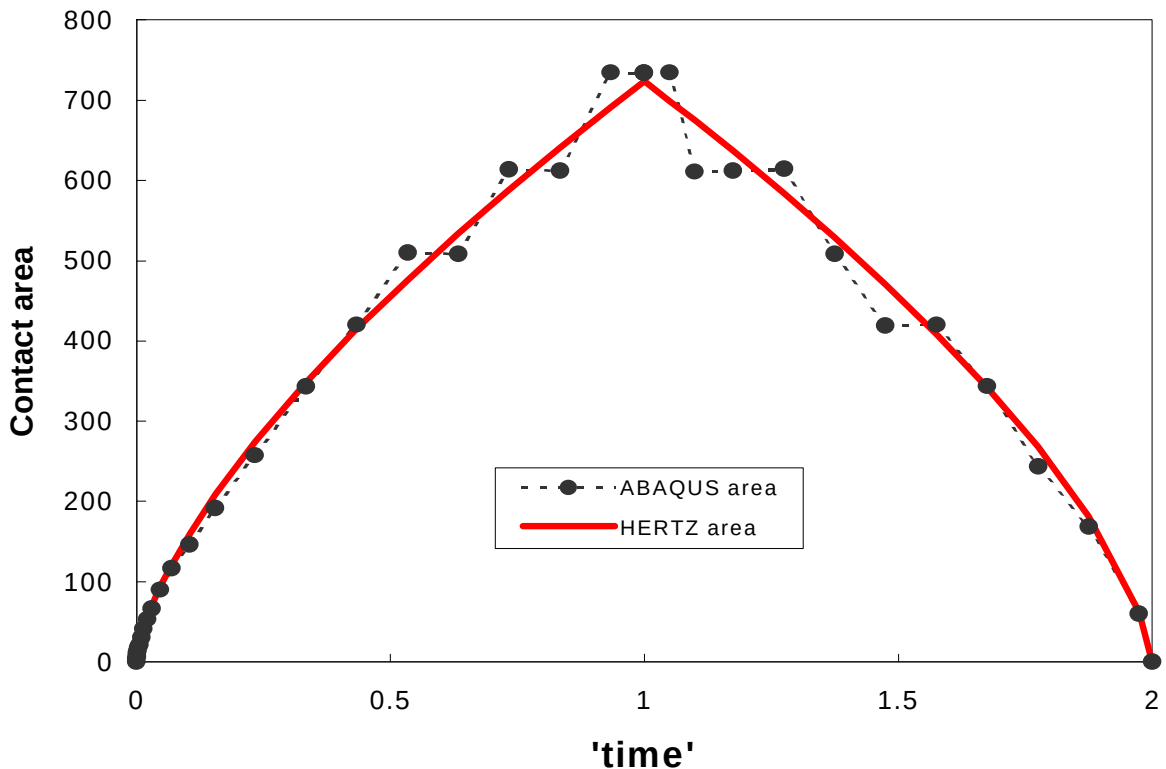


Fig. A3.1: Contact area in units of $(0.1\mu\text{m})^2$ generated from the finite element calculation using 'CAREA' and from the Hertzian formula. The load increases linearly with 'time' up to 'time' = 1 where it is 100 mN and decreases linearly to zero at 'time' = 2.

It is seen from Fig. A3.1 that 'CAREA' is certainly intended to represent the true area of contact and that its units are just the square of the length unit used in the input data (in this case $0.1 \text{ }\mu\text{m}$). The errors in the estimates of A_p increase considerably with increasing load on both the increasing and decreasing load curves. The reason for this is unclear but decreasing the mesh size reduces the effect.

The area of contact given by 'CAREA' is assumed to be the true area of contact i.e. the area of the spherical cap of the indenter in contact with the substrate but the area A_p used in the definition of hardness etc. is the projection of this area onto the plane of the undeformed substrate surface. The difference between these two area measures will be shown to be negligible compared with the uncertainty in 'CAREA' for the cases of interest to this project.

The contact area subtends a cone at the centre of the indenter. Let the included angle at the tip of this cone be 2α then the projected area $A_p = \pi R^2 \sin^2 \alpha$ and the area of the spherical cap $A = 2\pi R^2 (1 - \cos \alpha)$. Fig.A3.2 compares A_p and A up to areas of about $12.5 \text{ }\mu\text{m}^2$. Inspection of Table 9 shows that no contact area is greater than $13 \text{ }\mu\text{m}^2$.

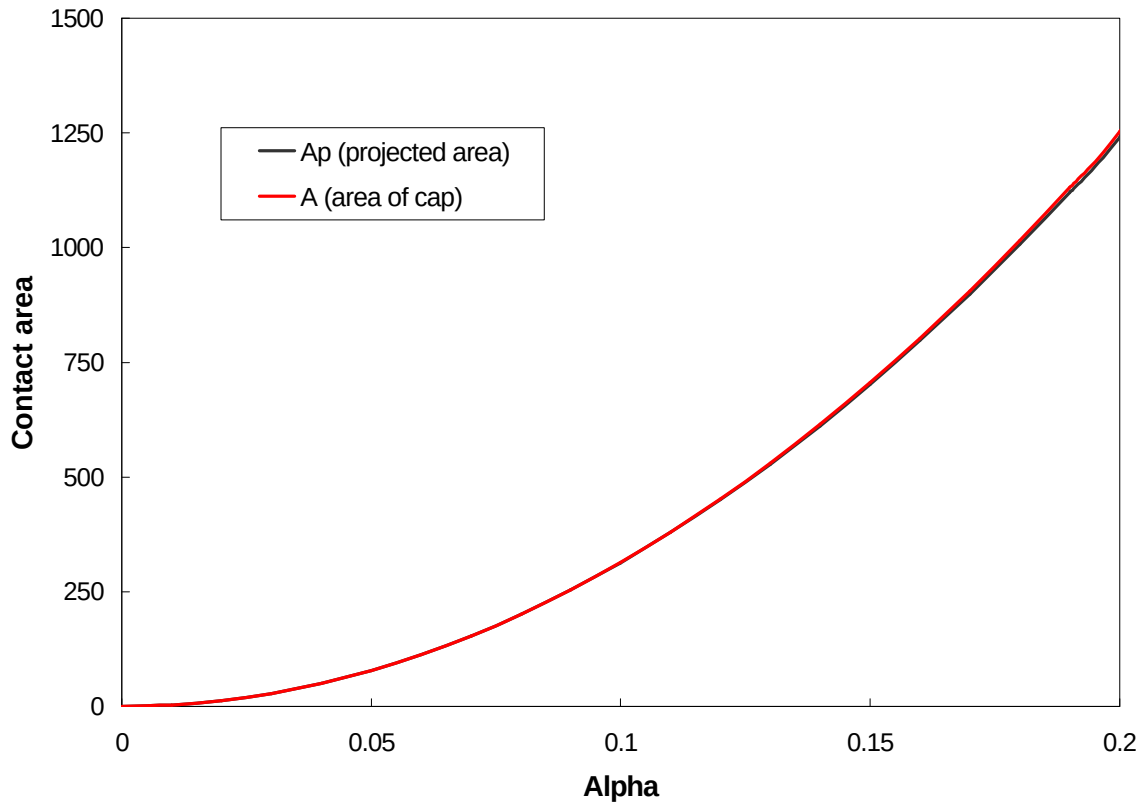


Fig. A3.2 : A and A_p in units of $(0.1\mu\text{m})^2$ against α in radians.

It is seen from the figure that at these small penetration depths this correction is negligible. What then is the major source of error in Table 9 ? Having looked at various possibilities the worst effect on the table would appear to be the interpolation of quite 'noisy' data. The estimates of area using the definitions (1) and (3) in Section 14 are derived from squares of estimates of R_1 and R_3 which are in turn derived from deformation profiles obtained from

finite element analysis. Looking again at the data reveals that this process could introduce uncertainties of about 5%. The estimate of area using definition (2) is obtained from equations (16) and (17) which rely on the assumption that the parameter $\alpha = 0.75$ that is the usual value used but it has no rigorous justification for elastic-plastic deformations. The area estimate based on definition (4) relies on finite element estimates of contact area versus load using the 'CAREA' function as exemplified in Fig. A3.3.

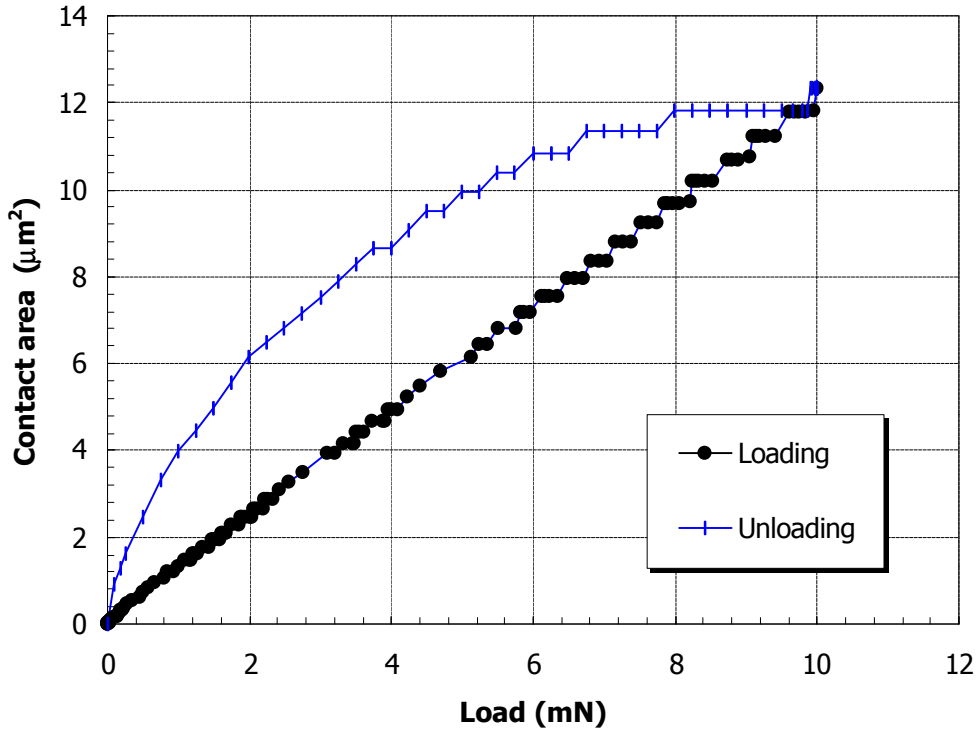


Fig. A3.3 : Contact area in units of μm^2 against load in mN for a $5.12\mu\text{m}$ layer of gold on nickel with a $10\mu\text{m}$ radius rigid indenter.

It is seen from the figure that one might sensibly take either the last point $A_p = 12.35 \mu\text{m}^2$ or the penultimate point $A_p = 11.83 \mu\text{m}^2$ as estimates of the contact area at 10 mN. This changes A_p by about 5%, quite enough to reverse the numerical order of estimates (3) and (4) in Table 9.

An interesting conclusion, when comparing Fig. A3.1 for a purely elastic case with Fig. A3.3 for an elastic-plastic model (with considerably more elements), is that the unloading curve in Fig. A3.3 is indeed elastic in form whereas the loading curve is very definitely plastic.