International Journal of Solids and Structures 80 (2016) 128–136

Contents lists available at ScienceDirect

International Journal of Solids and Structures

journal homepage: www.elsevier.com/locate/ijsolstr

A methodology for obtaining plasticity characteristics of metallic coatings via instrumented indentation

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ARTICLE INFO

Article history:
Received 7 April 2015
Revised 11 September 2015
Available online 6 November 2015

Keywords:
Indentation
Finite element analysis
Coatings
Plasticity

ABSTRACT

A methodology is presented for inferring the yield stress and work-hardening characteristics of metallic coatings from indentation data. It involves iterative use of FEM modelling, with predicted outcomes (load–displacement relationships and residual indent shapes) being systematically compared with experimental data. The cases being considered are ones in which the indenter penetration depth is a significant fraction of the coating thickness, so that the properties of the substrate, and possibly of the interface, are of significance. The methodology is thus suitable for the testing of thin coatings. In the present work, the coatings were in fact relatively thick (hundreds of microns) and the (spherical) indenter penetration was a substantial fraction of this. In this way, the basic validity of the methodology could be investigated with minimal complications from effects related to microstructure, oxide films, surface roughness etc. Furthermore, the properties of both coating and substrate (in the through-thickness direction) were established separately via conventional compression testing. The systems studied were copper (yield stress ~15 MPa) on stainless steel (yield stress ~350 MPa) and vice versa. Both exhibited significant work hardening. It is concluded that the methodology is basically reliable, with relatively good sensitivity and resolution, although this does depend on several factors, which are highlighted in the paper. It is unlikely to be suitable for very thin (sub-micron) films, but should be reasonably accurate for coatings of thickness down to a few microns.

1. Introduction

Instrumented indentation is routinely used for obtaining Young's moduli of materials from the load–displacement curve during unloading (elastic recovery). The procedure is also applied to coatings. If the penetration depth of the indenter is small compared with the coating thickness, then this situation is no different from that with a bulk sample. There is, however, the issue of how "small" should be defined in this context. A "rule of thumb" figure of 10% is often used, although there is no clear theoretical basis for this and it seems likely that the ratio of Young's moduli of coating and substrate will affect the outcome. The most straightforward approach, proposed by Jennett and Bushby (Jennett and Bushby, 2001), involves indenting to a range of depths and obtaining the 'combined' modulus of coating and substrate, as a function of the ratio of the penetration depth to the coating thickness, h/t. The value of E for the coating is then found by extrapolating back to $h/t = 0$. Since there is no well-defined functional form for the extrapolation, at least some measured moduli are needed for relatively low h/t values: obtaining reliable values for these, particularly with thin coatings, is, of course, the central problem. Nevertheless, the approach is clearly preferable to solely relying on data from one or two very shallow indents.

Various analyses and methodologies taking account of the presence of the substrate have been developed. For example, Doerner and Nix (Doerner et al., 1986) included a term for the substrate in their reduced Young's modulus equation. However, the scaling constants used are only appropriate for specific cases. King (King, 1987) presented a modified solution, using FEM, to arrive at an equation for the reduced Young's modulus, later validated by Saha and Nix (Saha and Nix, 2002). Gao et al (Gao et al., 1992) used a moduli perturbation method to develop a closed-form solution for the reduced Young's modulus of a coating, later shown to be inaccurate when the mismatch between Young's moduli of coating and substrate is large (Chen and Vlassak, 2001). Xu and Pharr (Xu and Pharr, 2006) suggested a modification to make it more accurate, verified using FEM. Investigations have also been made (Tricoteaux et al., 2010) into the effect of machine compliance in this context. In general, it is possible, using such approaches, to obtain a reasonably reliable value for the stiffness of a coating via indentation.

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http://dx.doi.org/10.1016/j.ijsolstr.2015.10.029
0020-7683/

However, the problem is clearly more complex when plasticity (and/or creep) is involved. Of course, this also presents much greater challenges for bulk samples than does stiffness. This statement does not really apply to hardness, which is defined in terms of an indentation response. However, hardness is not a fundamental or well-defined material property, since it depends, not only on yield stress and work hardening characteristics, but also on indenter shape and in some cases on indentation depth. While there has been a lot of work on the measurement of hardness for thin surface coatings, it is therefore excluded from the current discussion.

The difficulty in obtaining plasticity and creep characteristics from indentation experiments arises from the complex and continuously changing stress and strain fields under the indenter. As a consequence of this, while there have been many attempts to identify methodologies involving the use of analytical equations for evaluation of these characteristics from indentation data, it now seems clear that none of them are consistently reliable. Reliable inference of these characteristics requires these fields to be taken into account in a quantitative manner. The most suitable tool for this is the finite element method (FEM), which has been widely applied to indentation testing with the objective of obtaining information about plasticity parameters (Bouzakis and Vidakis, 1999, Bouzakis et al., 2001, Tunvisut et al., 2001, Liu et al., 2005, Pelletier, 2006, Yonezu et al., 2009, Pohl et al., 2014). In particular, a consistent methodology, based on iterative use of FEM, has recently been developed that allows the yield stress and work hardening rate (Dean et al., 2010) and the (primary and secondary) creep parameters (Dean et al., 2013) to be inferred from experimental indentation data. These capabilities, for which customised user-friendly software packages are currently being prepared, would be further enhanced if the methodology could be extended to (relatively thin) coatings, for which the effect of the substrate on the indentation response cannot be ignored (i.e. cases for which the penetration depth is not “small” relative to the coating thickness).

There have also been various attempts to establish the maximum h/t ratio for which it is acceptable to treat a coating as a bulk material for evaluation of plasticity parameters (Lebouvier et al., 1985, Sun et al., 1995, Panich and Sun, 2004, Gamonpilas and Busso, 2004) (and hardness (Cai and Bangert, 1995, Xu and Rowcliffe, 2004)). In general, values of around 10% are often quoted, although it is clear that there is considerable scope (Chudoba et al., 2002, Cleymand et al., 2005) for variations in this figure (more so for plasticity than for Young’s modulus) between different systems, which is unsurprising in view of the larger number of material properties (for coating and substrate) expected to be relevant. It appears that no rationale has been developed so far that has led to a reliable analytical expression for this “critical ratio”.

In the present work, a methodology is presented for extraction of plasticity parameters (including work hardening characteristics) of coatings from indentation data, applicable for any ratio of indentation depth to coating thickness (irrespective of coating and substrate properties). A spherical indenter has been used. Of course, unlike a Vickers or a Berkovich, or indeed a cone, this shape is not self-similar, which has implications for the development of the strain field. It might be argued that the changing strain field encompasses a wider range of conditions for a shape that is not self-similar, and that this is beneficial. Of course, a sphere is also transversely isotropic (as indeed is a cone), which allows a 2-D model to be employed (provided the sample is also transversely isotropic). In practice, it might be advantageous (in terms of converging rapidly to a unique solution) to employ at least two different indenter shapes in the same study. Nevertheless, in the present work only a spherical indenter has been employed.

An important point in the context of the current study concerns effects of scale. The work involves use of very coarse (thick) coatings (and large indenters). This is done so as to allow use of coating materials for which the properties (in the direction of indentation) can be obtained by conventional testing. The mechanical modelling (and indeed, at least in principle, the actual behaviour) is scale-independent – so that, for example, the stress and strain field around a spherical indenter that has penetrated a (bulk) sample to 10% of its radius is the same whether that radius is 10 μm or 10 mm. This allows universal deductions to be made from experiments carried out on a very coarse scale. In practice, scale effects may arise if the characteristic length scales of the testing become comparable to those relevant to the micro-mechanisms of deformation, such as the distances between dislocations or the grain size. However, the current work is focused on the extraction of “continuum” (macroscopic, or bulk) properties and these should, of course, be scale-independent.

2. Experimental procedures

2.1. Materials and microstructures

Two materials were used – an oxygen-free, high conductivity (OFHC) copper and an austenitic stainless steel (AISI 304). The steel microstructure is shown in Fig. 1(a), demonstrating that the grain size is ~30–50 μm. The copper, received in extruded rod form, was annealed inside vacuum-sealed ampoules for 2 h at 800°C, to stimulate recrystallisation and reduce the hardness. The resulting grain structure is shown in Fig. 1(b), where it can be seen that the grain size is ~100 μm. (These grain sizes are sufficiently coarse to create difficulties in ensuring multi-grain interrogation during conventional nanoindentation, but in the present work the indent diameters were of the order of at least several hundred microns.)

2.2. Macroscopic, uniaxial compression testing

In many systems, particularly for thin coatings, mechanical properties in the through-thickness direction (normal to the free surface) are largely unknown. However, it is these properties that dominate the indentation response and so it was important to obtain them for validation of the methodology. Specimens for uniaxial compression testing were machined from the as-received stainless steel and the annealed copper rod. Cylindrical specimens (12 mm in height and 10 mm in diameter) were tested in compression, at room temperature, using a 100 kN ESH servo-hydraulic mechanical test machine. The ends were lubricated with molybdenum disulphide, to minimise barrelling. Displacements were measured using a scanning laser system, with a resolution of ~3 μm.

2.3. Soft (copper) coatings on a hard (steel) substrate

Thin discs of Cu (300 μm thick) were machined (sliced) from extruded rods by electro-discharge machining (EDM). Some of these were polished down, to generate discs about 165 μm thick. Both types of disc were attached to stainless steel substrates, using a high-strength Araldite adhesive. In order to minimise the thickness of the adhesive layer, it was first heated over a hot-plate. The consequent reduction in viscosity allowed a thin, continuous layer (~10 μm thick) to form. It was cured for 24 h at room temperature.

Samples were indented using a custom-built, screw-driven mechanical test machine, with a load capacity of 2.5 kN. The indenter was a commercially-available 3 mm diameter sphere of tungsten carbide (a WC cermet). The indenter was therefore very large, at least in comparison to more conventional micro- and nanoindenters, giving benefits in terms of sampling a representative volume, being immune to errors associated with oxide films, surface roughness etc., and creating depth data with excellent relative accuracy. (Outcomes