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Identification of elastic–plastic anisotropic parameters using instrumented indentation and inverse analysis

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Abstract

Mechanical responses of thin films or coatings often display anisotropic behaviors because of their unique microstructures. However, their small size scales can also make determination of material properties difficult. The present paper introduces a simple yet versatile procedure with advanced data interpretation scheme to identify key anisotropic parameters. This procedure utilizes instrumented indentations and an inverse analysis to extract unknown parameters of elastic–plastic transversely isotropic materials. In particular, it post-processes load–displacement records of depth-sensing indentations to obtain best estimates of Young's moduli and yield stresses along longitudinal and transverse directions, respectively. Major advantages of this method are the minimal specimen preparations and the straightforward testing procedure. To enhance the accuracy, the method utilizes two differently profiled indenter heads, spherical and Berkovich. Prior to actual testing, detailed simulations were performed to verify the method's applicability and robustness. In the experiment, a thermally sprayed NiAl coating which possesses process-induced anisotropic features is considered. The load–displacement records of spherical and Berkovich nano-indentations are post-processed with the proposed inverse analysis scheme. The estimated results predict dissimilar responses along the longitudinal and transverse directions. Separate tests are also conducted with micro-indenter heads under larger loads. They demonstrate lesser anisotropic effects but with more compliant responses. These results are attributed to the unique morphology of thermally sprayed coatings, which inherently exhibit size and anisotropic effects.

Keywords: Elastic–plastic transversely isotropic; Inverse analysis; Kalman filter; Thin films; Thermally sprayed coatings; Instrumented nano-indenter; Spherical and Berkovich indenters

1. Introduction

Thin films and coatings have been widely used in various applications such as large-scale integrated circuits, electronic packaging, sensors, optical films as well as protective and decorative coatings (e.g., Elshabini-Riad and Barlow, 1976). For effective designing and accurate evaluation of their structural integrity, determinations of their mechanical properties are vital for product development. Fabrication techniques for thin film/coating include physical vapor deposition, chemical

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vapor deposition, chemical methods (e.g., electroplating), and thermal spray (e.g., plasma spray, combustion spray). Due to these fabrication processes and resulting microstructures, thin films/coatings often exhibit anisotropic mechanical responses. Crystalline/columnar microstructures observed in vapor deposited materials and lamellar microstructures by solidified splats in thermally sprayed coatings are some good examples. Fig. 1 shows a cross-section of atmospheric-plasma sprayed Ni–5wt.%Al coating containing splat and pore/crack morphology. These geometrical features are predominantly aligned along the transverse/horizontal direction (i.e., perpendicular to spray direction) to induce different responses along the longitudinal and transverse directions. Here the longitudinal is defined as the normal to the coating surface or the spray direction while the transverse refers to the two in-plane directions. Although these anisotropic responses of thin films/coatings have been well known, due to their small scales, there are limited experimental procedures to measure their inelastic responses.

For *isotropic* characterizations of thin materials, several notable experimental procedures have been introduced in recent years. Many of them utilize depth-sensing indentations (Fischer-Cripps, 2000). For Young's modulus and hardness, Oliver and Pharr (1992) and Doerner and Nix (1986) proposed methods based on maximum loads and unloading slopes. Field and Swain (1993) also investigated yield stress and strain hardening characteristics. Suresh and Giannakopoulos (1998) and Giannakopoulos and Suresh (1999) introduced effective procedures to quantify residual stresses and elastic–plastic properties of homogeneous materials. Guidelines and assumptions needed for accurate measurements were also discussed by Cheng and Cheng (1999) and Venkatesh et al. (2000). Nakamura et al. (2000) proposed an inverse analysis procedure and instrumented micro-indentation to estimate properties of graded medium using two differently sized spherical heads. Dao et al. (2001) utilized a forward–reverse method and instrumented sharp indentation to characterize elastic–plastic isotropic properties. Chollacoop et al. (2003) improved this method by employing two different indenters with Berkovich and cone heads. In addition, specialized experimental techniques such as bulge testing, substrate curvature, X-ray diffraction, micro-Raman spectroscopy, and electron diffraction contrast imaging, have been developed to determine mechanical properties of both free-standing films and films bonded to substrates (Vinci and Vlassak, 1996). While most of them did not consider the anisotropy, some have considered such effects (Vlassak and Nix, 1994; Vlassaka et al., 2003; Meng and Eesley, 1995; Wang and Lu, 2002). These, however, investigated only *elastic* properties of anisotropic based on indentations along different orientations. For significantly larger specimens, such as rolled-aluminum alloy sheets, there are several studies which considered elastic–plastic anisotropic properties (e.g., Barlat et al., 1997; Wu et al., 2003).

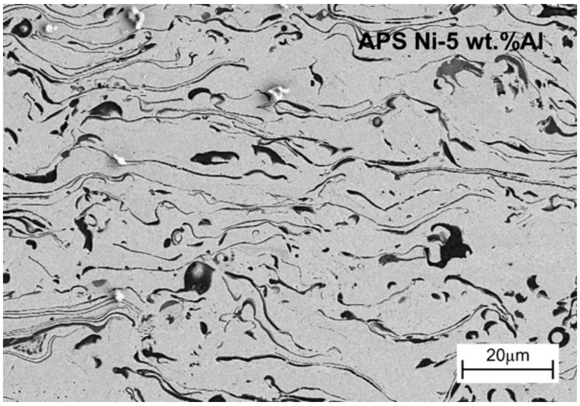

Fig. 1. SEM micrograph showing cross-section of air plasma sprayed Ni–5wt.% Al. The vertical direction corresponds to spray direction (longitudinal). Very dark regions correspond to cracks and pores.

Clearly, a simple examination of load–displacement records from instrumented indentations does not reveal any anisotropic effects. In fact, no explicit correlation between such measurements and anisotropic properties exists. This is the reason an inverse analysis is needed to extract the properties through suitable post-processing of measured records. In general, such processing schemes are necessary when direct relations between measurements and unknown variables are not available or obvious. In the past, various inverse approaches have been utilized in mechanics of material problems. For examples, it was used to detect and quantify critical damage mechanisms like cracking, delamination, and corrosion in aerospace and civil structures (e.g., Chang, 2000). However, an inverse analysis technique is not valuable unless it satisfies convergence and consistency conditions. In many cases, direct implementations of inverse analysis techniques lead to inaccurate solutions due to ill-posed conditions (i.e., non-convergence to unique