

A Special Method to Evaluate the Strong Adhesion between Brittle Coating and Ductile Substrate

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Abstract. The evaluation of the interfacial adhesion of coating system has always been a rough task. In this paper, a special testing method of cross-sectional indentation is applied on a model coating system, i.e. electroplated chromium on a steel substrate which is generally regarded as an example of materials pair with strong adhesion. Based on fractography analysis with SEM and interfacial stress simulation with FEM, it is found that interfacial shear stress may induce coating spalling. More interestingly, spalling location is sensitive to substrate pretreatment process. This shows the feasibility of cross-sectional indentation to distinguish interfacial strength at a high level.

Introduction

The interfacial adhesion is of importance for coating system when considering its reliability [1]. However, the evaluation of interfacial strength is a great challenger for the researchers due to the complexity of the loading and materials conditions of interface cracking and coating spalling [2]. More embarrassingly, interface cracking and coating spalling are sometimes difficult to induce by conventional methods such as tensile strain or scratch testing for brittle coating/ductile substrate composite systems with strong adhesion. For example, conventional tensile test can only be applicable to interface of relatively low strength, say, under 70MPa.

Recently, a special testing method of cross-sectional indentation has been applied to examine the interfacial fracture properties of brittle coating or ductile coating on brittle substrate [3,4,5,6]. Due to the loading on the substrate and the effect of edge chipping, the deformation mismatch between coating and substrate is accelerated. In this case, interface cracking and coating spalling are easily induced. This paper demonstrates the feasibility of this method to evaluate strong adhesion between brittle coating and ductile substrate.

Experimental

The material considered in this work consists of an electroplated chromium coating with the thickness 80-130 micrometer on a steel substrate. Such a coating system is generally regarded as an example of materials pair with strong adhesion. In order to reveal the feasibility of cross-sectional indentation to distinguish interfacial strength at a high level, two different substrate pretreatment processes (I and II) are used. Process I refers to conventional pretreatment and Process II a new developed pretreatment, the latter of which is affirmed to have better adhesion in practice.

Fig.1 illustrates the schematic of cross-sectional indentation procedure. The cross-section of each sample is prepared by refined grinding to reveal the interface. A materials testing fixed with a indenter with cone angle of 90 degree and tip radius of 50 micrometer is used. The loading velocity of the indenter is limited constantly at 0.04mm/min. Before indentation, sample surface is glued with adhesive tapes chiefly made up of copper to collect the spalled coating chips. Fracture surfaces of the chip side and substrate side are inspected by SEM with EDS after indentation.

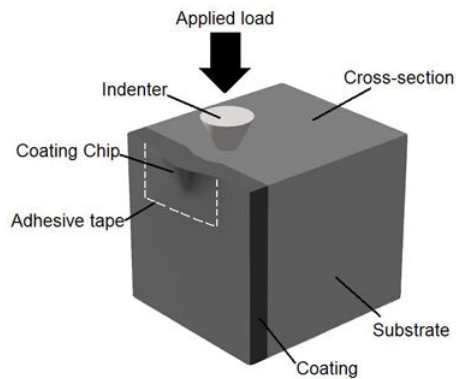


Fig.1. Schematic of indentation procedure

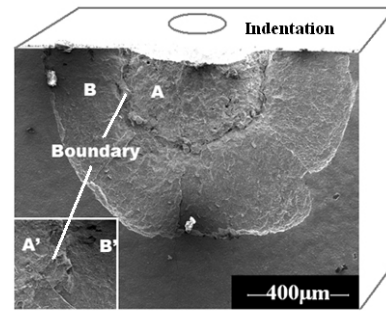


Fig.2. SEM picture of naked substrate side and coating chip side (process I)

Experimental Results

Cross-sectional indentation may induce coating spalling regardless of coating thickness and substrate pretreatment process. However, spalling location depends on substrate pretreatment process, i.e. interfacial adhesion strength.

For process I, as indicated in Fig.2, naked substrate surface exhibits interfacial fracture surface (A) and coating fracture surface (B) distinguished by a boundary curve. Image at the left bottom corner of Fig.2 shows coating chip side and exhibits the surface A' and B' corresponding to A and B, respectively. Fig. 3 and Fig.4 show the high magnification picture and mapping spectra for the elements corresponding to surface A and A' of process I. It may be noted from Fig.3 that some of Cr debris remains on the exposed substrate and most of coating chip surface is covered by Fe element.

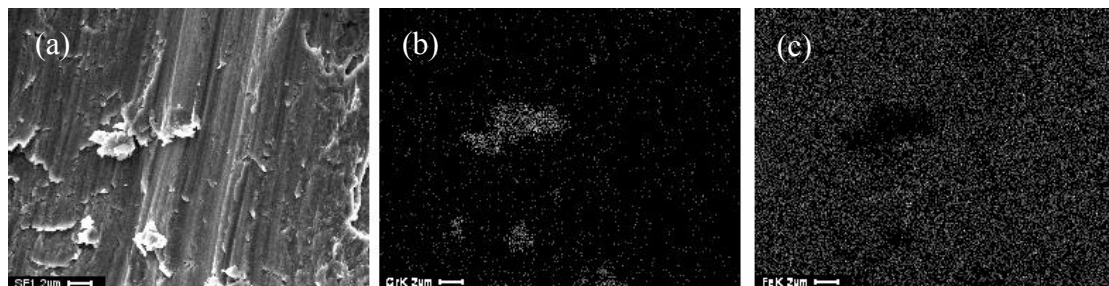


Fig.3. SEM picture (a) and mapping spectra for the elements Cr (b) and Fe (c) of the naked substrate side (process I)

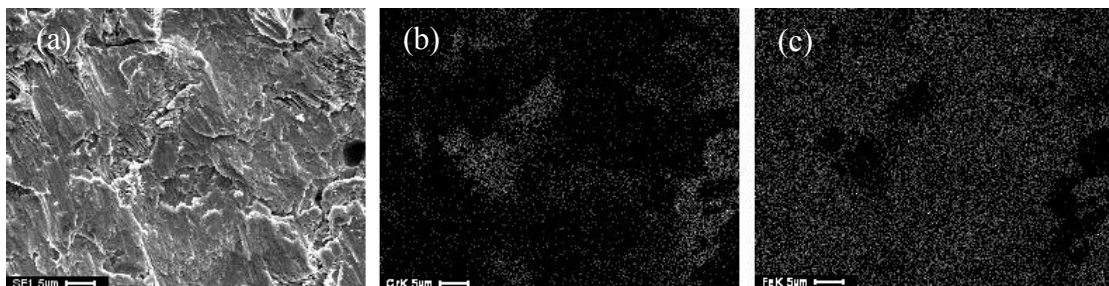


Fig.4. SEM picture (a) and mapping spectra for the elements Cr (b) and Fe (c) of the coating chip side (process I)

Moreover, the slippage evidence can be easily found in Fig.3 (a) and Fig.4 (a). Compared to the coating, the substrate is softer and more ductile. It can be deduced that interfacial slip occurs during

coating spalling. In this case, Cr debris behaves as a cutting tool to substrate and substrate cut by coating sticks on the coating chip surface. In other words, coating spalling occurs in “real” interface.

For process II, the morphology of fracture surface is very different from the above characteristic. In general, brittle fracture exhibits in both side of substrate and coating chip (Fig. 5). On the surface of coating chip, there is no element Fe detected by EDS. However, a small amount of element Fe is detected on the surface of substrate side. Because the penetration depth of X-ray is in the order of micrometers, a thin Cr layer is believed to be still adherent to substrate surface. In other words, coating spalling occurs in coating near the interface.

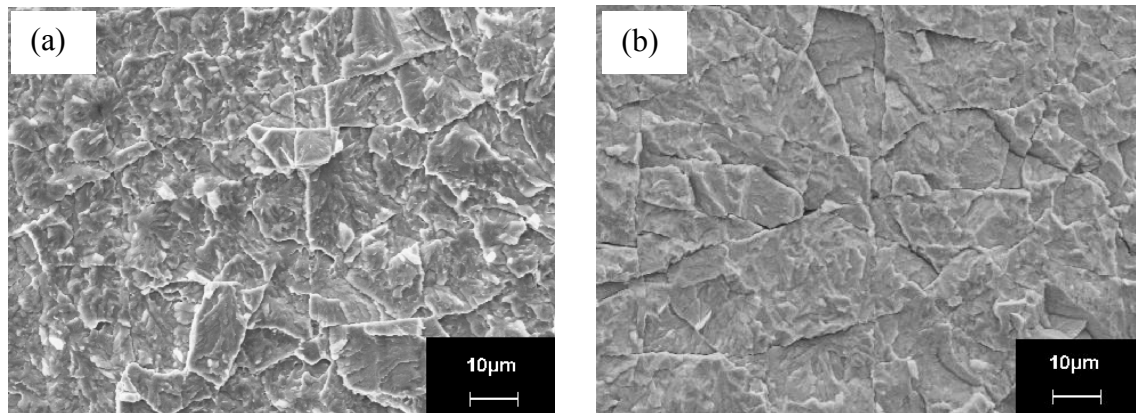


Fig.5. SEM picture of naked substrate side (a) and coating chip side (b) (process II)

Mechanics analysis

In addition to the above differences in interface fractography, the differences in interfacial cracking shape and indentation load vs. indentation depth curve are also observed (not shown here). In order to judge the usability of these evidences and help the ongoing quantification for interfacial adhesion, one should begin with interfacial stress analysis.

With FEM (Finite Element Method) software ABAQUS, stresses of the interface can be determined. Because of the symmetrical geometry, only a half of the entity is modeled. The indenter is assumed to be an analytical rigid body. For a brief analysis, substrate is modeled with linear element C3D4 with distortion controlled and elastic-plastic properties while coating is modeled without plastic property.

Coating and substrate are constrained by TIE method. Meshes in the vicinity of the contact area are refined (Fig.6). The Young's modules, Poisson's ratio, yield strength and strain-hardening modules of substrate are 210GPa, 0.21, 0.6GPa, and 50GPa, respectively. The Young's modules and Poisson's ratio of coating are 290GPa and 0.3, respectively. The indenter is loaded at a distance of 0.3mm near the interface on the cross-section.

The simulation result of interfacial stress distribution is shown in Fig.7 (the displacement of indentation being 0.12mm). Contour area with compressive stress, S33 of negative value, corresponds to the area where coating spallation occurs. Similarly, contour area where S33 vanishes corresponds to the vicinity of boundary curve as indicted in Fig.2. On the interfacial fracture area, the magnitude of shear stress S12 is remarkable. Therefore, type II and type III crack rather than type I crack occur. In this case, the evidence of slippage driven by the interfacial shear stress can be found easily on the interfacial fracture surface when spalling occurs in the “real” interface.

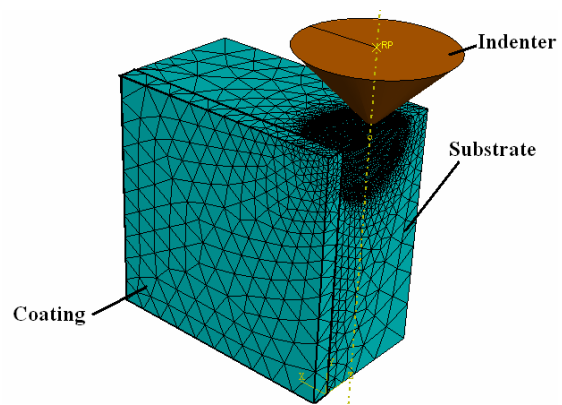


Fig.6. FEM modeling of indentation

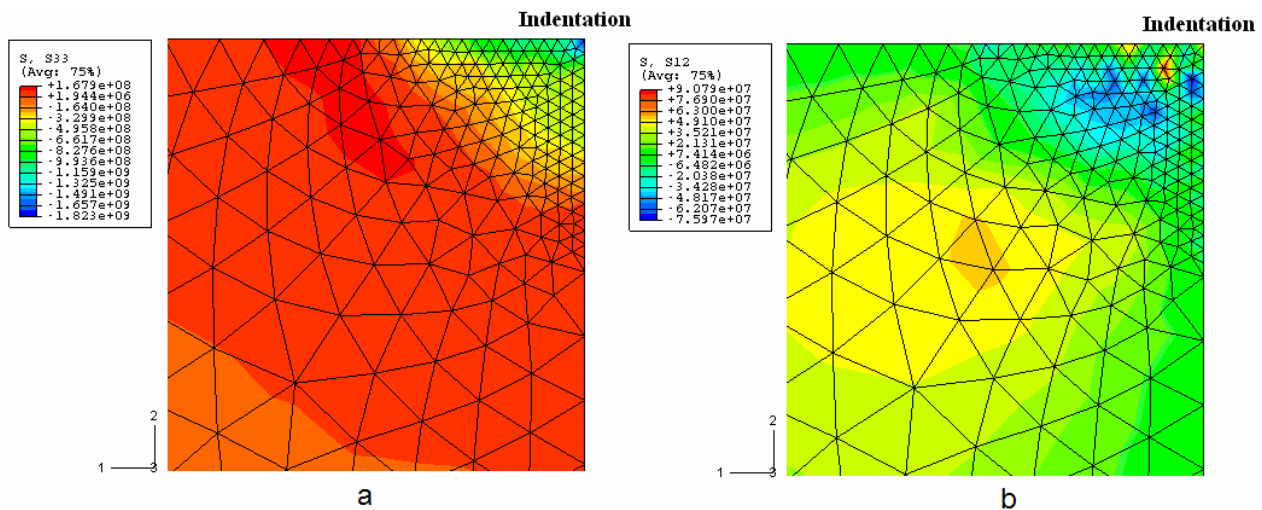


Fig.7. Contour of stresses S33 (a) and S12 (b) during indentation

Conclusions

Cross-sectional indentation may be extended to evaluate the interfacial adhesion in brittle coating/ductile substrate system with strong adhesion. Fracture mode of interfacial cracking is a mixed mode of sliding mode and tearing mode. Shear stress is the dominant factor for interfacial fracture.

Acknowledgements

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