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Uniaxial tensile and cross-sectional indentation tests to investigate the adhesion between the brittle Cr coating and the ductile steel substrate

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Abstract. Adhesion of a coating on its substrate is a crucial parameter determining performance and reliability of coating-substrate system. In this work, the uniaxial tensile and cross-sectional indentation tests are used to investigate the adhesion between the brittle Cr coating and the ductile steel substrate. The uniaxial tensile test results show the maximum shear flow stress level of the ductile steel substrate can only serve as a lower bound estimate on the ultimate shear strength of the interface. The cross-sectional indentation test results show the interfacial decohesion does not occur prior to the cracking of the brittle Cr coating.

Introduction

The brittle Cr coating is widely used in industries due to its fine properties against wear and erosion. Field observations as well as laboratory test results indicate that the predominant mode of failure in this material system is the spallation or loss of the Cr coating resulting from a decohesion or debonding in the coating parallel and close to the interface [1]. Thus, the adhesion of the coating on its substrate is considered to be a crucial parameter determining performance and reliability of coating/substrate system. Also, the evaluation of the adhesion is considered to be an important task. Although great efforts have been made to develop effective techniques for determination of the adhesion between coating and substrate, industries still need quantitative adhesion tests that should be simple, reliable and representative as possible [2]. As stated in [3], more than 200 methods were developed to quantify the adhesion of the coating on its substrate. Some typical test methods, such as the Pull-off test [4], bend test [5], scratch test [6], indentation test [7] have been used to evaluate the adhesion between the coating and the substrate. However, the methods mentioned above for adhesion measurement have limitations. For example, for the pull-off test, the adhesive used to glue a sample to the sample holder is required to have its adhesive strength higher than that of the interface. Generally, this test is suitable for the measurement of the adhesive strength that is weaker than 90MPa [8]. For the scratch test, a small diamond tip moves over the thin hard coating surface under a progressively increasing load. The initiation of interfacial decohesion is detected from acoustic emission signals or the load-displacement curve. The critical load corresponding to the initiation of interfacial decohesion is used to evaluate the adhesion. However, the test is influenced by a number of intrinsic and extrinsic factors that are not adhesion-related and the results of the test are usually regarded as only semi-quantitative [6]. It should be noticed that, for a brittle coating on a ductile metal substrate, the uniaxial tensile test has been adopted to evaluate the adhesion of this material system in some recent investigations [9]. As stated in [9], like any other type of mechanical tests, the interpretation of the uniaxial tensile test data to extract intrinsic interfacial mechanical properties of the coating attached to

a substrate is still non-trivial. A tensile test not only requires a relatively simple and inexpensive testing instrument, but also can produce in a well-controlled manner a large array of parallel cracks over the nominally homogenously deformed ductile substrate and allow in situ observation of cracking and decohesion of the coating via various microscopy tools [9]. One of the microindentation tests is the cross-sectional indentation test performed on the cross section of the substrate near the interface, and this method has proved to be successful in quantitatively determining the interfacial adhesive strengths between the brittle ceramic coating and the ductile metal substrate [7]. In this work, we perform the uniaxial tensile and cross-sectional indentation tests to investigate the interfacial adhesion properties between the brittle Cr coating and the normal medium carbon steel substrate.

The uniaxial tensile test

Uniaxial tensile experiment of normal medium carbon steel electroplated with a brittle Cr coating was performed in our study. The test materials were provided by No.47 factory (Northern Heavy Industry groups, China). The gage section of the dog-bone shaped specimen had dimensions of 100mm long, 10mm wide and 2.5mm thick while the thickness of the Cr coating was 0.1mm. After mechanical polishing, the tensile testing of the specimen was carried out quasi-statically on a universal test setup for coating-substrate system. A pair of tensile loads was applied at the two ends of the specimen. After each certain displacement increment, the test was interrupted to observe whether the interfacial decohesion occurred or not by using an optical microscope. Interestingly, even when the specimen, no interfacial decohesion and buckling of the coating could be observed, and only the quasi-periodic and through thickness cracks of the coating existed, as shown in Fig. 1. Five specimens were tested, and the results were similar. Optical micrograph of the cracking characteristic of the surface Cr coating is shown in Fig. 2, in which the arrows indicate the fracture profile. The case that the deflection of the cracks of the coating in Fig. 2 occurred can be attributed to the heterogeneous deformation and initial surface defects in the Cr coating.



Fig. 1. Cross-section optical micrograph of the fractured specimen.



Fig. 2. Optical micrograph of the Cr coating surface of the fractured specimen.

The cross-sectional indentation test

The material used for cross-sectional indentation test is the same as that used for the uniaxial tensile experiment. The specimens were cut using a diamond saw and final dimensions of a specimen containing Cr coating/steel substrate interface were $30 \times 30 \times 10$ mm. The cross-sectional surface of the specimens was polished. Microindentation was performed at the cross section of the substrate near the interface. A cone-shaped diamond indenter having a tip radius of 0.2mm and tip angle of 1200 was used. The schematic illustration of this experiment is shown in Fig. 3. A normal load was applied to

the indenter, and the rate of the crosshead displacement was 0.04mm/min. During these tests, the distance from the center of the indenter tip to the interface and the maximum indentation load could be changed. Due to the large size of the specimens, the distance between the indentations could be far enough and the interactions between these indentations could be neglected. Interestingly, a number of the indentation tests showed that cracking of the Cr coating occurred first, and the cases that interfacial decohesion occurred prior to the cracking of the coating could not be found. One typical micrograph of the indentation test results is shown in Fig. 4, in which the maximum indentation load was 200N and the distance from the center of the indenter tip to the interface was 0.3mm.



Fig. 3. Schematic illustration of the cross-sectional indentation test.

Fig. 4. A typical failure mode of the brittle Cr coating/ductile steel substrate material

Results and discussions

From the tensile test, we can obtain that even when the whole specimen fractured under the extreme tensile load, the interfacial decohesion and buckling of the coating is completely absent throughout the parallel length of the specimen. And the test results are different from those presented in [9] though the identical test method is taken to determine the adhesion of the brittle coating/ductile metal substrate system. In the research [9], the uniaxial tensile experiment was taken to estimate the interfacial shear strength of different brittle coating/ductile metal substrate materials using the crack density or spacing data obtained from this test or using a 2D finite element analysis incorporating a cohesive interface model of the cracked coating segments. During the test [9], the cracking of the brittle coating occurred first as the tensile strain increased, the crack density i.e. the number of cracks per unit axial distance and the average opening displacement of each crack of the coating were found to increase with increasing axial strain, and when the crack density of the coating started to reach the saturation state, i.e. the number of cracks per unit axial distance nearly became a constant even though the tensile strain is still applied, the decohesion and the buckling of the brittle coating can be obviously observed. But in our work, even when the whole specimen fractured under the extreme load, the interfacial decohesion and buckling of the coating cannot be found throughout the parallel length of the specimen. Thus, the cracking density or spacing data or the 2D finite element analysis incorporating a cohesive interface model cannot be used here to extract the ultimate interfacial shear strength. The ultimate elongation of the specimen under the extreme tensile load can reach nearly 16%. This indicates that the substrate has undergone large plastic deformation. Based on the theoretical analysis presented in [9], the maximum shear flow stress of the substrate can be exerted along the interface through the ductile substrate undergoing fully plastic deformation. Since interfacial decohesion is completely absent during these tests, we can obtain that the ultimate interfacial shear strength of this material system is high, and the maximum shear flow stress level of the ductile steel substrate can only serve as a lower bound estimate on the ultimate shear strength of the interface. The tensile normal strength of the normal medium carbon steel substrate is 540MPa.

According to the von Mises isotropic plasticity, the shear strength of the substrate is only 1/3 of the tensile normal strength of the substrate. So, we can calculate the shear strength of the substrate, and it is nearly 311.8MPa. It should be emphasized that this value only serves as a lower bound estimate on the ultimate shear strength of the interface between the Cr coating and the medium carbon steel substrate.

From the indentation test, we can obtain the interfacial decohesion does not occur prior to the cracking of the Cr coating. As mentioned above, although the identical test method is taken to determine the adhesion of the brittle coating/ductile metal substrate system, the test results are different from those presented in [7]. In the research [7], the cross-sectional indentation test was taken to determine the interfacial adhesive strengths between different brittle ceramic coating/ductile metal substrate materials. As stated in [7], when indentation was carried out near an interface, the constraint around the indenter would be no longer symmetrical due to the difference in mechanical properties between the coating and the substrate, and when the indentation load was sufficiently large, interfacial decohesion would occur. An important test result from the literatures [7] is that the cracking of the coating did not occur, and only the interfacial decohesion occurred. As for our test, though the mechanical properties between the brittle Cr coating and the ductile steel substrate exist great differences, the interfacial decohesion cannot be observed prior to the cracking of the Cr coating. This case can be attributed to one important factor that the interfacial adhesion of the Cr coating on the ductile steel substrate is very strong, and this situation will lead to the first cracking of the Cr coating during the indentation. This case is also consistent with the situation that the tougher the interface, the more likely the coating breaks [10]. The first cracking of the coating will make the quantitative determination of the interfacial adhesion become difficult in that the three-dimensional problem of the cracking of the coating will dynamically change the stress states of the interface, the coating and the substrate during the indentation. Furthermore, even if the interfacial decohesion does occur after the cracking of the coating under an increasing indentation load, the first difficulty is how to determine the critical indentation load that corresponds to the interfacial decohesion. So, if we want to determine the adhesion between the Cr coating and the medium carbon steel substrate using this test method, we still need further investigations.

Conclusions

Two different test methods were adopted to investigate the interfacial adhesion between the brittle Cr coating and the ductile steel substrate. The test results presented in this paper are different from those appeared in the literature though the identical test methods and the brittle coating/ductile metal substrate system are taken. The uniaxial tensile test results presented in this work show the maximum shear flow stress level of the ductile steel substrate can only serve as a lower bound estimate on the ultimate shear strength of the interface. The cross-sectional indentation test results presented in this work show the interfacial decohesion does not occur prior to the cracking of the Cr coating, and this case can be attributed to one important factor that the interfacial adhesion of the Cr coating on the ductile steel substrate is very strong.

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References

- [1] S. Sopok, C. Rickard and S. Dunn: Wear Vol. 258 (2005), p. 659.
- [2] G. Marot, J. Lesage, Ph. Démarécaux, M. Hadad, St. Siegmann and M. H. Staia: Surf. Coat. Technol. Vol. 201 (2006), p. 2080.
- [3] A. A. Volinsky, N. R. Moody and W. W. Gerberich: Acta Mater. Vol. 50 (2002), p. 441.
- [4] M. P. K. Turunen, P. Marjamaki, M. Paajanen, J. Lahtinen and J. K. Kivilahti, Microelectron. Reliab. Vol. 44(2004), p 993.
- [5] H. Zhang and D. Y. Li: Surf. Coat. Technol. Vol. 155 (2002), p.190.
- [6] S. J. Bull and E. G. Berasetegui: Tribol. Int. Vol. 39(2006), p. 99.
- [7] H. Zhang, Q. Chen and D. Y. Li: Acta Mater. Vol. 52(2004), p. 2037.
- [8] H. F. Qi, A. Fernandes, E. Pereira and J. Grácio: Diam. Relat. Mater. Vol. 8(1999), p. 1549.
- [9] C. J. Xie and T. Wei: Acta Mater. Vol. 53(2005), p. 477.
- [10] V. Gupta, J. Yuan and D. Martinez: J. Am. Ceram. Soc. Vol. 76(1993), p. 305.