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Mechanical characteristics of Fe-based coating obtained by nanoindentation

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ABSTRACT

In this study, clad layers of iron-based alloy with a nature of self-fluxing were melted on low carbon steel by plasma cladding process. Nanoindentation with atomic force microscopy (AFM) has been used to investigate the mechanical properties of the coating. Hardness and elastic modulus at ultra-low loads were first determined using the method proposed by Giannakopoulos and Suresh (G&S method). The true contact area and mechanical properties were then determined using atomic force microscopy (AFM) combined with the Oliver and Pharr method (new proposed method) as the correction group. The mechanical properties calculated by the two methods showed the same distribution while had deviation in specific values. The effect of surface roughness to the calculated mechanical properties was investigated. Both hardness and elastic modulus were found to exhibit certain surface roughness dependence. When root mean square (RMS) roughness ranged from 2.2 nm to 4.4 nm, hardness calculated by both the methods increased obviously and reached maximums around 4.1 nm. Elastic modulus calculated by G&S method at different RMS showed the same distribution with that of hardness, while reduced elastic modulus obtained by AFM was insensitive to the range of RMS.

1. Introduction

Instrumented indentation methods, which provide a continuous record of the variation of indentation load, P, as a function of the depth of penetration, h, into the indented specimen, have widely been used in the characterization of mechanical behavior of materials at small scales. A great many researchers have made efforts in this field [1–8]. The most general and widely used model in this area is the one proposed by Oliver and Pharr method [1,2]. However, one significant problem with this model is that it cannot account for material that pile-up. When pile-up occurs, the contact area obtained by this method will be overestimated sometimes by as much as 50% which directly results in errors in the calculation of mechanical properties. Nowadays much work has been done to develop methods that can be used to correct for pile-up at the same time avoid using the image of the contact impression [3,4]. A good example of work in this area is that of Giannakopoulos and Suresh [3], who outline unique correlations between penetration depth h and true contact area A for sharp indenters which based on the work of indentation that can be measured from the areas under indentation loading and unloading curves. In this study, this method (G&S method) was used to investigate the mechanical properties of the iron-based coating and the results were compared with the values obtained based on the atomic force microscopy (AFM) images.

Specimens used in nanoindentation tests are required to be polished while different preparation processes would result in differences in surface roughness. Though the surface roughness produced by a sample preparation would be as small as possible, the minor changes of it may lead to discrepancy in the results [7,9]. In this study, the influences of surface roughness to mechanical properties obtained by instrumented indentation technique were also investigated.

2. Experimental details

2.1. Materials

Low carbon steel containing 0.18 wt.% carbon was taken as the substrate. No previous heat treatments were required. The compositions of Fe-based alloy powders are listed in Table 1. Prior to plasma cladding, the substrate was degreased in acetone solution.

2.2. Preparation

The plasma cladding equipment produced by the Academy of Armored Forces Engineering (China) was adopted as the experimental equipment. Ar was used as both the protective gas and the plasma gas. The parameters of plasma cladding are shown in Table 2.

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Table 1
Chemical composition of Fe-based alloy.

Element	C	Cr	Ni	B	Si	Fe
Mass fraction %	0.1	15	10	1	1	Bal.

2.3. Nanoindentation

Prior to indentation tests, the surface of the 15 specimens was grinded with sandpaper and polished with diamond paste for different times (arranging from 10 min to 25 min). A Berkovich three-sided pyramidal indenter with a tip radius <30 nm was used throughout this study. The applied indentation loads and the penetrating depths were recorded simultaneously during an indentation loading–unloading cycle by nanoindentation equipment (Hysitron TriboIndenter® system). The system is capable of "AFM", or atomic force microscopy, style imaging which allows imaging with high resolution. For one sample load was applied by a linear ramp up to certain maximum loads (3 mN, 5 mN, 6 mN, 7 mN, 8 mN, 9 mN; for the rest of samples middle load 5 mN was chosen for its good contact impression as the only maximum load to investigate the influence of surface hardness on the calculation of mechanical properties) within 5 s, holding the maximum load for another 5 s and with a linear unloading segment lasting 5 s. For each load, 15 indents were made on the investigated surface and the results presented were the average of these fifteen indentations. The spacing between adjacent indentations was 10 μm (which is more than 5 times of the indentation diameter and is far enough to avoid the effect of adjacent indentations). The indents on the polished surface were observed by AFM. The root mean square roughness of the surface and contact impression was given and investigated by TriboView software. Variation of the loading and unloading rates was 1000 μN/s. Indentations were made approximately at the centre of the sample to avoid and testing near the edges. All experiments were performed at room temperature. The basic parameters used in the following calculation are from the standard method proposed by Oliver and Pharr [1,2].

3. Results and discussion

3.1. Analysis of load–depth curves

Average indentation load–depth curves at different load levels of 3, 5, 6, 7, 8 and 9 mN are shown in Fig. 1. The load is defined as the total force on the indenter and the depth is measured from the indenter's starting position. It is obvious that the residual indentation depth of the specimens increased evenly with the increasing indentation load. This clearly reveals that the coating is homogeneous under ultra-low loads.

According to Oliver and Pharr's theory, the ratio h_r/h_{max} can be used to identify the expected indentation behavior of a given material [1]. When the ratio $h_r/h_{max} > 0.7$, pile-up can be obviously observed and then the Oliver–Pharr method is no longer applicable. In fact the values of h_r/h_{max} extracted form the unloading curve in Fig. 1 are all greater than 0.75. Combined with the 3D map obtained by AFM the coating can be concluded belonging to material of pile-up. In this case, the contact depth h_c is larger than the maximum depth h_{max}, which cannot be explained by Oliver and Pharr method and will result in large errors in the calculation of real contact area, further affect the calculation of mechanical characteristics. New methods should be taken to investigate the mechanical properties of the coating.

Table 2
Parameters of plasma cladding.

Current	120 (A)
Voltage	22 (V)
Scanning velocity	1.8 (mm/s)
Shielding gas	Ar
Feeding powder flow	2.2 (g/min)
Plasma gas flow	5.0 (L/min)

3.2. Hardness and elastic modulus

Giannakopoulos and Suresh established a general theoretical framework for instrumented sharp indentation and invented a new methodology which avoids applying contact depth to get the true contact area to determine the mechanical properties of materials. The hardness and elastic modulus then can be calculated by the following equations (G&S method) [3,4], where W_t is the total work which equals to the area under the loading portion of the load–depth curve, done by the indenter in deforming the material. The total work W_t is then decomposed into elastic and plastic parts [3]: $W_t = W_e + W_p$. The hardness and elastic modulus obtained by this method are shown in Figs. 2 and 3, respectively.

Though developing a method that can be used to correct for pile-up in a manner that does not involve imaging the contact impression has been a great target of instrumented indentation research, calculating the true contact area through the contact impression imaged by optical instruments is a significant means of calibration. In this study, the contact depth h_c is redefined as the summation of h_{max} and the average height of pile-up $h_{pile\text{-}up}$, according to the image of contact impression taken by AFM. With the redefined parameter, h_c, the true contact area can be finally determined and combined with the Oliver and Pharr method the hardness and elastic modulus then can be calculated (new proposed method). The values obtained by this method are also shown in Figs. 2 and 3 which are compared with the results calculated by G&S method.

Fig. 2 shows the influence of nanoindentation load on the measured hardness of the specimens by both methods. Each data point

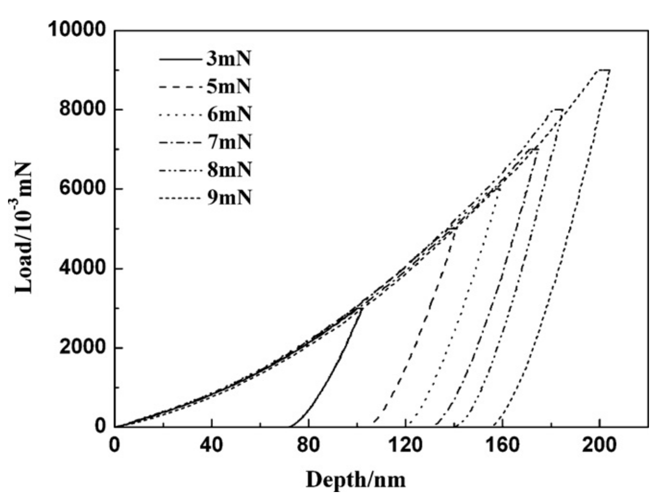

Fig. 1. Load–depth curves of iron-based coating obtained by nanoindentation at different loads.