Article ID: 1003 - 6326(2004)S2 - 0282 - 04

Metallographic sample prepared by ion beam etching①

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Abstract: Ion beam etching technique was used to reveal the metallograhpic microstructure and interface morphology of electroplating chromium coating, in particular, whose substrate surface layer was treated in advance by laser quenching. Chemical etchings were also conducted for comparison. The reveal microstructures were observed and analyzed by scanning electron microscopy. The results show that ion beam etching can reveal well the whole microstructures of composite coating-substrate materials.

Key words: ion beam etching; metallography; microstructure; electroplating; chromium
CLC number: TG115.21+1.3; TQ153.1+1 **Document code:** A

1 INTRODUCTION

It is very important to really reveal metallographic microstructure of materials in order to establish the relationship of the processing and properties of materials. In general, chemical and electro-chemical etching method was adopted for this purpose[1,2]. However, if the etched material is heterogeneous, for example, coating-substrate materials, dissimilar weld bead, and composite, where the great difference in chemical properties depending on those individual phases occurs, normal chemical and electro-chemical etching are often limited[3].

In contrast to chemical etching, ion beam etching is a universal etching method because it does not depend upon chemical affinity but only on a mechanical effect. Because of this it can be used to develop the structures of complex materials in which the component materials exhibit a great difference in their respective chemical potentials[4]. But it has currently only a few researches and applications in this respect. Graf[5] investigated ion beam etching to prepare the samples of composite layered materials such as thermal spraying layers on the various substrates. Some other materials such as ceramic and metallic materials were also involved. Some international corporations, which product ion beam apparatus, also expressed that excellent metallographic microstructure of aluminum, titanium, aluminum bronze and superconductor could be obtained by ion beam etching.

Electroplating chromium coating has extensively applications due to high hardness, excellent wear and corrosion resistance. However, in the situation of high temperature, high pressure and combustion medium, the chromium coating especially readily spalls along the interface between the coating and the substrate[6]. Thus, a novel processing method that surface layer of substrate was quenched by high power laser scanning prior to electroplating chromium was presented. The results showed that the spallation resistance of chromium coating could be significantly improved and the service lifetime could be increased.

In order to clarify the mechanism that laser quenching can improve the bonding, it is necessary to reveal the microstructures of not only the chromium coating, but also the laser quenching zone, in particular the interface between the coating and substrate. But, because of the greatly different chemical properties, chemical or electrical-chemical etchings either completely failed or have inadequate results.

In this investigation, ion beam etching technique is used to reveal the microstructure of laser plus electroplating composite chromium coating. Chemical etchings are also conducted for comparison. The revealed metallographic microstructures by two different methods are observed, compared and analyzed by scanning electron microscopy.

2 EXPERIMENTAL

The substrate material was a 30Cr2NiMo steel plate. A 2 kW YAG laser was used to quench the surface layer of the steel plate. Afterwards, by standard electroplating processing, a soft chromi-

① **Foundation item:** Project (40404120204) supported by Prior Research Project of Science and Technology Commission of Defensive Industry of China.
Received date: 2004 - 05 - 10; **Accepted date:** 2004 - 07 - 15
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um layer was firstly electro-deposited on the substrate, and then a hard chromium layer on the soft chromium layer. The processing scheme is shown in Fig. 1.

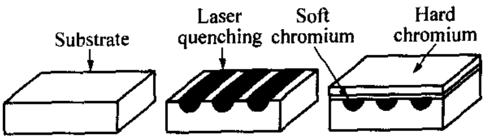

Fig. 1 Schematic diagram of laser plus electroplating composite processing

The cross sectional samples were cut off by an electro-sparking machine for the preparation of metallographic samples. Afterwards, they were grinded and polished mechanically. An ion sputtering apparatus, KYKY-FD1500, was used for ion beam etching experiment. Argon was used as ion gas. The ion beam etching parameters are given in Table 1. In addition, chemical etching experiments were conducted for comparison. Two solutions, alcohol with 4% (volume fraction) nitric acids, and nitric acid plus hydrochloric acid in ratio of 3 to 1 by volume, were used as reagents.

Table 1 Ion beam etching parameters

Current/mA	Voltage/V	Time/min	Incidence angle/(°)
60	1 000	30	45

3 RESULTS AND DISCUSSION

Fig. 2 shows the metallographic microstructures etched by alcohols with 4% nitric acids. It can be seen that the chromium coating can not be completely etched due to the excellent corrosion resistance. The refine structure of the interface can not be revealed. The substrate can be etched very well so that the refined lath martensite and carbide particles can be clearly identified. The longitudinal cracks are inherent in electroplating chromium coating.

Fig. 3 shows the metallographic microstructures etched by nitric acid and hydrochloride acid mixed solution. In contrast to the above case, here the substrate can hardly be etched, and the chromium coating is etched so intensively that serious surface detachment occurs. In fact, they should not be thought of as metallographic microstructure, but corrosion morphology. However, because corrosion maybe occur along the crystal grain boundaries, to some extent, the morphology reflects the microstructure scales. In addition, the difference in the microstructure of the soft chromium and hard chromium can be roughly distinguished. The above results indicate that the normal chemical etching is unsuitable to the complex coating-substrate materials. They usually result in selective etching, or even disable.

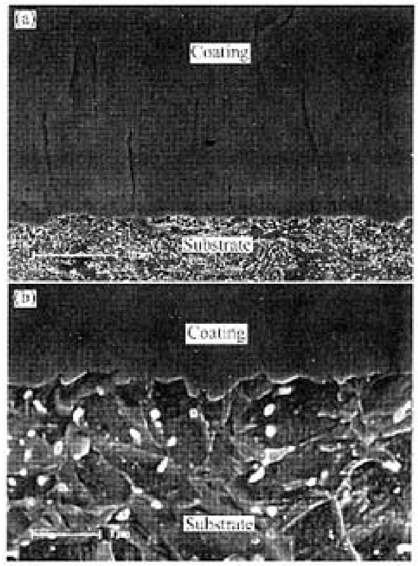

Fig. 2 Metallographic microstructures etched by alcohol plus acid(SEM)
(a)—Cracking coating not etched;
(b)—Substrate consisting of lath martensite and refined carbide particles

Fig. 4 shows the metallographic microstructures etched by ion beam. Fig. 4(a) shows the microstructure near to the interface of laser quenching zone. The soft chromium microstructure consisting of refine crystal grains with definite orientation can be identified. Fig. 4(b) shows an enlarged image of the interface. It can be found that the soft chromium is characterized by epitaxial growth from the substrate upwards. Thus solid interface bonding is realized by laser quenching. This may be a critical factor that results in the increased spallation resistance.

On the contrary, in the situation of non-laser quenching on the substrate, the microstructure of the soft chromium seems to be finer, but the evident transient layer with a thickness of 2 - 3 μm exists at the interface as shown in Fig. 4(c). As a result, two new interfaces form respectively between the chromium coating and transient layer, and between the substrate and transient layer as shown in Fig. 4(d). The crystal structure and formed mechanism of the transient layer need be further researched in the future. They may have a definitely influence on the bonding characterization of chromium coating. Fig. 4(e) shows the microstructure of the hard chromium. They consist of a lot of

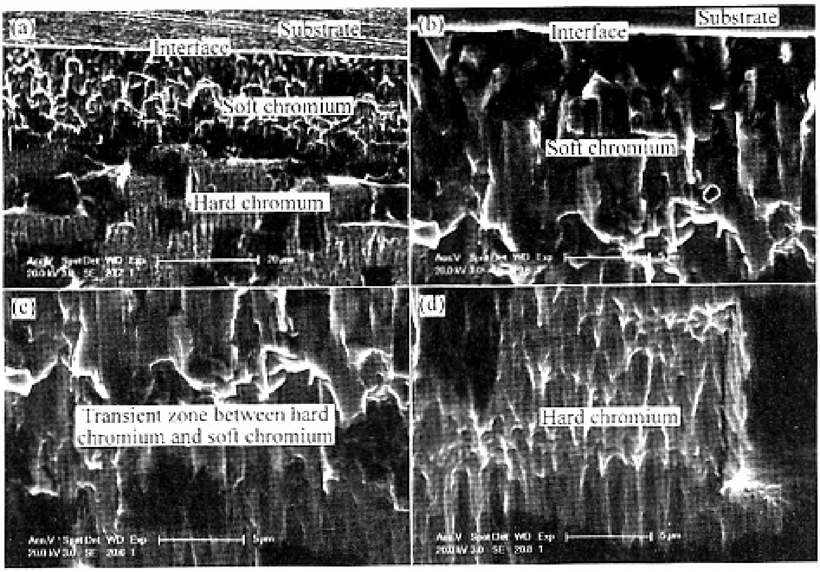

Fig. 3 Metallographic microstructures etched by nitric and hydrochloric acid solution(SEM)
(a)—Whole morphology; (b)—Morphology of interface and soft chromium;
(c)—Transient zone between soft chromium and hard chromium; (d)—Hard chromium morphology

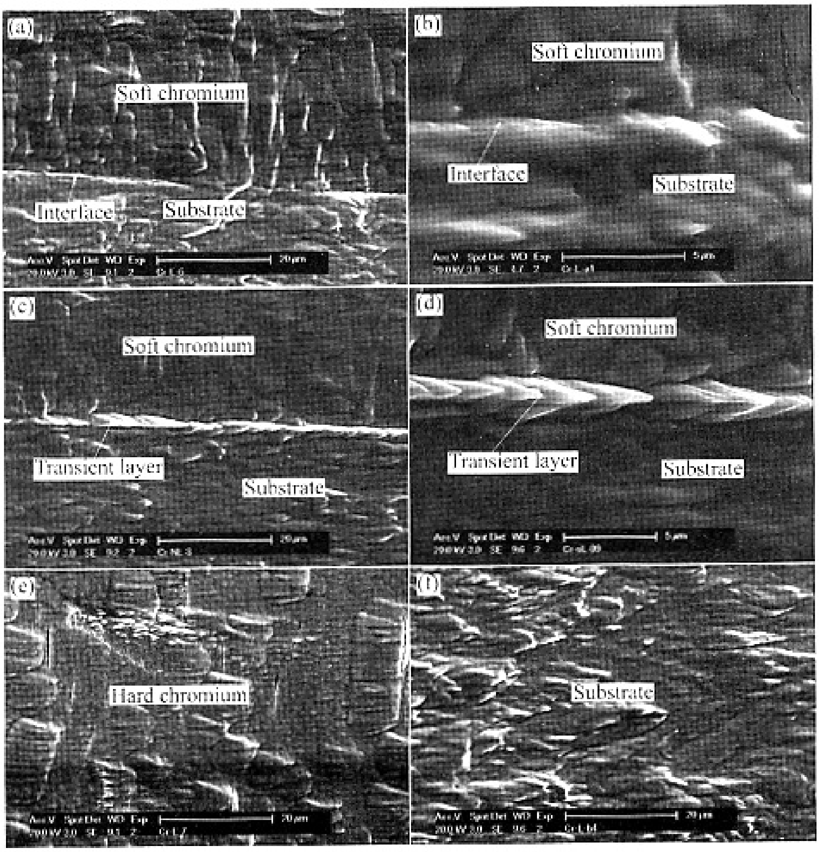

Fig. 4 Metallographic microstructures etched by ion beam (SEM)
(a)—Laser processing microstructure; (b)—Epitaxial growth after laser processing;
(c)—Non-laser processing microstructure; (d)—Transient layer without laser processing;
(e)—Hard chromium microstructure; (f)—Substrate microstructure of laser processing

clusters with significant orientation difference. The more refined microstructure has also been revealed. It is very interesting that micro-cracks basically initiate along the boundaries of the clusters. This indicates that the interfaces between the clusters are weaker due to inclusions and additives segregation. Fig. 4(f) shows the microstructure of the substrate treated by laser quenching. It is typical martensite microstructure.

Ion beam etching is a physic process, the rates of material removal depend on the compositions, structures as well as crystallographic orientations materials. When heterogeneous and polycrystalline surfaces are attacked by ion beam and the topography of the surface, should be altered, which results in the correct metallographic microstructure revealed by the suitable instrumental settings[7]. The experimental results on laser plus electroplating composite chromium coating show that ion beam etching is very effective to reveal the microstructure of complex material system, involving every phases with the great chemical properties difference and in particular the interface. More refined structures could be revealed than other etching methods.

4 CONCLUSIONS

1) Ion beam etching has significant advantages over chemical etching in revealing the microstructure of laser plus electroplating composite chromium coating.

2) Ion beam etching shows that the epitaxial growth of the coating can be realized by laser quenching. This will result in the excellent bonding and consequently increased spallation resistance.

3) The soft chromium consists of refined equiaxed crystal grains that grow vertically upwards. The hard chromium consists of a lot of clusters with different orientations. The individual cluster consists of extremely refined crystals. The inherent cracks are found to initiate and propagate along the interface of the different orientation clusters.

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(Edited by LONG Huai-zhong)