Research on Organizational Structure of TiN-based Multiple composite Coating

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Abstract. In this paper, the TiN-based of CrTiAlN multiple composite coating were deposited on die steel substrates .The phase structure, chemical composition and morphology of the coating were tested by XRD, SEM and EDS respectively. The adhesive strength between film and substrate and microhardness about the coating were tested by Rockwell hardometer and Vickers hardometer. The results showed that the coating surface morphology of CrTiAlN had become tightly clustered granular particles, and the section morphology had a structure with columnar crystal. The main crystalline phases of CrTiAlN coating were consisting of TiN, CrN, Cr_2N and so on. Rockwell indentation test showed that it had good bonding force between the CrTiAlN coating and the die steel substrates.

Introduction

In recent decades, with the modern science and technology developing, the researches of surface coating technology and materials have made rapid developments. Surface coating technology is a inter-technology which involves materials, chemistry, physics and other disciplines, and it is aiso an important means of materials surface modification. It has been widely used in the semiconductor, instrumentation, aerospace, nuclear energy , mechanical and other fields[1-3].The tool or die steel can form refractory metal hard coatings (such as TiN, CrN, etc.) or non-metallic compound soft coating (such as MoS2 etc.) which have wear resistance, high temperature resistance and other special properties by using PVD, CVD or other deposition method, so these technologies have aroused public concern[4-6].

In this paper, based on the TiN hard coatings, we use Al target, Cr target to prepare CrTiAlN coating, and to analyze the microstructure of the coating.

Experimental Materials and Methods

Use die Steels (hardness about HRC 60) as base materials.In order to get good adhesion between the coating and the substrate, use different grades of waterproof abrasive paper to polish samples' surface progressively, and then use diamond polishing slurry to polish the polished samples on the polishing machine until the surface roughness Ra≤0.5μm. Then washed sequentially with acetone, absolute ethanol to clean 20 minutes on ultrasonic cleaning machine to remove surface oil and other impurities, and put the cleaned samples in a vacuum oven to dry them completely.

Using ΣIGMA type Field Emission Scanning Electron Microscope (FESEM) to observe and

analyze the surface coating, fracture appearance and the wear morphology which had been tested by friction and wear tests. Samples preparation method was to snap samples directly in the liquid nitrogen cooling condition, and then put them into electron microscopy to observe. Using ΣIGMA type FESEM attachment, Bruker Nano XFlash Detector 5010-type spectrometer, to test coating surface elemental composition and the coating section changes of elemental composition.

Using Rockwell hardometer to test the hardness of the coating, and the hold time is 10s. In order to reduce error, each sample should be measured 5 times, and then took the average value.

Using SEM to measure the thickness of the coating. First, using wire-electrode cutting to cut out the fracture traces (not cut into the coating) of the samples in the uncoated side, and then placed them into liquid nitrogen to cool a few minutes.When it done, took the samples out and snap out immediately. And then put them into the electron microscope to scan samples fracture surface, using electron microscopy its own scale to come out the coating thickness.

Experimental results and analysis

Morphology and chemical composition of the coating.

CrTiAlN coating surface morphology is shown in Figure 1 (a). The coating surface had become tightly clustered granular particles, which size was uniform. and the average size was about 150nm. Figure 1 (b) is fracture surface of CrTiAlN coating. It can be seen that the coating section structure was obvious columnar crystal, and columnar crystal was dense,and there were no obvious pores and defects on it.In addition, to the coating section , it can also be observed that near the substrate region showed a fine crystalline structure, which ensured good adhesion between the coating and the substrate.

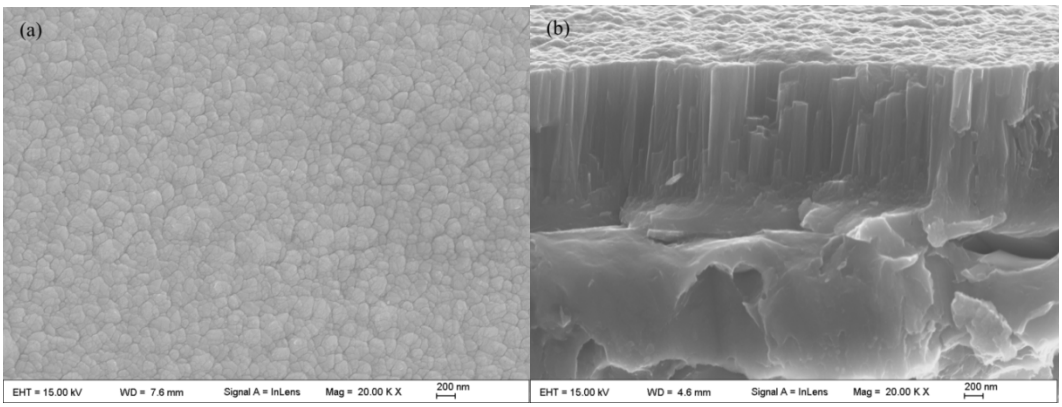

Fig.1. Microstructure of CrTiAlN coating

Figure 2 is the test results about the chemical composition of CrTiAlN coating. It can be seen that coating its Cr content is 44.80 at%, Al is 5.26 at%, Ti is 6.45 at%,and N is 44.80 at%.

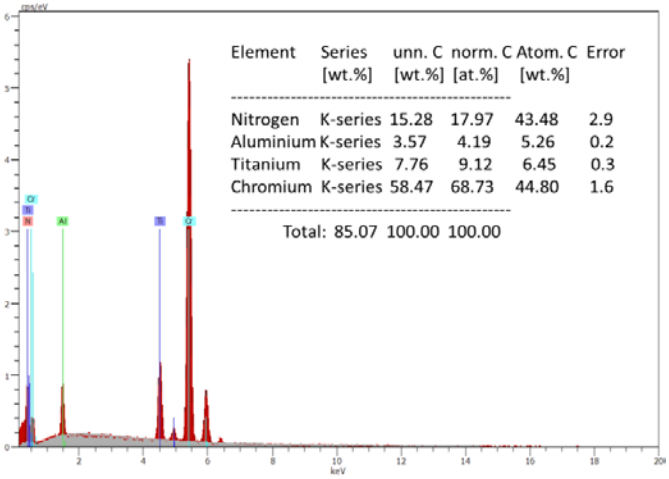

Fig.2. The chemical composition of CrTiAlN coating

Phase structure of the coating.

The phase structure of CrTiAlN multiple composite coatings were analyzed by XRD, and the results are shown in Figure 3. It can been see that the phase structure of the coating CrTiAlN was similar to the crystal structure of NaCl B1, and the main crystal phase of the coating contains CrN, Cr and Cr2N phases , also it may contain small amounts of AlN and TiN phase. It can be known CrN diffraction planes are (111), (200) and (220).

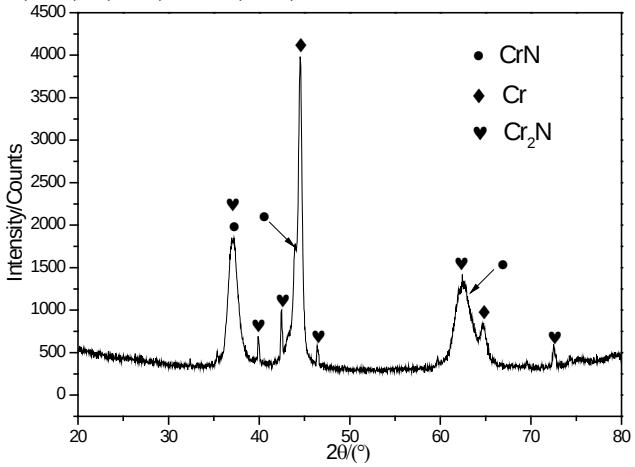

Fig.3. XRD diffraction of CrTiAlN coating

Bonding condition between the coating and the substrate.

Using Rockwell hardness indentation method and SEM to analyze the bonding condition between the coating and the substrate, the results had been shown in Figure 4. The figure showed that the substrate of Rockwell indentation surface became the deformation zone, the coating also had be deformed. But the phenomenon of peeling or warped did not occur, indicating that the coating has good adhesive with substrate. Because the coating has high hardness, so in order to adapt to the deformation of the substrate under the coating, few circumferential cracks occurred on the surface of the deformation zone, but the cracks didn't throughout the indentation.

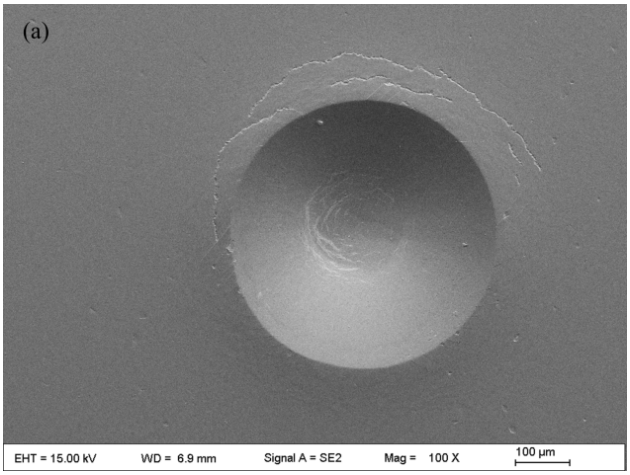

Fig.4. Rockwell hardness indentation of CrTiAlN coating surface

Conclusions

(1) The coating surface had become tightly clustered granular particles, which size was uniform, and the average size was about 150nm. The coating section structure was obvious columnar crystal, and columnar crystal was dense. There were no obvious pores and defects on it, which ensured good adhesion between the coating and the substrate.

(2)The phase structure of CrTiAlN coating was similar to crystal structure of NaCl B1, and the main phases of coating were consists of TiN, CrN , Cr2N and so on.

Acknowledgement

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