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RESEARCH ON MODIFIED LAYERS OF MATERIAL SURFACE FOR CR12MOV COLD DIE

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ABSTRACT

Multi-ion beams (MIB) is one of several material surface modification techniques based on ion implantation and deposition. The service life of Cr12MoV cold die treated by MIB in site has been increased 2-4 times. The treated tool or die has obviously higher hardness, lower friction coefficient and higher wear resistance. The modified layer is composed of (Ti_2N +TiN), (αFe+TiN) and (αFe(N)+Cr_7C_3) from surface to inner layer. The adhesion between the film and substrate is strong, and the grain of the layer is of the size of several nm.

Keywords*:* Ion beam; Material surface modification; Modified layer

1. INTRODUCTION

The service life of a tool or a die directly influences the precision of spare parts and the efficiency of production. Impact resistance is also considered to be an important characteristic of tools. When treated by CVD, their shape changes dramatically due to high temperatures. Though superhard coat can be obtained by PVD, tools can not resist impact because there is not any transitional layer between the film and substrate, and the coat is easy to come off. Ion implantation can solve the above problems. But population of the method is limited by its higher process cost, high price of an ion implanter and thin coat layer. We propose technology of multi-ion beams (MIB) to solve the problems better. The service life of tool or die treated by MIB in site has increased by a factor of 2~4 times.

2. MATERIALS AND EXPERIMENTAL PROCEDURE

Samples of Cr12MoV code die steel, with chemical composition (wt%): 1.55C, 11.5Cr, 0.5Mo, 0.225V, 0.40Si, 0.40Mn, had been polished and cleaned after conventional heat treatment.
The nitrogen ion current densities of the implanting source were 10.14μA/cm, 19.3μA/cm, 24.2μA/cm, 28.2μA/cm respectively. Ion beam sputtering energy was 4keV. Ti atom deposition rate: 200□/min.

3. RESULTS AND DISCUSSION

3.1 Test of the properties of the deposition-implantation layer

3.1.1 HK Hardness

We used DMH-2LS microhardness tester under the condition of 2g/15s, the hardness of the substrate is HK801. Results are shown in table 1. The highest hardness value, which was three times as hard as the substrate, was achieved when the current density of nitrogen ion was 19.3μA/cm.

3.1.2 Wear Test

The wear testing was carried out on Mus-ion-1 wear tester, using 700 emery paper, under 445gf load and 2000 times wear. The result obtained is shown in Fig.1, from which we can see the wear propertyes of the samples processed by MIB technique. And the best result has been gotten when the nitrogen ion current density is 19.3μA/cm. The result just corresponds to that of the hardness.

3.1.3 Measurement of friction coefficient

A TIPE-HEDON-10 static friction coefficient tester has been used for the measurement stainless steel as the contrasted sample, the testing results are shown in table 2. From table 2 it can be seen that the friction coefficient of most samples processed by MIB decreased as compared with their original states. When the N ion current density is 19.3μA/cm, the friction coefficient was the smallest.

3.1.4 The service life of cold die steel in site

Materials of dies tested were LD steel, W, Cr, V and $Cr_{12}MoV$ steel. Testing results were listed in table 3. The service life of die treated by MIB was increased by 2~4 times.

3.2 Microstructure of sample treated by mib

3.2.1 Phase characterization of the modification layer

Using PW1710 X-ray diffraction apparatus, we have obtained the results of the surface phase construction of the modification layer under different conditions shown in table 4 from which we can see that with N current density increasing, the diffraction peaks of Ti_2N and Ti phase in the film decreased gradually and even died way. Moreover, the diffraction intensity of TiN phase also decreased. This is resulted from the implantation of high ion current density under which the opposite sputtering is formed and makes the film thin, when the nitrogen ion current density was 19.3μA/cm, there was little Ti phase, but more TiN and Ti_2N phase in the film were formed. As a result, the layer possesses high microhardness and high wear resistance.

3.2.2 Formation of nm size crystal particle

The superhard coat formed by MIB technique was composed of the compact mixture phases of TiN and Ti_2N. The crystal particles were about the size of 200□. Fig.2 shows the TEM bright field image of the coating treated by MIB.

3.2.3 The distribution of the elements in the modification layer

We studied the distribution of the main elements N, Ti and Fe in the depth direction in the modified layer by DHI-600 multiple function electronic energy spectrum apparatus. Fig3 is the energy spectrum image of all elements on the sample surface under such condition that the Nitrogen ion current density was 19.3μm/cm. Testing condition was that the primary energy of electronic gun was 3kV (E_p=3kV), the current density of the electronic gun was 3μA (I_p=3μA), the testing energy of the analysis apparatus (V_{mod}) was 6eV and the Voltage of electron multiplier was (V_{mui}) 1200V. We can see from Fig.3 that higher C peak was gotten on the sample surface. In addition, O peak (O_{510ev}), Ti and N peak also existed at the surface.

3.2.4 AES analysis of the transverse section

In the test of AES, We have used Ar ion beam to Carve-Corrosion at a rate of 66□/min related to Ta_2O_5, simultaneously traced by Anger electrons. A distribution in the depth direction is shown in Fig.4.

(1)C.O element peaks at the surface of the film are of higher and become lower along the layer depth with the increase of carving corrosion time. And all are less than 3%.The Carbon at the surface comes mainly from air and diffusion pump oil, the remainder carbon decompose under ion beam radiation to become activation state from steady state. They were easy to act with other elements, and easy to diffuse. Oxygen comes mainly from the air that the samples absorbed when they were placed in air.

(2)The distribution of N and Ti element in the depth direction is still stable after carve-Corrosion 3300□. The atomic percentage of N element in the film is 69.18% and of Ti element is 25.8%.

Fig.5 is the surface AES transverse section analysis of modified layer after 2000 times of wear. From Fig.5, we can see: When the time of Ar carve-corrosion is 60 minutes, the caving depth reaches 6501□. When the time of Ar carve-corrosion is 32 minutes, the depth is 2497□ and during this time the content ratio of N and Ti element at the modified layer keep steadily. Over 32 minutes of carving-corrosion, the atomic contents of N and Ti element decrease obviously, among which of N element have a more rapid decrease. When the time of Ar carve-corrosion is 50 minutes, the depth become 5071□, the relative atomic percentage content of N□Ti□O and C element is 24.7%□21.4%□43.1%□4%□1.7% respectively. Here Fe atom increased dramatically. When the time of carving-Corrosion was 60 minute, the depth was 6510□, the relative atomic percentage content of N□Ti□Fi and O element were 13.7%□9%□74%□3.2% respectively, and that

of C element was nearly zero and Fe becomes the main element existed.
from the above analysis, We can see, the main element distribution rule for the modified layer treated by mixing ion beams (N□Ti) is (Ti+N□→□Ti+Fe+N□→(Fe+N□.

3.3 Analysis of tem for the modified layer

We use JEM-100CX □ transmission electronic microscope to analyse the modification layer treated by MIB with 13.9□/cm of the N ion beam density.

3.3.1 The preparation of transverse section sample

As modified layer possesses the properties of different kinds of layers, if we made level film, we could only see the organization and image of its surface, and couldn't seen the characteristics of the layer, So it is impossible to reflect the real image of the modified layers. We prepare the transverse sample for TEM.

3.3.2 Analysis of tem of transverse section

Fig.7 is the bright field image and electron diffraction image of transverse section sample. A area is of substrate, B area is transitional layer and C area is of the deposition layer.

4. CONCLUSION

1,Multi-ion beams of MIB mixing technology is a new type of practical modified technology to material surface.
2,The modified layer is composed of three layers as coat (Ti+N), transition layer□Ti+Fe+N□, implantation (Fe+N□by AES analysis
3,The analysis of TEM is proved that structure of modified layer is composed of surface □TiN+TiN□, Subsurface of □α-Fe+TiN□ and the inlay of (α -Fe(N)+CrC). The wider transitional layer improves adhesion between the modified layer and substrate and the ability to resist impact
4,The surface Polycrystal film of ($TiN+Ti_2N$) is very fine, its size is around 200□.
5, The service life of a tool and a die treated by MIB in site is has increased by 2~4 times.

REFERENCES

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TABLES

Table 1 hardness of MIB deposited and implanted

value \ coefficient		N current density(μA/cm)			
		10.14	19.3	23.12	28.2
Hardness HK	1	1008.8	2120	2001.7	1845.4
	2	1272.0	2561.1	2351.9	1451.9
	3	1105.6	2724.6	1976.3	1439.6
	4	1071.9	2334.9	1881.0	1485.7
	Average value	1114.6	2435.9	2.67.7	1555.7

Table 2 Value of friction coefficient

coefficient value \ technology		N current density(μA/cm)			
		10.14	19.3	24.12	substrate
Friction coefficient	1	0.19	0.1	0.165	0.42
	2	0.15	0.11	0.21	0.34
	3	0.165	0.15	0.20	0.42
	Average value	0.168	0.12	0.185	0.38

Table 3 Result of MIB dynamics mixing treated dies test life

name	Die material	specifications	Service life		Service life comparing
			Before treated	After treated	
hole punching die	W18Cr4V	12.77	6000	30000	5
	LD	M18	7000	21000	3
cutting die	Cr12MoV	M5	8000	25000	3.1
		M6	7000	21000	3

Table 4 The film phases of MIB process in different condition

	TiN	TiN	Ti
	HKL	HKL	HKL
10.14	(111),(210),(220)	(111),(200),(202)	(011),(012) (201),(002)
19.3	(111),(200),(220)	(200),(211),(220)	(002),(012)
24.12	(111),(200),(220),(311)	(211),(202)	(011)
28.2	(111),(200),(220)	(211)	

FIGURES

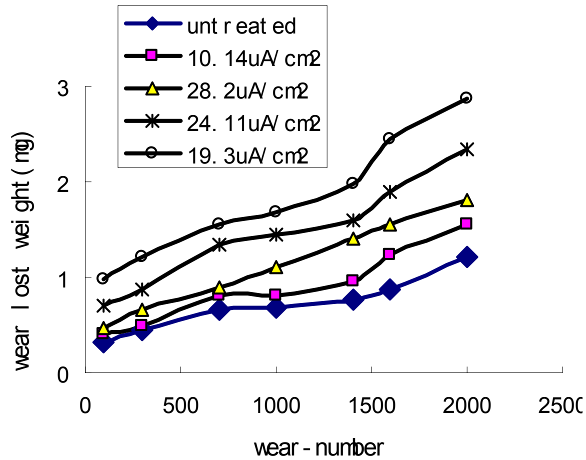

Fig.1 wear curve of different treatment

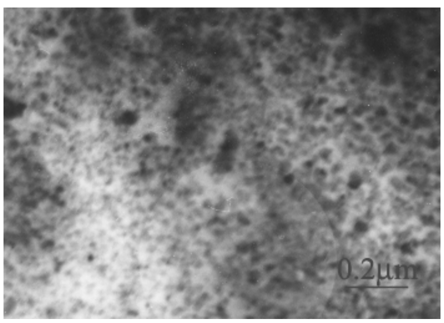

Fig.2 B.F image of TEM of MIB film

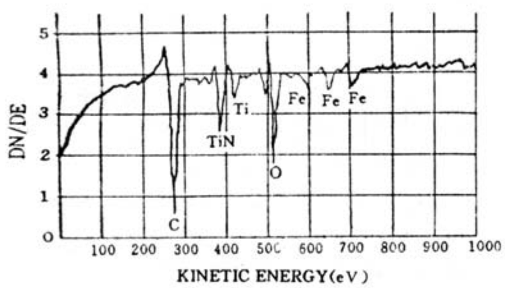

Fig.3 The AES for the surface of sample treated by MIB With N^+ current density of $19.3\mu A/cm^2$

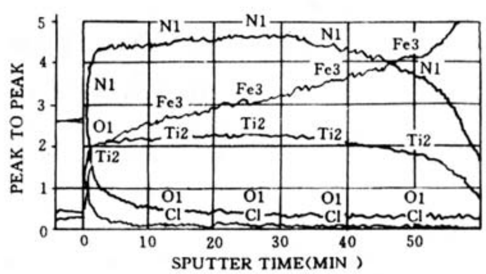

Fig.4 Auger depth profiles for sample treated by MIB with N^+ current density of $19.3\mu A/cm^2$

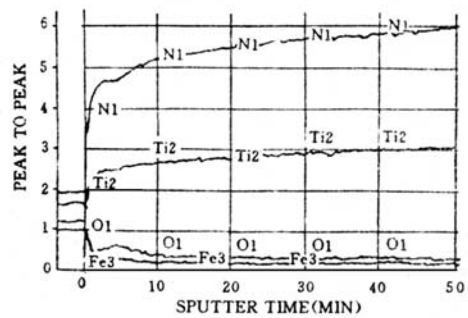

Fig.5 Auger depth profiles for sample treated by MIB dynamics mixing after wearing 1600 times

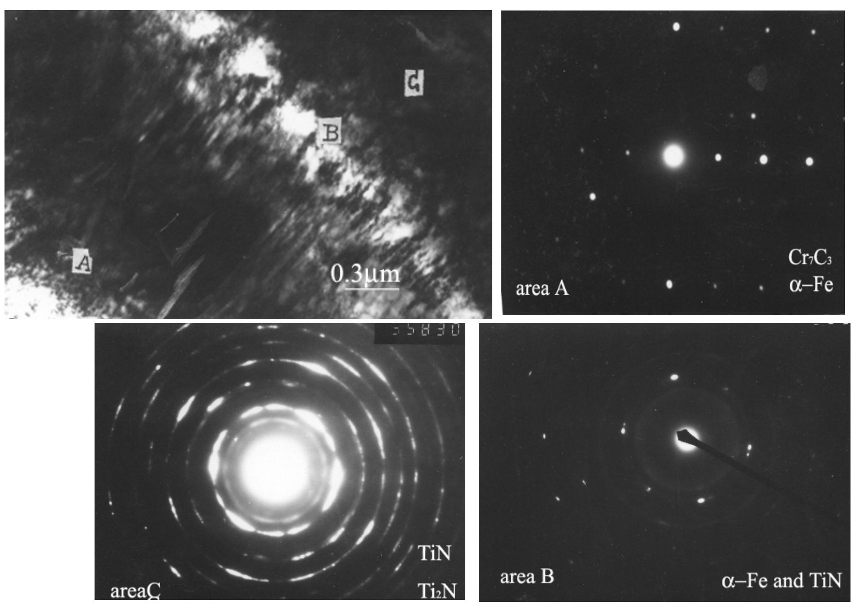

Fig.7 B.F. image of cross-section TEM 3600× and Electron diffraction pattern of A, B, C area