Temperature Effect and Ionic-Beam Treatment on Structure and Mechanical Properties of Nanocrystal Intermetallic Coatings on the Basis of Ni-Al

M.V. Fedorischeva, V.P. Sergeev, A.V. Voronov, O.V. Sergeev, N.A. Popova*, E.V. Kozlov*

* Institute of Strength Physics and Material Science SB RAS, Tomsk, 634021, Russia, fax (382) 49 10 32, E-mai: fmv@pochta.ws Tomsk State University of Architecture and Building, Solyanaya Sq.2, Tomsk, 634003, Russia

Abstract – Phase composition, structure and mechanical properties of the intermetallic coating are investigated by transmission electron microscopy (TEM), X-ray analysis. It is shown that Ni₃Al is the main phase of the intermetallic coating for all investigated samples. There is a small amount of the NiAl and AlFe₃C phases. The application of ion beam treatment (IBT) changes the atomic longrange order parameter and the elastic microstresses of the crystalline lattice. The deposition of coating with IBT of every layer by argon ions decreases wear rate in ~10 times, friction coefficient ~2.5 times, increase microhardness by ~25% and degree of elastic deformation by 18%.

1. Introduction

The last decade has seen a significant interest in nanostructural materials. Nanostructural materials are solids composed of structural elements-mostly crystallites-with a characteristic size (at least in one direction) of a few nanometers [1-3].

The intermetallic compounds Ni_3Al and NiAl have a number of unique properties, and they are objects of numerous investigations [4, 5]. They have a high heat resistance, wear resistance and resistance to oxidation. NiAl (superstructure B2), and Ni_3Al (superstructure $L1_2$) are the most promising for usage as a coating. Magnetron deposition is the most widespread and reliable way of coating deposition of Ni–Al alloys. Highenergy ion beams are an effective way of modification of a structural – phase condition and mechanical properties of the surface layer.

It is well known that the film properties are determined by its microstructure, phase and chemical composition. What microstructure is formed depends on the parameters of the deposition process under which the film is produced [6]. As a result, if the energy and the ions flux are varied, it is possible to fabricate films with different microstructures [7, 8] and hence different properties such as resistivity, strength, microhardness, etc. [9]. The production of nanostructured films is of great importance from the research perspective and also from a technological point of view.

The purpose of the paper is investigation of the temperature effect and ion-beam treatment on the

structure, phase compound and mechanical properties of Ni₃Al nanostructured coatings deposited on the α -Fe substrates.

2. Experimental Details

Ni₃Al films were deposited by a magnetron sputtering system together with an argon ion beam source with an energy of 12.5 keV or without the last. The α -Fe substrates of 15×6×6 mm³ were cleaned, polished to the roughness value $R = 0.8 \mu$ m. The coatings thickness was about 2 μ m for all coatings.

Transmission electron microscopy (TEM) and the X-ray were used to investigate the microstructure, phase composition and the crystalline structure parameters of the intermetallic films [10, 11]. The methods of structured characteristics used were described earlier [12]. The elemental composition of the coating was carried out using mass-spectrometer of secondary ions.

The tests for friction and wear resistance were performed using the 2070SMT-1 machine in conditions of slip in a bosh with synthetic vacuum oil under a constant load of 980 N, the rotation rate was 300 rpm and the testing time was 30 min. The total thickness of the coating and wear was determined by the weight method and controlled by a profilometer 296. The data of the two methods coincided within the limits of measurement errors.

Microhardness and elastic modulus were measured by a nanoindenter "NanoTest 600" using the Bercovich indentor.

The phase composition, structure and mechanical properties were investigated on the following samples at a temperature of 293K: (1) coatings without IBT, (2) coatings with IBT and the radiation dose of $1.2 \cdot 10^{17} \text{ cm}^{-2}$, (3) coatings with IBT and dose of $3.6 \cdot 10^{17} \text{ cm}^{-2}$, (4) at a temperature of 473K with IBT and a dose of $3.6 \cdot 10^{17} \text{ cm}^{-2}$.

3. Results and Discussion

It was shown by X-ray analysis and TEM of thin foils that Ni₃Al is the main phase of the deposited coatings (Figs. 1, 2).



Fig. 1. X-ray diffraction pattern of the Ni₃Al deposited at substrate temperature 473 K



Fig. 2. Diffraction patterns of the Ni₃Al intermetallic coating (a) and the AlFe₃C phase (b)

There is some amount of NiAl and AlFe₃C phases in the samples with IBT (< 5%). It is necessary to note that there are ring-type and separate reflexes for the given condition of this phase. It means that the AlFe₃C phase can be both in the amorphous and crystalline conditions. This phase is located inside the Ni₃Al grains and on the interface as particles a few nanometers in size. The appearance of the carbide phase is connected with the penetration of the carbon atoms in the Ni_3Al coating during bombardment by argon ions of the condensed vacuum oil molecules on the deposited layer of coating.

The results of sectioning the element of the coating obtained by SIMS support this assumption. It is shown (Table 1) that there is carbon ($\sim 2 \text{ at.}\%$) in coatings (set of samples 2, 3, 4) deposited in conditions of ionbeam processing. In coatings without IBT (set 1) no carbon is observed.

Table 1. Element composition of the Ni₃Al coating (relative units)

Set of sample	Ni	Al	С
1	140	115	0
2	142	101	35
3	141	113	43
4	144	110	38

In Table 2 the structural characteristics of the intermetallic coatings are shown. The crystalline lattice parameter of coating with IBT is a little bit lower than in the coatings without ion-beam treatment. The lattice parameter of the coating with IBT at the temperatures of 293 and 473 K is a little bit higher and is close to the values of the lattice parameter of stoichiometric composition [5].

The long-range order parameter for all coatings is low enough and it is equal to $\eta \approx 0.13$ and 0.09 correspondingly for the coatings with IBT at the RT. It is a higher for the coating without IBT and is equal to $\eta \approx 0.18$. The coating with IBT, obtained at the deposition temperature of 473 K is higher and is equal to $\eta \approx 0.25$. In [13] it was shown that the ordering in the intermetallic coating is achieved by annealing at a temperature of 1073K. In our case, the temperature of the coating deposition is much lower. This temperature results in some increase in the order parameter in coatings. In [14] the ordering of the Ni-Al system in the concentrations range of 22-27 at.% was studied. At increase of the substrate temperature up to 790 K, the Ni₃Al they obtained the Ni₃Al ordered phase with the lattice parameter of 0.3560 nm. At the same time, the increase in the temperature leads to increasing the coating grain size. In our case, the coating grains size increase with IBT at elevated temperature up to 55 nm.

The elastic stresses of the intermetallic coating with IBT are in an interval of 36–58 MPa. The elastic stresses of the coating with IBT have minimal values (Table 2) at elevated temperature. The IBT of coatings provides fuller intermixing of the coating layers and inhibits crystallization preventing from ordering and grain growth. At the same time, the temperature rise accelerates these processes, being a competitive factor. Apparently IBT also prevents the grain growth in the coating, with the grain size of Ni₃Al in samples without IBT being always higher than in those with IBT.

Wi	State	Lattice parameter,	Long-range order	Internal elastic	Grain size,
Т. К	Dose	11111	parameter	suesses, wil a	11111
373	$1.2 \cdot 10^{17}$	0.3565 ± 0.0005	0.13 ± 0.03	40 ± 5	22
373	$3.6 \cdot 10^{17}$	0.3560 ± 0.0005	0.09 ± 0.03	58 ± 5	10
473	$3.6 \cdot 10^{17}$	0.3570 ± 0.0005	0.25 ± 0.05	36 ± 5	55
Without	t IBT. 373 K	0.3567 ± 0.0005	0.18 ± 0.05	85 ± 10	48

Table 2. Structural characteristic of the Ni₃Al intermetallic coatings

Table 3. Mechanical characteristics of Ni₃Al intermetallic coatings

Set No	Microhardness, GPa	Elastic strain/ total strain	Wear rate, mg/hour	Friction coefficient
1	5.61 ± 22	0.27	7.8 ± 2.2	0.027 ± 0.003
2	6.99 ± 23	0.32	2.8 ± 0.8	0.019 ± 0.002
3	-	-	1.3 ± 0.2	0.016 ± 0.004
4	—	-	0.8 ± 0.2	0.011 ± 0.001

Microhardness and elastic recovery were measured from the load versus nanindenter penetration depth curve. Microhardness of the coatings without IBT (set 1) is close to 6.21 Gpa for nanocrystal Ni₃Al with an average grain size of 26 nm obtained by the intensive plastic deformation method [15]. The elastic deformation is 27% from the total one. The microhardness and elastic deformation increase up to 25% and 18% correspondingly at the deposition of coatings under ion beam bombardment (set 2). The observed microhardness can be related to decreasing of the average size of grains of the Ni₃Al phase, and the appearance of the fine-dispersed NiAl and AlFe₃C phases.

The ion beam treatment of the deposited coating also leads to a considerable (2.8 times) reduction of the wear rate (Table 3). When the intensity of the ionbeam effect (radiation dose) increases 2 times the wear rate of coatings decreases 2.2 times in addition. The subsequent decreases of the wear rate of the coatings can be achieved when the ion deposition with IBT is applied at a temperature of 473 K. Such coatings demonstrate a wear rate ~ 10 times less than that without IBT.

A similar nature can be observed for the coatings friction coefficient, but in lesser degree. The coatings from the fourth set demonstrated the maximal decrease in the friction coefficient ~ 2.5 times compared with the coatings from the first set (Table 3).

The observed decrease in the wear rate and coatings friction coefficient obtained under ion beam processing at the room temperature can be related to decreasing in the grain size (Table 2) of the coating, and to decreasing of the internal elastic stresses.

Thus, the probability of brittle failure of the coating into large hard debris, which acting as an abrasive, usually result in increasing the wearing of the α -Fe soft substrate. Increase in the wear resistance of the coatings in the fourth set can be related to further decrease in the internal elastic stresses, which the least compared with the samples from the second and third sets, and with some increase in the long-range order parameter (Table 2).

4. Conclusions

1. With the help the X-ray and TEM methods it was shown that the main phase of the deposited coating was the Ni_3Al with average grain size of 10–55 nm.

2. Using the layer-by-layer deposition of the coating with the treatment of every layer by argon ions with an energy of 12.5 keV, it is possible to decrease the wear rate ~10 times, friction coefficient ~2.5 times, and to increase microhardness by ~25% and the degree of elastic deformation by 18%.

3. IBT of the coatings reduces the grains size of the main phase, decreases the internal elastic stresses, decreases the long-range order parameter, and leads to the appearance of a small amount of NiAl and AlFe₃C phases.

4. The temperature rise of the coating deposition with IBT results in increasing of the order parameter, the lattice parameter and the decreasing the internal elastic stresses compared with the coatings deposited at the room temperature.

References

- [1] H. Gleiter, Nanostructed Materials 6, 3 (1995).
- [2] C.T. Liu, P. David, Intermetallic Compounds, Principles and practice. Practice. **2**, 17 (1994).
- [3] B. Daniel Miracie Ramgopal Darolia, Intermetallic Compounds, Principles and practice, Practice 2, 54 (1994).
- [4] T. Lin, K.Y. Ahn, J.M.E. Harper, P.V. Madakson, P.M. Fryer, Thin Solid Films 154, 81 (1987).
- [5] N.S. Stoloff, International Materials Reviews 34 4, 153 (1989).

- [6] J. Musil A.J. Bell, M. Cepega, J. Zeman, Surface Coating Technology 96 359 (1997).
- [7] J. Musil, J. Vlcek, Material Chemictry and Physics 54, 116 (1998).
- [8] R.W. Knoll, E.R. Bradley, Thin solid Films 117, 201 (1984).
- [9] J. Musil, V. Jesek, M. Benda, J. Vlcek, Thin Solid Films **317**, 468 (1998).
- [10] B.E. Warren, X-ray diffraction, Addison Wesley Publision Company Reading Massachusetts Menlo Park California, London Don Mealls, Ontario, 1969, pp. 381–400.
- [11] K. Endrus, D. Dayson, S. Kwien, *Ellectrono-gramm and its interpretation*, Moscow, Mir, 1971, pp. 256–300.
- [12] V.P. Sergeev, M.V. Fedorischeva, N.A. Popova, E.V. Kozlov, Chernaya Metallurgiya 6, 70 (2003).
- [13] Sandip Bysakh, P.K. Das, and K. Chattopadhyay, Scripta Mater. 44, 1847 (2001).
- [14] H.T.G. Hentzell, B. Anderson, and S.E. Karlson, Acta Metal **31**, 1131 (1983).
- [15] A.V. Korznicov, S.P. Idrisova, I.Yu. Pishmentsev, Metalls 2, 83–86 (2000).