



# Structure, hardness and corrosion behavior of a gradient CrN<sub>x</sub> thick coating applied to turbine blades

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## ABSTRACT

In order to protect turbine blades from solid particle erosion, a gradient CrN<sub>x</sub> coating was deposited on 10% Cr heat-resistant steel by ion plating; the thickness of the coating was about 40 μm. The chemical composition, microstructure and nano-hardness of gradient CrN<sub>x</sub> coating were analyzed. The bonding force was determined using scratch test. The potentiodynamic polarization and high-temperature oxidation tests were respectively conducted to investigate the corrosion behavior. The results indicate that gradient variation of phase structure, chemical composition and nano-hardness in the coating is found, and the bonding force with substrate is excellent. The microstructure obtained can enhance the corrosion performance of the substrate. The corrosion resistance improvement is not only attributed to the increase of coating in thickness, but also to internal microstructure and chemical composition of coating. Based on SEM and TEM observations, the cross-sectional fracture of the coating shows nano-crystalline and fine columnar crystalline structures. There are no penetrable pinholes, which could validly reduce the electrolyte transferring to inner substrate. In addition, the corrosion resistance of coating is further improved by the formation of nitrogen and chromium rich transition layers.

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## 1. Introduction

CrN coatings, which exhibit high hardness, superior wear resistance, excellent corrosion resistance, and good oxidation resistance, have been extensively used as protective coating on various tools and dies [1,2]. A number of studies on the oxidation of CrN coatings have indicated that the limit of oxidation temperature is commonly about 700 °C [3,4].

Solid particle erosion (S.P.E.) of steam path surfaces is a major concern in steam turbines. This is mainly due to the formation of magnetite, an oxide of iron, on the inside of steam generator-ferritic alloy tubes, headers and steam lines exposed to high steam temperature [5]. The component materials of steam turbines tend to be damaged by high-temperature oxidation and erosion wear caused by the impact of solid particles under actually operational condition. Ceramic coating materials are expected to have high erosion and heat resistance. However, a very thin, hard layer on a steel substrate can hardly give rise to the formation of a mechanically sound structure: the differences of both hardness and Young's modulus between coating and substrate do not provide a good distribution of contact stresses; furthermore, in

some application the coated system can behave even worse than the uncoated one.

To increase the bonding force of coating with substrate and minish the differences of both thermal expansion coefficients and mechanical properties between substrate and CrN coating, a gradient CrN<sub>x</sub> thick coating by ion plating was deposited on 10% Cr heat-resistant steel. Adhesion is enhanced by deposition of a high Cr interlayer which accommodates the CrN internal stresses and allows thicker coatings to be produced, with significant improvements in toughness, adhesion, impact resistance and corrosion resistance [6,7]; however, the presence of a relatively thick Cr buffer layer often involves a significant reduction in hardness and erosion resistance of the plated coating [7]. To overcome these limits, a high N layer was deposited between the high Cr layer and the CrN coating.

The research work was focused on the microstructure, mechanical property, bonding force and corrosion resistance of CrN<sub>x</sub> coating, with the objective of applying a gradient ion-plated coating on dry steam turbine blades operated at high temperature.

## 2. Experimental procedure

The base material used in this study is 10% Cr heat-resistant steel. This material is used in steam turbine blades that will be seriously subjected to solid particle erosion under operational condition. The steel was forged after the electroslog remelting

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**Table 1**

Chemical composition of 10% Cr heat-resistant steel.

	Element									
	C	Cr	Ni	Mo	V	Nb	N	Si	Mn	Fe
wt.%	0.19	10.8	0.4	1.0	0.17	0.41	0.06	0.55	0.58	Balance

process. It was quenched by oil at 1100 °C and tempered at 660 °C for 2 h. The hardness of the prepared substrate was in the range of HB 305–311. The chemical composition of substrate is listed in Table 1. The dimensions of the substrate are 20 cm × 20 cm × 5 cm. All of the specimens were firstly ground with a series of SiC abrasive papers and subsequently polished through 1.0 μm diamond powder. The CrN<sub>x</sub> coating was deposited by an arc-ion plating method. Prior to deposition, the substrate was degreased by acetone ultrasonically and dried, and then it was cleaned by chromium ion bombardment with a bias voltage of −1000 V for 10 min. At substrate bias of −400 V, the chromium and nitrogen rich layers were deposited, the nitrogen pressure varied from 0 to  $6 \times 10^{-2}$  Pa and lasted for 40 min; the nitrogen pressure was then adjusted to about  $3.5 \times 10^{-1}$  Pa and lasted for 20 min, the nitrogen rich layer was followed by the thick chromium nitride layers deposition. The detailed deposition parameters are shown in Table 2.

The phase formation of the CrN<sub>x</sub> coating was analyzed with low-angle X-ray diffraction (XRD, Panalytical X'pert PRO diffractometer) using Cu Kα radiation and X-ray photoelectron spectroscopy (XPS, PHI 5700 ESCA). The depth compositional profiles were measured using electron probe microanalysis (EPMA, Shimadzu, EPMA 1610). The microstructural evaluation was conducted using scanning electron microscope (SEM) and transmission electron microscope (TEM) (SEM, CamScan MX 2600; TEM, JEM-2010). Microhardness were measured using a Nano-Indenter XP Tester with a Berkovich indenter at a load resolution of ±75 nN and displacement resolution of ±0.04 nm. The bonding force of coating with substrate was determined at loading rate of 100 N/min using scratch tests. The primary principle of scratch test is that a specific indenter slips on the coating surface with continuously increasing load (maximum applied load 100 N). Coating and substrate convert and transmit acoustic emission signals with continuous fluctuation when the load reaches its critical load ( $L_c$ ). The first value,  $L_c$ , of which acoustic emission peak fluctuates, is used to evaluate the bonding force of coating with substrate.

Corrosion behavior was assessed by potentiodynamic anodic polarization in a saline solution of 3.5% NaCl at room temperature and high-temperature oxidation experiment at 800 °C. The potentiodynamic experiments were conducted through a three-electrode cell connected to a ZAHNER electrochemical workstation. An Ag/AgCl electrode was used as a reference electrode and a platinum electrode was the auxiliary one. Polarization curves were obtained by sweeping the potential with a potential scan rate of 1 mV/s. Oxidation experiments were carried out in static air isothermally at 800 °C. The weight gain during oxidation was measured by an analytical balance with a precision of ±0.0001 g. The specimen oxidized after 100 h was examined by SEM, energy dispersive spectrometer (EDS) and XRD respectively.

The solid particle erosion tests of the substrate and the deposited coating material were carried out using an erosion test

**Table 3**

Details of erosion test conditions.

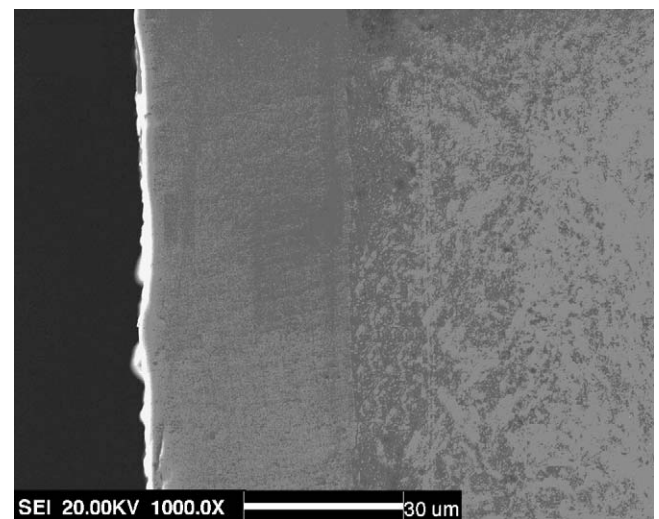
Test conditions
Erodent material: Fe <sub>2</sub> O <sub>3</sub> ferric oxide
Particle size range: 40–45 μm
Particle velocity: 210 m/s
Test temperature: 600 °C
Impact angle: 30°, 90°

rig. For the detailed description of the erosion test rig and the test procedure refer Ref. [8]. The parameters of erosion testing are presented in Table 3. The erosion resistance performance was characterized by the mass wear rate, defining as mass loss of coating per unit mass of erodent once steady-state conditions had been obtained. Prior to and after the erosion test, the substrate and the coated specimens were respectively cleaned ultrasonically with acetone three times, dried and weighed using an electronic weighing balance with an accuracy of 0.1 mg.

### 3. Results and discussion

#### 3.1. Characterization of the coating

Fig. 1 shows a typical cross-section view of a coated specimen, which indicates the deposition of a very uniform and dense coating of approximately 40 μm in thickness. The substrate–coating interface is observed to be quite smooth and free of cracks and pores. Fig. 2 shows SEM cross-sectional images of a fractured coating after bending test. It reveals to be very dense and smooth without specific feature associated with the columnar crystallite. In order to obtain microstructural information at a finer scale, TEM observation of cross-sectional specimen of the CrN<sub>x</sub> coating was carried out. Fig. 3 shows the bright-field TEM image. The inset (1) in Fig. 3 is the selected area diffraction pattern (SADP) containing some nanocrystalline phases. Therefore, it is labeled by arrow 'a' in the directed zone. The inset (2) is the SADP obtained from a



**Fig. 1.** SEM cross-sectional micrographs of the coated CrN<sub>x</sub> specimen on 2Cr10MoVnNbN heat-resistant steel.

**Table 2**Nominal experimental parameters of depositing CrN<sub>x</sub> coating.

Substrate	Target	Nitrogen pressure (Pa)	Temperature (°C)	Bias (V)
10% Cr heat-resistant steel	Cr > 99.5%	$6 \times 10^{-2}$ to $2 \times 10^{-1}$	350–400	200

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