

Article

Structure, Mechanical Properties and Water Vapor Corrosion Resistance of AlCrNbSiTiN High-Entropy Nitride Coatings Deposited by RF Magnetron Sputtering

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Abstract: High-entropy nitride AlCrNbSiTiN coatings were deposited by RF magnetron sputtering at different bias voltages. The structure, mechanical properties and water vapor corrosion resistance of the coatings were systematically studied. The coatings exhibit a face-centered cubic (FCC) structure, while achieving a hardness up to 35.8 GPa. The main wear mechanisms of the coatings are adhesive wear and oxidation wear. After 200 h of water vapor corrosion, the content of O in the coatings is 4.30 at.%.

Keywords: high-entropy nitride coatings; RF magnetron; structure; nanohardness; tribological properties; water vapor corrosion resistance



Citation: Wang, X.; Liu, J.; Liu, Y.; Li, W.; Chen, Y.; Yang, B. Structure, Mechanical Properties and Water Vapor Corrosion Resistance of AlCrNbSiTiN High-Entropy Nitride Coatings Deposited by RF Magnetron Sputtering. *Coatings* **2024**, *14*, 1006. <https://doi.org/10.3390/coatings14081006>

Academic Editor: Aivaras Kareiva

Received: 16 July 2024

Revised: 25 July 2024

Accepted: 6 August 2024

Published: 8 August 2024



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1. Introduction

Machine components and industrial facilities suffer from serious tribological damage and corrosion, which deteriorate the mechanical properties of material surfaces and ultimately result in material failure [1–4]. The deposition of hard coatings by physical vapor deposition (PVD) can effectively improve the corrosion resistance properties of materials [5,6]. However, traditional protective coatings can barely satisfy harsh industrial production applications.

In 2004, Cantor [7] first proposed a new alloy system, multi-principal-element alloy, which was subsequently defined by Yeh as a high-entropy alloy (HEA) [8]. HEAs contain at least five elements, with each element content ranging from 5 at.% to 35 at.%. Based on a sluggish diffusion effect and lattice distortion effect [9–11], HEAs exhibit excellent mechanical properties [12] and outstanding corrosion resistance [13,14]. In particular, high-entropy alloy coatings containing aluminum (AlCrFeNi series) show substantially improved resistance to water vapor corrosion [15]. FeCrAlMoSiY coatings prepared on a Zr alloy have a dense amorphous structure, which retain smooth morphology and structural integrity with no major defects such as cracks, holes and spallation, after oxidation at 1200 °C in water vapor for 2 h, [16]. The CrCuFeMoNi coatings prepared by magnetron sputtering on a Zr alloy not only have a hardness as high as 12.5 GPa, but also show excellent corrosion resistance at 360 °C and 18.6 MPa in water vapor [17]. The dense continuous oxides (Cr₂O₃ and FeCr₂O₄) formed in the process of water vapor oxidation of the coatings can effectively prevent further diffusion of oxygen into the inner coatings and provide protection for the substrate. High-entropy nitrides (HENs) have been widely investigated by researchers to obtain both the high hardness and thermal stability of the corresponding ceramic coatings. Compared to conventional nitride, HENs exhibit excellent thermal stability [18,19], enhanced corrosion resistance [13,14] and wear resistance [12] due

to the high-entropy effect [20,21], lattice distortion effect [10,11], slow diffusion effect [9] and cocktail effect [22,23]. In addition to high hardness, it also has excellent plasticity, toughness and oxidation resistance and other comprehensive properties [19,24]. Du-Cheng Tsai et al. [25] prepared (TiVCrZrHf)N multiple-principal-element coatings by magnetron sputtering. With the increasing thickness of the coatings, the coatings transformed from continuous amorphous (TiVCrZrHf)N layers to randomly oriented small (TiVCrZrHf)N grains, and finally into FCC-phase columnar grains. The coatings exhibit a hardness of 33.5 GPa at a bias of -100 V and a substrate temperature of 723 K. The (AlCrTiZrMo)Si_xN coatings under various silicon contents were investigated. With the increase of silicon content, the coatings changed from (200) preferred-growth columnar crystals to a nanocrystalline/amorphous structure. When the silicon content is 4.5 at.%, the maximum hardness and modulus of the coatings are 28.5 GPa and 325.4 GPa, respectively [26]. Because of the high hardness of HENs and the complex composition and dense structure of the oxide films, HENs can withstand intense temperature loads and maintain a desirable protective effect on the substrates, which can meet the requirements of temperature and oxidation resistance under harsh working conditions.

In this study, the mechanical properties and water vapor corrosion resistance of AlCrNbSiTiN coatings were investigated. The AlCrNbSiTiN nitride coatings were prepared by RF magnetron sputtering with different bias voltages. The morphology, phase composition, mechanical properties and water vapor corrosion resistance of AlCrNbSiTiN coatings were systematically analyzed, which could provide significant scientific support for the water vapor corrosion protection of high-entropy nitride coatings.

2. Experimental Details

2.1. Coatings Preparation

The AlCrNbSiTiN coatings were deposited on Si (100) and stainless steel by RF magnetron sputtering. The deposition system has been described in detail in previous work [27,28]. The substrates were washed in acetone for 15 min using an ultrasonic cleaning machine before the coating deposition process. The substrates were first etched with Ar⁺, and then the Cr and CrN interlayers were deposited, respectively. The AlCrNbSiTiN coatings were prepared by RF magnetron sputtering. The deposition temperature of all coatings is 200 °C. The detailed deposition parameters are shown in Table 1.

Table 1. Deposition parameters of the AlCrNbSiTiN coatings.

Layer	Bias Voltage, V	Gas/ Pressure, Pa	Target/ Current (Power)	Duration/ min
Ar ⁺ etch	−150	Ar/0.5	Ti/150 A	15
Cr	−150	Ar/0.5	Cr/65 A	5
CrN	−150	Ar+N ₂ (1:1)/1	Cr/65 A	5
AlCrNbSiTiN	−50, −100, −150, −200	Ar+N ₂ (1:1)/1	AlCrNbSiTi /700 W	60

2.2. Coatings Characterization

The surface and roughness of the coatings were examined using Atomic Force Microscopy (AFM, Shimadzu, SPM-9700HT, Kyoto, Japan) and Scanning Electron Microscopy (SEM, TESCAN MIRA 3, TESCAN Brno, Ltd., Oxford, UK). With the use of an energy dispersive spectrophotometer (EDS, Oxford Instruments X-MAXN, Oxford, UK), the coatings' chemical composition was examined. X-Ray Diffractometry (XRD, PANalytical, XPert Pro, Xi'an, China) was employed to obtain the phase structure of the coatings. The hardness and elastic modulus of the coatings were measured using a continuous-stiffness approach with a nano-indenter (NANO, KLA, G200, Milpitas, CA, USA) [29]. Using a force of 2.5 N and a rotation speed of 200 rpm, a ball-on-disk tribometer (HuaHui, MS-T300, Lanzhou, China) was used to assess the coatings' resistance to wear. A detailed description of the water vapor corrosion evaluation can be found in earlier work [30].

3. Results and Discussion

3.1. Morphology and Chemical Composition Analysis

The morphologies of the AlCrNbSiTiN coatings are shown in Figure 1. It can be clearly observed that the particles and holes, as well as the surface roughness, decrease with increasing bias voltage. Specifically, the surface roughness decreases from 19.28 nm to 11.67 nm when the bias voltage increases from 50 V to 200 V. The cross-section morphology of the AlCrNbSiTiN coatings is shown in Figure 2. The thickness of the coatings decreases from 2.56 μm to 1.63 μm with increasing bias voltage. As the bias voltage increases, the kinetic energy of the ions will increase, resulting in an enhanced resputtering effect and hence a denser coating layer [31–34]. Chemical composition as a function of bias voltage for AlCrNbSiTiN coatings is illustrated in Figure 3. No significant change in the proportion of coating elements could be identified, indicating that bias voltage does not noticeably affect the chemical composition of the coatings.

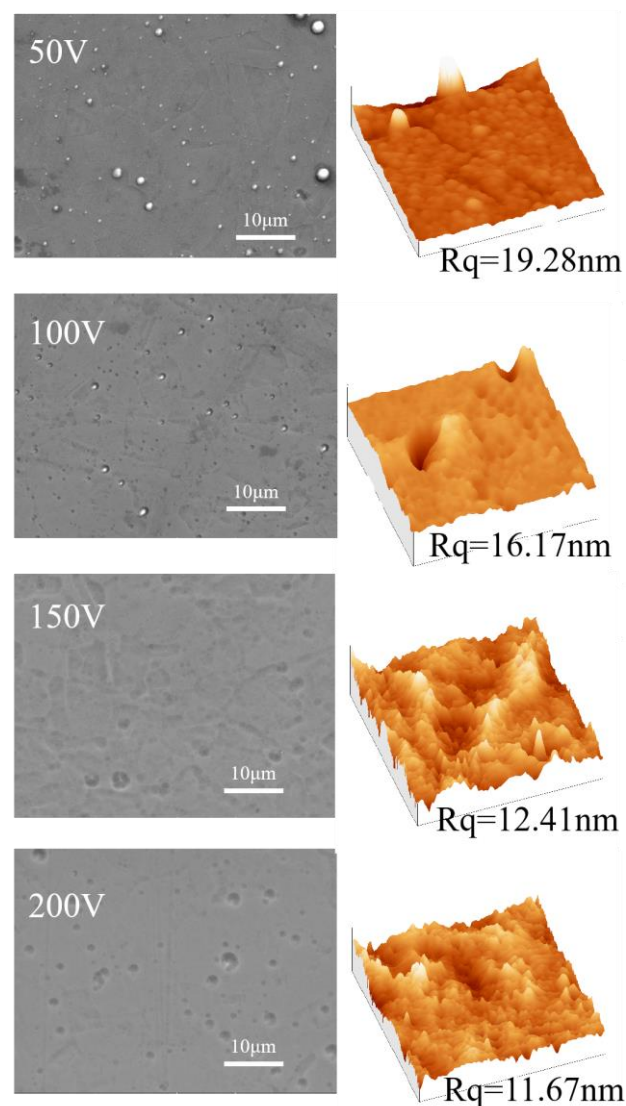


Figure 1. The surface, AFM and three-dimensional morphology of the AlCrNbSiTiN coatings with different bias voltages.

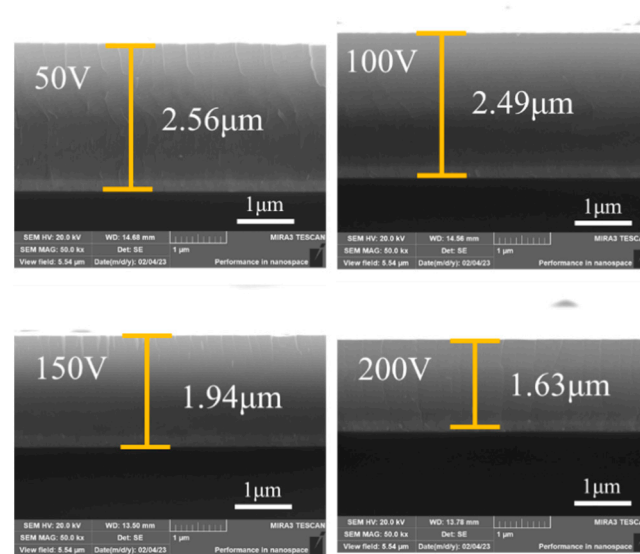


Figure 2. The cross-section morphology of the AlCrNbSiTiN coatings with different bias voltages.

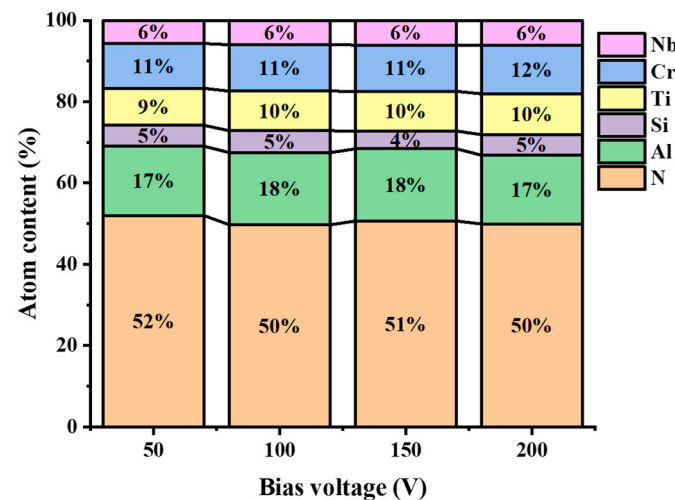


Figure 3. The chemical composition as a function of bias voltage for AlCrNbSiTiN coatings.

3.2. XRD Analysis

Figure 4 plots the XRD patterns of AlCrNbSiTiN coatings. The powder diffraction file numbers of the AlCrNbSiTiN coatings and SS are 65-2899 and 88-2324, respectively. All coatings are face-centered cubic (FCC) structures. When the bias is 50 V, there are both (200) and (220) diffraction peaks. With increasing bias, the (200) peak gradually disappears, while the diffraction peak of (220) gradually increases. The (220) peak is obviously widened under a 150 V bias, suggesting the refined grain structure of the coatings. The influence of bias voltage on the grain growth of the coatings is listed below [35,36]. The first point is that with increasing bias, the higher energy bombardment increases the movement speed of the atoms, and the atoms move to the grain boundary faster, which is conducive to grain growth. The second point is that the high-energy bombardment will also bring about high-density defects, which provide additional nucleation sites and are beneficial to grain refinement. The preferred orientation of the coatings is a common phenomenon in physical vapor deposition, while the crystalline orientation of the coatings is affected by various deposition parameters [37,38]. In general, surface energy, strain energy and stopping energy together determine the preferred orientation of the coatings [39,40]. The surface energies of (200) and (220) nitride coatings with face-centered cubic structures are 4.94 J/m² and 6.99 J/m², respectively [41–43]. Therefore, the (200) crystal face has the lowest surface energy. The strain energy is related to the thickness and stress of the coatings. The increase

in bias voltage will produce a higher energy ion bombardment effect, resulting in increased stress and strain energy. The larger the thickness, the larger the strain energy of the coating. The stopping energy refers to the deposition energy density of an ion in a certain crystalline direction. With increasing bias, the deposition energy density will increase, resulting in increasing stopping energy. Zhao et al. [40] demonstrate the relationship between the preferred orientation of the coatings and the thickness/bias voltage of the coatings. When the bias voltage is relatively low, the surface energy dominates the preferred orientation of the coatings because of the low-level strain energy and stopping energy. Therefore, at relatively low bias voltages, the (200) crystal face is preferentially oriented. At higher bias voltages, although the strain energy and stopping energy of the coatings will both increase, the strain energy has little effect on the preferred orientation because of the thinness of the coatings, while the stopping energy remains a dominant factor in determining preferred orientation. Therefore, at high bias voltages, the (220) crystal face is preferentially oriented in line with its minimum stopping energy.

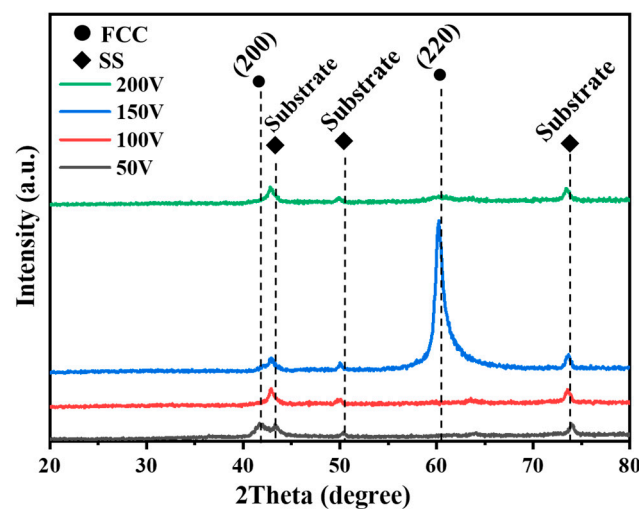


Figure 4. XRD patterns of AlCrNbSiTiN coatings deposited at different bias voltages.

3.3. Mechanical and Tribological Properties

The hardness and elastic modulus of the AlCrNbSiTiN coatings are shown in Figure 5a. The elastic modulus decreases consistently from 349.7 GPa to 329.7 GPa as the bias increases from 50 V to 200 V, while the hardness of the coatings initially increases from 29.76 GPa to 35.8 GPa and slightly decreases to 34.37 GPa at a 200 V bias voltage. The coatings exhibit finest hardness of 35.8 GPa, which precede many reported coatings [27,44,45]. At low bias voltages, the structure of the coatings is relatively loose with noticeable particle defects, resulting in low hardness. At high bias voltages, the coatings become denser with a refined grain structure, leading to the improved hardness of the coatings. When the bias voltages increase from 50 V to 150 V, the result of H/E increases from 0.0856 to 0.1078, and the result of H^3/E^2 also increases from 0.2275 to 0.4166 GPa, respectively. Nevertheless, the H/E and H^3/E^2 results decrease to 0.1043 and 0.3794 GPa at a 200 V bias, respectively. The coatings prepared at a 150 V bias voltage are considered to exhibit optimal hardness and toughness.

The wear scar morphology and EDS results of the AlCrNbSiTiN coatings are shown in Figure 6. No substrate elements could be identified, indicating that the protection of the coatings has not failed. The white phase on the surface of the abrasion is oxide. At a 50 V bias voltage, the grinding mark surface is covered with oxides (the white phase). It can be seen that the proportion of O increases significantly in the scratched surface. When the bias reaches 100 V, the oxides cannot completely cover the abrasion mark. For samples at a 150 V bias, a local increase in the proportion of Cu is detected on the surface of the wear marks, which is due to the adhesive wear caused by the Cu-Zn alloy grinding ball. The wear width of the coatings at a 200 V bias voltage is at its smallest at 275 μm .

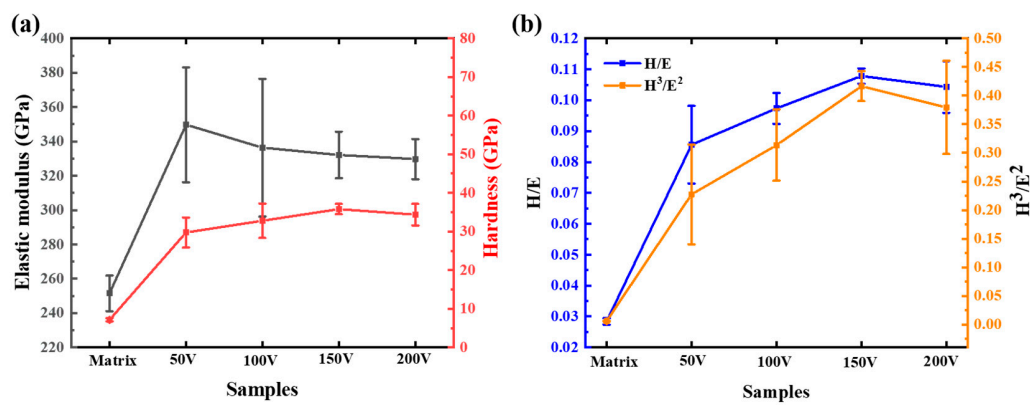


Figure 5. Variation of the hardness and elastic modulus (a) and the H/E and H^3/E^2 values (b) for the AlCrNbSiTiN coatings as a function of bias voltage.

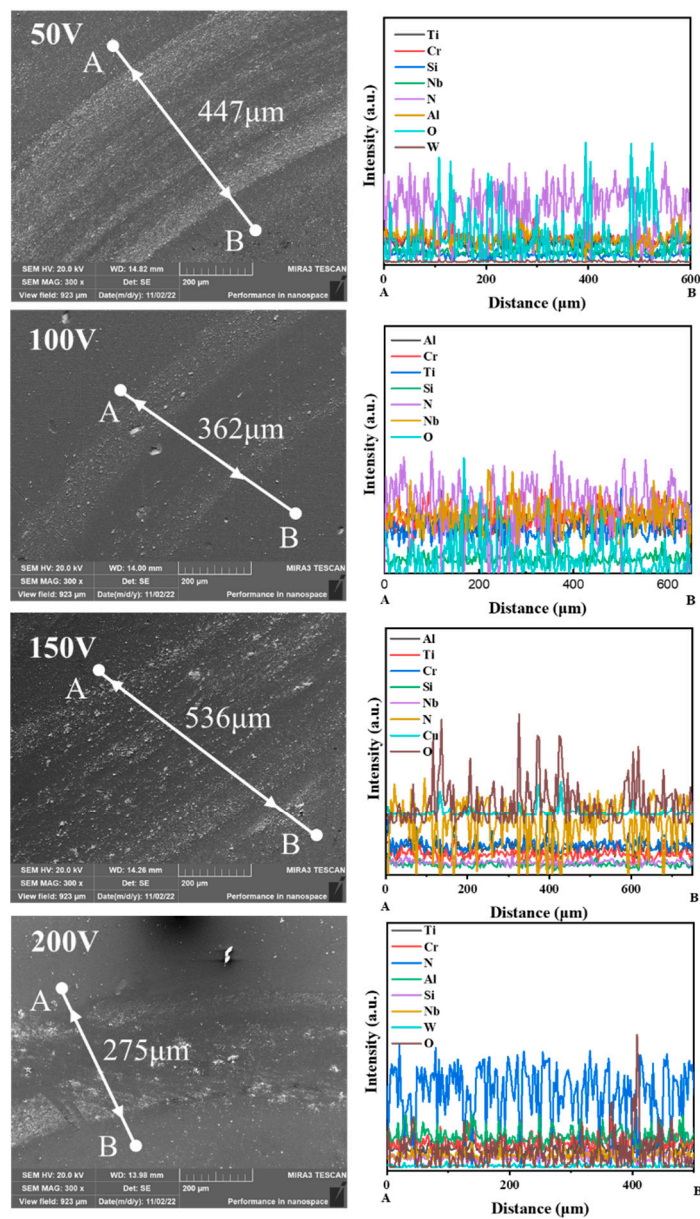


Figure 6. The wear scar morphology and EDS results of the AlCrNbSiTiN coatings.

3.4. Water Vapor Corrosion Resistance Properties

In order to analyze the water vapor corrosion resistance of the AlCrNbSiTiN coatings, a water vapor corrosion resistance experiment was carried out at 700 °C for 100 h, 150 h and 200 h. The morphology of the coatings under different corrosion periods is shown in Figure 7. It was found that the coatings at 150 V and 200 V biases exhibit no obvious morphological changes after 200 h of corrosion. The EDS results of the coatings surfaces are shown in Figure 8. No significant change in the proportion of elements in the coatings can be observed. After 200 h of corrosion, the maximum O ratio on the surface of the coatings is 4.30 at.% at a bias of 150 V.

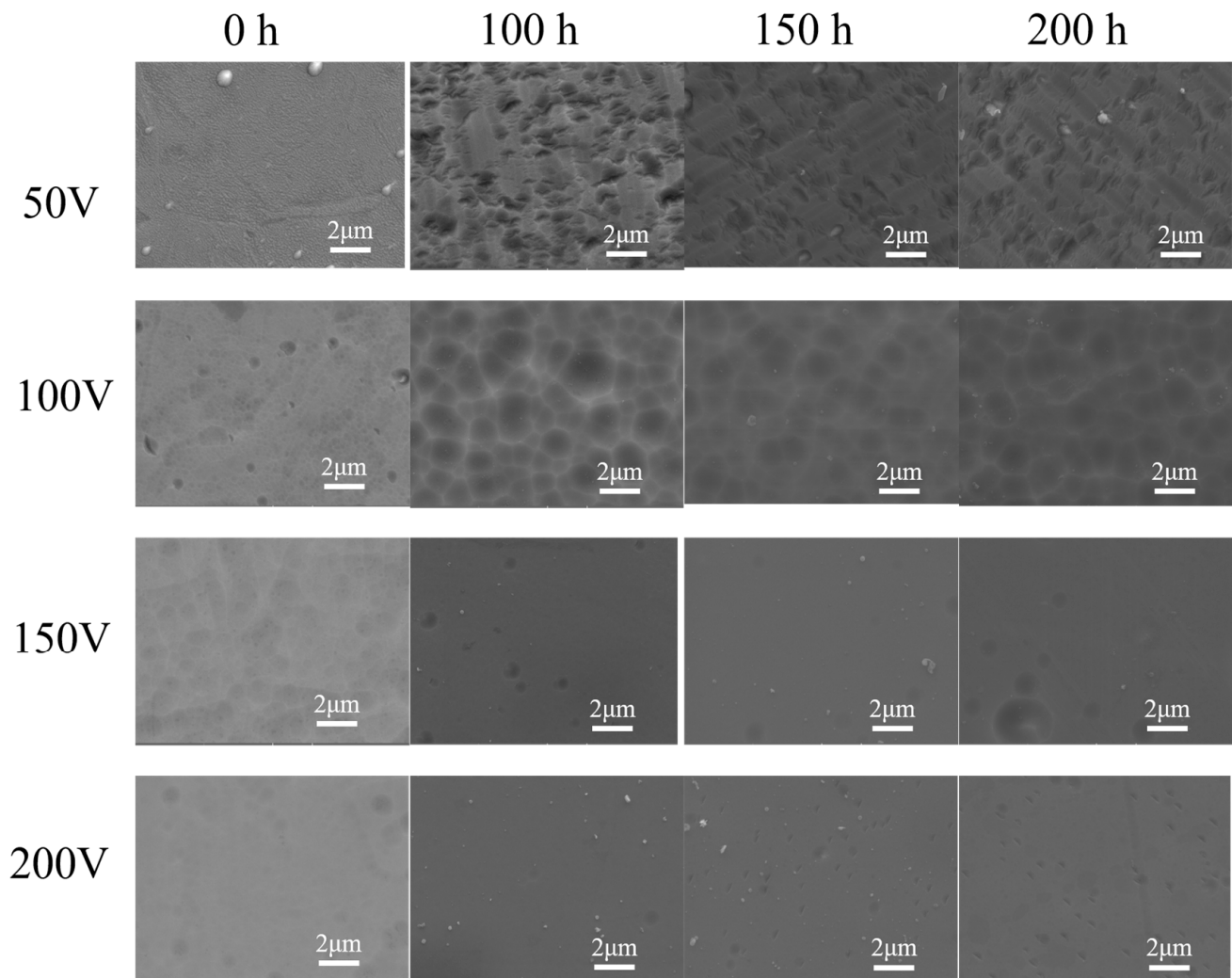


Figure 7. Surface morphology of AlCrNbSiTiN coatings after water vapor corrosion.

Regarding the water vapor corrosion rate, it is suggested that Cr forms $\text{CrO}_2(\text{OH})_3$ or $\text{Cr}(\text{OH})_3$ with water vapor, and its evaporation causes damage to the coatings [46,47]. In the initial oxide formation stage, the corrosion rate is relatively low. However, the subsequent oxide damage stage will cause serious damage to the coatings. After 200 h of water vapor corrosion at 700 °C, the proportion of O elements on the surface of the coatings is lower than 5 at.%, which indicates that the coatings are still in the initial oxide formation stage with a low corrosion rate even after 200 h of corrosion.

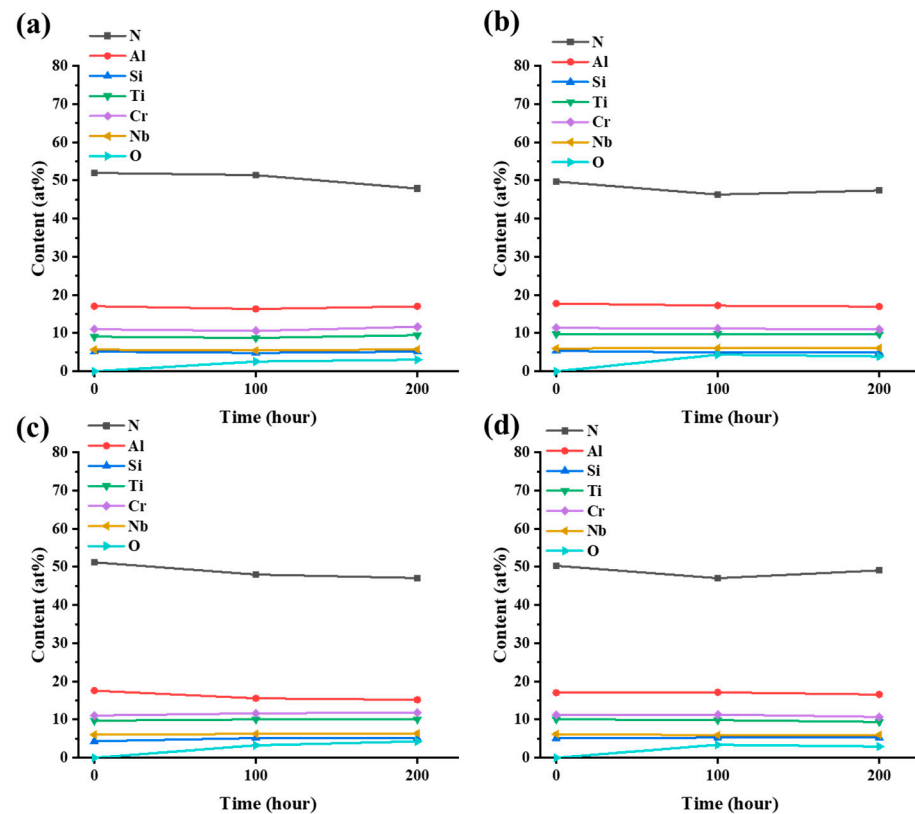


Figure 8. Variation of elemental proportions after water vapor corrosion of AlCrNbSiTiN coatings. (a) 50 V, (b) 100 V, (c) 150 V, (d) 200 V.

4. Conclusions

High-entropy nitride AlCrNbSiTiN coatings were deposited by RF magnetron sputtering at different bias voltages. The structure, mechanical properties and water vapor corrosion resistance of the coatings were systematically studied. At low bias voltages, the (200) crystal faces are preferentially oriented, while the (220) crystal faces are preferentially oriented at higher bias voltages. At a 150 V bias, the coatings achieve a maximum hardness of 35.8 GPa, as well as optimal H/E and H^3/E^2 values, indicating that the coatings have desirable toughness. The main wear mechanisms of the coatings are adhesive wear and oxidation wear. After 200 h of water vapor corrosion, the content of O in the coatings is 4.30 at.%. The high-entropy nitride coatings prepared by RF magnetron sputtering demonstrated excellent mechanical properties and water vapor corrosion resistance, in terms of which the coatings with a bias of 150 V have the best mechanical properties.

Author Contributions: Conceptualization, J.L.; formal analysis, Y.L.; funding acquisition, B.Y.; investigation, W.L.; methodology, Y.L.; project administration, J.L. and B.Y.; resources, W.L. and Y.C.; supervision, Y.L. and Y.C.; validation, Y.L.; writing—original draft, X.W. and J.L.; writing—review and editing, J.L., Y.C. and B.Y. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by National Natural Science Foundation of China (52371073) and the Shenzhen Science and Technology Program (JCYJ20220530140601002).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article.

Conflicts of Interest: Authors Xuanzheng Wang, Yingfan Liu, and Wentao Li were employed by the company CNOOC Safety & Technology Service Co., Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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