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Microstructures and mechanical properties of duplex-treated composite ceramic coatings with and without compound layer

J. Zheng[a], S.H. Zhang[a,**], P. Wu[a], S.C. Kwon[b], M.X. Li[a], S.Y. Liu[a], W.L. Chen[a,*]

[a]School of Materials Science and Engineering, Anhui University of Technology, Maanshan City, Anhui Province 243002, PR China
[b]Department of Advanced Materials Engineering, Chungbuk National University, Chungbuk 641773, South Korea

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Abstract

Duplex-treated composite ceramic coatings with and without compound layer were produced by the combination of plasma nitrocarburizing and follow-up multi-arc ion plating. The microstructures and mechanical properties of the ceramic coatings were investigated by means of X-ray diffraction, X-ray photoelectron spectroscopy, scanning electron microscopy and transmission electron microscopy, in association with property characterization. The results show that the composite ceramic coatings are composed of an inner nitrocarburized layer, a CrN interlayer and an outmost AlCrTiSiN layer. The AlCrTiSiN layer consists mainly of alternately nanoscaled (Cr,Al)N sublayer and amorphous sublayer with a few (Ti,Al)N phases embedded in a matrix of amorphous Si_3N_4 and Si. The compound layer has transformed to the γ′-phase sublayer, which enhances considerably the adhesion strength of the CrN interlayer to compound layer. The improved adhesion strength is attributed to the γ′-phase acting as nucleation sites of epitaxial growth for the CrN phase. The composite ceramic coating with compound layer reveals much higher hardness, bearing capacity and wear resistance when compared with the composite ceramic coating without compound layer. The improvement of mechanical and tribological properties is associated with presence of the γ′-phase sublayer, which provides a smooth transition, gradient in hardness and stress between the substrate and the CrN/AlCrTiSiN coating. In addition, the hard γ′-phase sublayer provides a strong supporting effect for CrN/AlCrTiSiN layer under loading.

Keywords: Multi-arc ion plating; Plasma nitrocarburizing; Composite ceramic coating; AlCrTiSiN superlattice coating

1. Introduction

Plasma nitrocarburizing has been used widely to improve mechanical properties of ferrous components through the formation of a nitrocarburized layer, which is composed of an outmost compound layer and an inner diffusion layer. The compound layer consists commonly of ε-Fe_{2-3}(C,N) phase or/and γ′-Fe_4(C,N) phase [1–5]. In some cases, the compound layer cannot demand for industrial application such as high speed and dry cutting, which is due to its intrinsically brittle characteristic and relatively low wear resistance. Compared with the compound layer, the PVD coatings, such as TiN and CrN, show much higher surface hardness, wear resistance and thermal stability [6–10]. However, the tribological property is often limited by plastic deformation of the substrate, which results in eventual coating failure [5,11]. Therefore, application of these PVD coatings is restrained to some extent. In order to obtain the higher tribological property and load-bearing capacity, a duplex surface treatment combined with nitrocarburizing (or nitriding) and PVD treatment for steels has been proposed and developed [12–14].

The duplex surface treatment can be achieved by nitrocarburizing and follow-up depositing a thin PVD hard coating on the surface of the nitrocarburized samples. The duplex coating is composed commonly of an outmost PVD hard coating and an inner nitrocarburized layer. This duplex coating shows the higher hardness and wear resistance compared with the nitrocarburized layer or PVD hard coating alone due to the strong supporting effect of the nitrocarburized layer for the hard PVD

*Corresponding author. Tel./fax: +86 555 3553789.
**Corresponding author.
E-mail address: chenlin1038@163.com (W.L. Chen).

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coating [10,15]. However, this supporting effect can lose when the hard compound layer decomposes to a soft α-Fe layer (also called as black layer) due to much higher substrate temperature (more than 500 °C) and/or improper ion etching methods. So the compound layer is grounded commonly before PVD treatment. But this process is not always right for industrial application. Because the compound layer is removed, meaning that surface hardness of the nitrocarburized layer is decreased.

In the past decades, the CrN, TiN and Ti(C,N) coatings deposited by CVD or PVD on the surface of nitrided samples have been investigated widely [12–14]. But due to unreasonable deposition parameters, the hard compound layer can decompose to the soft α-Fe layer, resulting in the reduction of adhesion strength of hard coating to substrate. In order to avoid the decomposition of the compound layer to the α-Fe layer, the substrate temperature should be properly lowered and the Cr ions should be selected for ion etch instead of Ti ions or argon ions during the ion bombardment process. To the best of our knowledge, the superlattice coatings of nanocrystalline grain sizes deposited by multi-arc ion plating on the surface of nitrocarburized samples have not been reported yet. In the present study, a commercial T10 steel was performed to be plasma nitrocarburized, and follow-up a CrN/AlCrTiSiN coating of nanocrystalline grains was deposited on the surface of the nitrocarburized T10 steels with and without compound layer by means of multi-arc ion plating. The microstructure, mechanical and tribological properties were systematically investigated.

2. Material and methods

2.1. Sample preparation

The initial material was a commercial high carbon steel (T10 steel) with the chemical composition (wt%) of Fe–0.99C–0.25Mn–0.015S–0.011P–0.025Si. The samples were cut from a hot-rolled bar and then machined to 20 mm diameter and 3 mm thickness. Before plasma nitrocarburizing, all of specimens were polished and cleaned in ethanol. All of the specimens were plasma nitrocarburized in a vacuum chamber at 560–580 °C for 5 h in a mixture of 500 L/h ammonia and 4.2 L/h acetone gases, followed by slow furnace cooling. The nitrocarburized samples were divided into two groups: one group was directly polished to an average surface roughness Ra ≤ 0.05 μm; and the other group was ground mechanically off about 15 μm under a gentle load with water cooling as the compound layer was about 15 μm in thickness, and then polished an average surface roughness Ra ≤ 0.05 μm. Follow-up a superlattice AlCrTiSiN coating was deposited on the surface of these experimental samples by multi-arc ion plating.

Table 1
Description of the labels.

Name	Nitrocarburizing	Grinding	Multi-arc ion plating
PNC	+	−	−
NC-CrN/AlCrTiSiN	+	+	+
CrN/AlCrTiSiN	+	−	+

One source fitted with cathode Cr target (99.9% purity) was installed on one side of the chamber wall, and the second AlCr (70 at% Al and 30 at% Cr, 99.9% purity) cathode and the third CrTiSi cathode (60 at% Cr, 30 at% Ti and 10 at% Si, 99.9% purity) were symmetrically installed on the opposite side of the barrel-shaped vacuum chamber wall. Follow-up these samples

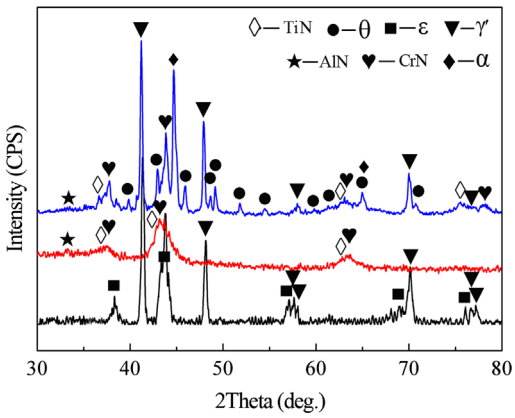

Fig. 1. XRD patterns showing the constitutional phases in each of the samples with different treatment processes. Note that the PNC and the CrN/AlCrTiSiN samples correspond to the high angle XRD, and the NC-CrN/AlCrTiSiN sample corresponds to the grazing incidence XRD.

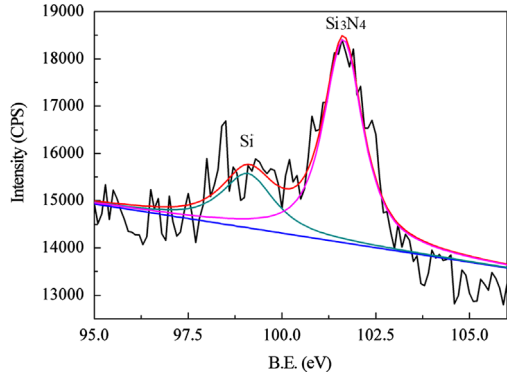

Fig. 2. Fitting spectrum by XPS for Si of a NC-CrN/AlCrTiSiN sample.

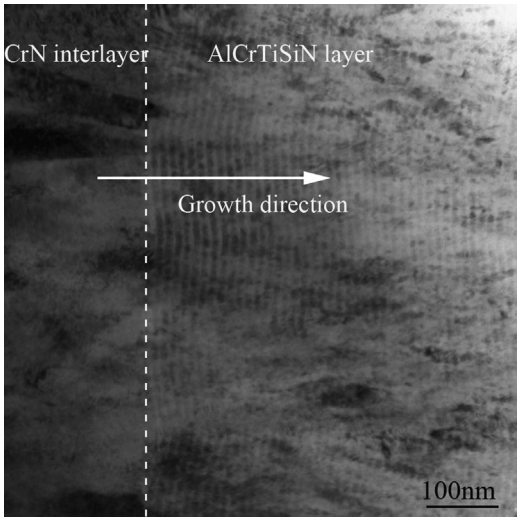

Fig. 3. Typical cross-sectional TEM image of a CrN/AlCrTiSiN sample.