Mechanical, Structural and Tribological Properties of TiAlZrN Coated AISI H13 Steel

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Abstract

Wear behavior of AISI H13 steel for service life is not adequate for many applications in the manufacture industry. Heat treatment such as conventional and nitrating is commonly performed to enhance service life of it. In last decades, some coatings technique including PVD methods were used to improve wear resistance of this steel. In this study, TiAlZrN coating deposited on the surface of AISI H13 steel using Pulsed DC closed field unbalanced magnetron sputtering method. Structural, morphological properties of coated samples were examined with scanning electron microscope. Wear tests were performed with using ball on disk type tribometer at room temperature. In addition, adhesion strength of coatings was evaluated with using scratch tester. The wear test results showed that TiAlZrN coating increases the wear resistance of H13 steel 20 times. The scratch test results showed that the highest adhesion strength was attained as 56 N at 5 A Zr target content, 50 V bias voltages and 2x10^-3 Torr working pressure deposition parameters. Worn surfaces were characterized with SEM.

1. Introduction

Hot work steels are widely used in the manufacture industry for many applications such as casting dies, extrusion tools, and steel forging. Among the hot work tool steels, AISI H13 is one of the favored die materials mostly used in aluminum extrusion mold industry owing to its remarkable properties such as good resistance to abrasion at both low and high temperatures, high level of toughness and ductility, uniform and high level of machinability, good high-temperature strength and resistance to thermal fatigue, excellent through-hardening properties [1]. Besides, extrusion molds have technological and economical importance for its service life, since it affects surface quality of product and increases cost of production [2]. However, service life of AISI H13 steel is not adequate for many industrial applications. Therefore, heat treatment such as conventional and nitrating have been performed to improve surface quality and increase service life of this molds made of AISI H13 steel. Currently, application of coatings using PVD methods on the surface of AISI H13 steels another method to enhance wear life of this steel [3]. Among the different PVD methods, Pulsed DC closed field unbalanced magnetron sputtering method is one of the most successful and sophisticated technology for the metal, alloy and ceramic coatings to control microstructure, surface morphology and phase composition. In particular, transition-metal nitrides have been successfully used as wear resistant coatings in many industrial applications [4]. TiN is one of the most well known and has been studied extensively [5]. However, its properties are not enough for some applications. For this reason ternary coatings such as; TiAlN, TiCrN, TiNbN were developed [6]. In these studies, it was observed that addition of Al improves thermal stability, hardness and wear resistance of TiN coating [7]. In addition, different chemical elements such as Nb, Zr, Cr, and Si were added to TiAlN coatings for enhancing the mechanical and wear properties of TiAlN coatings [8,9]. Addition of Zr element in TiAlN coatings also showed remarkable resistance against the oxidation and improved the mechanical and thermal properties of coating [10]. On the other hand, wear properties and scratch resistance of TiAlZrN coating was not investigated adequately.

Aim of this study, structural, mechanical and tribological properties of TiAlZrN coatings was investigated. Pulsed DC closed field unbalance magnetron sputtering technique was used for deposition of coatings.

2. Experimental Procedure

2.1. Deposition Process

In this study, TiAlZrN-graded composite coatings were deposited by pulse DC closed field unbalanced magnetron sputtering. Ti, Al, Zr elemental targets were used for deposition. Hardened (550 HV 0.2) AISI H13 steels were used for substrate material. Constant and variable parameters are given in Table 1.
Table 1. Deposition Parameters

<table>
<thead>
<tr>
<th>Constant Parameters</th>
<th></th>
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<tbody>
<tr>
<td>Target Currents: Ti/Al (A)</td>
<td>6/2</td>
</tr>
<tr>
<td>Interlayer Target Current Ti (A)</td>
<td>6</td>
</tr>
<tr>
<td>Deposition Duration (min.)</td>
<td>60</td>
</tr>
<tr>
<td>Duty Time (μs)</td>
<td>2.5</td>
</tr>
<tr>
<td>Working Pressure (Torr)</td>
<td>2.5x10^{-3}</td>
</tr>
<tr>
<td>Substrate Bias Voltage (-V)</td>
<td>90</td>
</tr>
</tbody>
</table>

Before deposition, the substrate surfaces were sputter ion cleaned for 10 minutes. In order to increase the adhesion of coatings, the depositions started with Ti under layer (5 minutes), which were followed by TiN layer (15 minutes). After this process, TiAlN/TiAlZrN for each layer was deposited for 20 minutes.

2.2. Characterization of Coatings

The surface morphology and thickness of the coatings were characterized using scanning electron microscope (SEM). Micro hardness values were measured by Struers Vickers micro hardness tester at 245.3 mN constant load. Five micro hardness measurements were performed for each sample to obtain the average values of hardness. Adhesion of the TiAlZrN-graded composite films was evaluated by scratch tester-CSM Revester equipped with a Rockwell C diamond stylus. Worn surfaces were characterized with scanning electron microscopy. The wear experiments were conducted using ball-on-disk tribometer. All experimental parameters were kept constant as follow; Al₂O₃ counter body, test period 1600 cycles, normal load 2N and sliding speed 60 mm/s. Wear volumes were measured with optical profilometer.

3. Result and Discussions

Thickness, scratch resistance and micro hardness and wear rate values of the coatings deposited with different deposition parameters are presented in Table 2.

Table 2. Thickness, hardness values, scratch values, wear rate and chemical composition of coatings

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Thickness (μm)</th>
<th>Hardness (HV)</th>
<th>Scratch Lc₂ (N)</th>
<th>Wear Rate</th>
<th>Chemical composition (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Ti</td>
<td>Al</td>
</tr>
<tr>
<td>R1</td>
<td>2.1</td>
<td>806</td>
<td>51</td>
<td>5.65 E-05</td>
<td>40.56</td>
</tr>
<tr>
<td>R2</td>
<td>2.4</td>
<td>915</td>
<td>48</td>
<td>4.59 E-05</td>
<td>43.24</td>
</tr>
<tr>
<td>R3</td>
<td>2.5</td>
<td>1005</td>
<td>56</td>
<td>2.42 E-06</td>
<td>45.00</td>
</tr>
</tbody>
</table>

Surface morphology of the TiZrN coating investigated by the SEM is shown in Figure 2.

![Figure. The surface morphology of coatings a) R1 b) R2 c) R3](image-url)
As shown in Figure 2a, the grain size of R1 coating is relatively bigger than the others. This can be related with the lower mobility of the adatoms (absorptive atoms) at low target current during deposition process. In the same figure, it can be seen that coatings show finer and more compact structure with increasing Zr target current from 2 A to 5 A. As the Zr target current increases, the grain sizes become smaller due to the enhanced energetic particles bombardment.

![Image](image1.png)

**Figure 3. Coating layer thickness measurements a) R1  b) R2  c)R3**

Thicknesses of the TiAlZrN coatings were determined using SEM photos of the crosssections obtained by brittle fracture in the radial direction of the films deposited on the glass substrates. It can be seen in Figure 3 that the coatings exhibit a dense and thin columnar structure. It was also observed that increasing Zr target current from 2 A to 5A decreases the column sizes of coatings relatively. This is related with the higher ion bombardments at higher target currents. Lv et al indicated that the high energy ion bombardment to coatings increase the number of preferential nucleation sites and prevent the migration of grain boundaries and leading to a decrease in grain size[11]. Attained result can be explained with this interpretation.

Micro hardness values of the coatings are given in Table 2. As shown in Table 2, the Zr current value increase in the R1, R2 and R3 samples hardness values are increased. This increase is due to the increase of the the Zr current, by the enhancement of the solid solution strengthening effect on the coating. This was similarly reported by Chen and colleagues as increasing the hardness values of the TiAlZrN coatings by increasing Zr current [12]. Scratch values of the coatings are shown in Table 2. The highest scratch resistance was attained from R3 deposition conditions. It is interesting to note that there is relation between Al amount and scratch resistance. The sample which has lowest amount of Al shows highest scratch resistance, the sample which has highest amount of Al shows lowest scratch resistance. This can be related with the soft structure of aluminium. Besides, Donohue et al. indicated that increasing Zr current during deposition process results smoother and denser microstructure and increase hardness and scratch resistance [13]. Increasing scratch resistance may be related this fact.

The scratch mechanisms of samples were characterized using scanning electron microscopy. During SEM inspection of scratch tracks, cohesive (conformal cracking) failure mode was observed for R1, chipping failure mode was observed for R2, chipping and adhesive failure mode was observed for R3. Also, recovery spallation failure mode was observed for all films as shown in Figure 4.

![Image](image2.png)

**Figure 4. Scratch pattern of TiAlZrN coating at Lc2**

The friction coefficients of coatings are given in Figure 5. According to Figure 5, it can be seen that R3 shows stable friction coefficient curve. However R1 and R2 shows former coating removed from the substrate about 800 cycles, later coating removed from the substrate 1200 cycles.
Figure 5. The friction coefficient of coatings

Wear rate of coatings are given in Table 4. From this table, it can be seen that R3 shows the highest wear resistant than the other coatings. This is related with its highest hardness and scratch resistance properties. Besides, TiAlZrN coating increases the wear resistance of H13 steel 20 times.

4. Conclusion

The TiAlZrN-graded coatings were successfully deposited on AISI H13 steel substrates by pulsed dc close field unbalanced magnetron sputtering system with the different deposition parameters. The surface morphology, chemical composition, wear and properties of coatings were analyzed. Following conclusions were obtained:

- All coatings show smooth and columnar structure.
- Increasing Zr target current decreases the grain size
- The highest hardness value was obtained at R3 deposition parameters with highest Zr current, the lowest hardness value was obtained at R1 deposition parameters with lowest Zr current.
- There is relation between scratch resistance and Al amount. The highest scratch resistance was obtained at R3 (smallest amount of Al), the lowest scratch resistance was obtained at R2 (highest amount of Al)
- The highest wear resistance was attained with highest Zr target current
- TiAlZrN coating increases the wear resistance of H13 steel 20 times.

Acknowledgment

This study was supported by the Scientific and Technological Research Council of Turkey. Grant No: 116M734

References