



## Surface Characterization of Duplex Surface Treatments: AISI H13 Tool Steel

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This research provides an extensive analysis of duplex-treated AISI H13 tool steel after gas nitriding and coating application with Titanium Carbide (TiC) combined with Chromium Nitride (CrN) and Aluminum Titanium Nitride (AlTiN). The popular tooling material of AISI H13 received 24-hour gas nitriding at 400 °C to form its nitrogen-enriched outer layer. Each coating received equivalent deposition parameters after the base conditions for maintenance of consistent outcomes between specimens. The SEM analysis together with EDS and XRD methods was used for investigating phase transformations and elemental dispersal patterns in the treated coatings. The mechanical property analysis used Vickers microhardness testing methodology. A hardness measurement of 242 HV was recorded for the untreated material but the amount increased to 1062 HV after the nitriding process. Among the different coatings AlTiN achieved the greatest surface hardness level at 2811 HV while TiC reached 2717 HV and CrN settled at 2105 HV. The study confirms that duplex surface treatment enhances H13 tool steel durability and its resistance to wear by showing AlTiN as the superior coating for demanding operating conditions with high heat and load requirements.

**Keywords:** AISI H13 Tool Steel, Duplex Surface Treatment, X-ray Diffraction (XRD), Energy-Dispersive X-ray Spectroscopy (EDS), Scanning Electron Microscopy (SEM), Microhardness

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## 1. Introduction

Tool steels primarily use AISI H13 for high-temperature industrial applications including die casting along with extrusion and hot forging because of their superior toughness and thermal fatigue resistance as well as their exceptional high-temperature strength [1]. AISI H13 shows resistance to high thermal and mechanical stresses because of these properties which make it suitable for tool applications under demanding conditions. Under extended use in harsh conditions the steel encounters important deterioration pathways defined by wear oxidation and thermal fatigue which limit its operational capability and shortens its operational duration while elevating maintenance needs and operational stoppages [2]. Advanced surface engineering techniques now focus on duplex surface treatments as a highly effective method to extend operational durability of AISI H13. Tool steel surface enhancement through duplex treatments employs gas nitriding procedures alongside PVD hard coatings to add strength features on top layers without changing bulk material properties [3]. Through gas nitriding steel receives both a compound layer (5–10  $\mu\text{m}$  thick) and a diffusion layer (100–150  $\mu\text{m}$ ) which collectively enhance bearing loads and surface hardness and decrease deformation [4-11]. The combination of nitriding with PVD coated overlays exhibits superior performance compared to stand-alone nitriding because it proves effective at resistance against wear while working under high temperatures and demanding frictional forces. Applications benefit from this collaborative process which produces an outstanding surface with remarkable hardness in combination with stronger bonding and improved tribological qualities [12-17].

## 2. Materials and Methods

### 2.1 Substrate Material Preparation

The research utilized AISI H13 tool steel as its base material because of its widespread tooling applications due to its outstanding properties which combine toughness with hardness and thermal fatigue resistance. The AISI H13 steel material received commercially led to the production of rectangular test samples with dimensions of 20 mm  $\times$  20 mm  $\times$  5 mm. The specimens received primary surface preparation by grinding with SiC papers

then subsequent polishing with alumina suspension for achieving a high-quality reflective surface finish before receiving further surface treatments.

### 2.2 Gas Nitriding Process

Each sample received gas nitriding treatment which created a hardened diffusion layer at its surface. Atmospheric controls surrounded the nitriding procedure that lasted 24 hours at 400 °C. Gas nitriding allowed the development of an iron nitrides rich compound layer together with a diffusion zone beneath the surface. The nitriding parameters remained identical for the entirety of the laboratory specimens to create uniform baseline properties before the coating implementation.

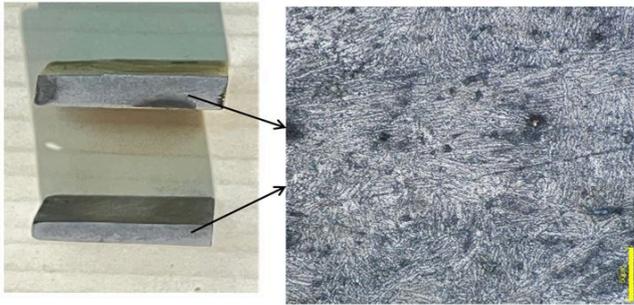
### 2.3 Coating Deposition

Three distinct hard coat materials Titanium Carbide (TiC) and Chromium Nitride (CrN) and Aluminum Titanium Nitride (AlTiN) coating on nitrided samples. The deposition process took place under uniform operational settings for maintaining equivalent treatment quality across all coatings. Each material received exclusive consideration because it provided distinct strengths for hardness, oxidation prevention and working at elevated temperatures.

## 3. Surface Characterization

### 3.1 Analysis of Core Microstructure in AISI H13 Tool Steel

The core area of AISI H13 tool steel shows a regular tempered martensitic lath structure according to Figure 2. During the quenching and tempering process AISI H13 tool steel develops this particular structure that creates its exceptional strength and toughness properties. The matrix contains uniformly spread fine elongated features which form martensitic laths. A combination of hardness and ductility results from the steel's morphology which allows H13 tool steel to meet requirements of hot forging and die casting and extrusion processes. Martensite distribution throughout the tool section generates uniform mechanical performance because of its uniform distribution pattern. The tool steel core provides an excellent basis for further treatments including gas nitriding and hard coatings through its stable foundation with sufficient strength and thermal management capabilities.



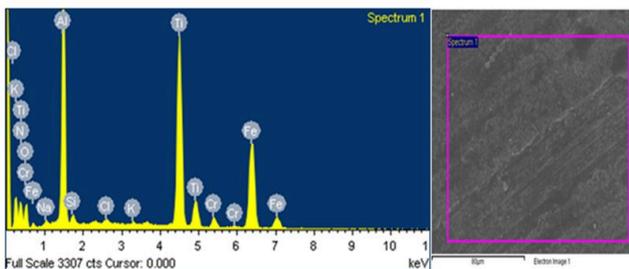
**Figure 1:** SEM micrograph showing martensitic laths in AISI H13 steel.

### 3.2 Energy-Dispersive X-ray Spectroscopy (EDAX)

EDS operates as a microanalytical instrument for analyzing material elemental composition. The sample produces characteristic X-ray emissions when it receives collisions from high-energy beams that typically employ electrons in SEM. The X-rays produce peaks on a spectrum through data capture procedures.

#### a. EDS Analysis of Aluminum Titanium Nitride (AlTiN) Coating

The EDS analysis of the AlTiN-coated sample performed a sufficient initial identification by revealing Al (18.04 wt%) and Ti (27.87 wt%) and N (10.60 wt%) while showing an irregular composition ratio and poor nitrogen content. Drive for substrate material transport and surface oxidation exist in coatings although they demonstrate that there are opportunities to advance the coating performance. Minor impurities contained within the trace elements meet acceptance limits. The recommended improvements for-aligning the analysis with standard AlTiN compositions includes re-testing the coating layer and optimizing nitrogen precision and lowering the oxidation rate.



**Figure 2:** EDS Analysis of AlTiN

#### Primary Coating Elements

- Aluminum (Al): 18.04 wt% (19.48 at %) The wt% of Al is lower than 30–50 wt% which means that it might be a Ti-rich composition

- or a diluted substrate. This may reduce oxidation resistance.
- Titanium (Ti): 27.87 wt% (16.95 at%) Within the expected range (20–40 wt%). The Al:Ti ratio (~0.65:1) is substantially lesser than typical (1:1 or 2:1) and consequently, points towards nonstandard deposition suitable for hardness.
- Nitrogen (N): 60 wt% (22.04 at%) Generally, the content is between 15-25 wt%; however, oxygen interference or deposition loss may have occurred affecting nitride stability.

#### Oxygen and Contaminants

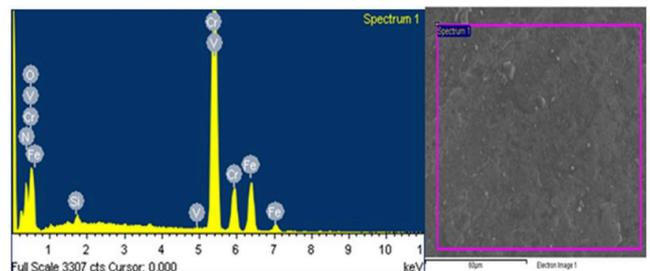
- Oxygen (O): 13.62 wt% (24.80 at%) Surface oxidation or troublesome nitrogen detection along with air exposure after the coating application explains the high oxygen content.
- Na, Si, Cl, K – All <1 wt% : The detected minor contaminants stem from reference standard and sample handling procedures. The presence of these elements at such low levels does not impact coating performance

#### Substrate Influence

- Iron (Fe): 25.64 wt% (13.38 at%): High Fe indicates penetration of the steel substrate, due to thin coating (2–3 μm) or partial diffusion.
- Chromium (Cr): – 2.14 wt% (1.20 at%)

The EDS analysis of the AlTiN-coated sample performed a sufficient initial identification by revealing Al (18.04 wt%) and Ti (27.87 wt%) and N (10.60 wt%) while showing an irregular composition ratio and poor nitrogen content. Minor impurities contained within the trace elements meet acceptance limits. The recommended improvements for-aligning the analysis with standard AlTiN compositions includes re-testing the coating layer and optimizing nitrogen precision and lowering the oxidation rate.

#### b. EDS Analysis of Chromium Nitride (CrN) Coating



**Figure 3:** EDS Analysis of CrN

**Primary Coating Elements**

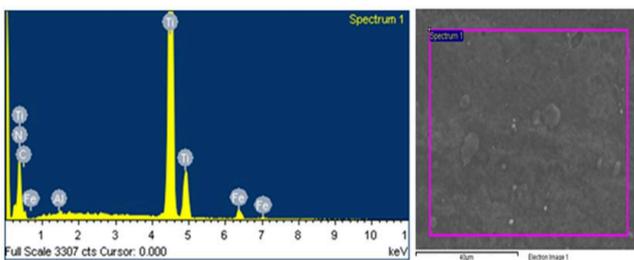
- Chromium (Cr): 64.13 wt% (42.04 at%) Cr represents the major chemical component of the coating surface just as predicted for chromium nitride coatings. Such high chromium content proves the formation of a strong chromium-rich nitride layer that provides essential properties for hardness and resistance against wear.
- Nitrogen (N): 14.53 wt% (35.35 at%) The elemental composition contains substantial N corresponding to standard CrN coatings that amount to 10–20 weight percent N. The significant amount of atomic percentage indicates the powerful Cr–N bonding which enables the formation of hard CrN phase.
- Oxygen and Minor Elements: Oxygen (O): 6.03 wt% (12.85 at%) These levels of oxygen indicate surface oxidation which occurs as CrN interacts with air elements. The coating composition contains low enough levels of nitrogen that it should not affect the nitride stability negatively.
- Silicon (Si): 0.65 wt% (0.79 at%) Likely a contaminant or from substrate/pre-treatment. Its low level poses no issue. Vanadium (V) – 0.39 wt% (0.26 at%) Trace amount, possibly from substrate or tool steel alloy. Not a typical CrN component.

**Substrate Signal**

- Iron (Fe): 14.27 wt% (8.71 at%) Fe indicates that both substrate interferences and diffusion occur possibly because of coating thinness or high beam penetration depth.

Overall CrN coating exists with Cr as the major component and high nitrogen content. The observed levels of oxygen together with iron suggest simultaneous surface oxidation and a role of the substrate material in the analysis results.

**c. EDS Analysis of Titanium Carbide (TiC) Coating**



**Figure 4:** EDS Analysis of TiC

**Primary Coating Elements**

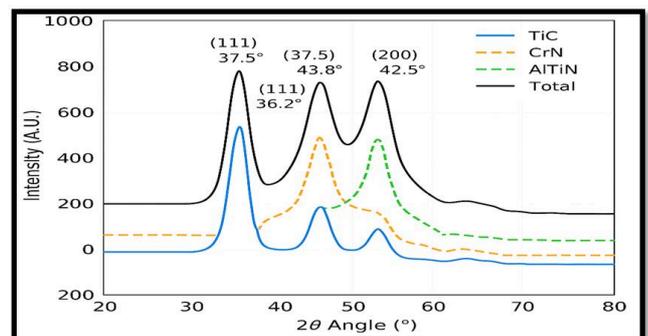
- Titanium (Ti): 69.51 wt% (43.14 at%) The main component Ti emerges in the analysis which verifies the Ti-based carbide coating structure. The high titanium content produces a coating that shows standard characteristics such as enhanced mechanical strength and high resistance to wear which are typical of TiC.
- Carbon (C): 8.68 wt% (16.14 at%) C is a key component in TiC. The ratio of atoms supports the necessary Ti–C bond for creating the required carbide structure. While below the perfect ratio the composition matches conventional TiC compositions.
- Nitrogen (N): 18.18 wt% (38.58 at%) High N content in the coating signifies the formation of Ti(C, N) composition which provides good toughness and oxidation protection.

**Minor Elements and Substrate Signal:**

- Aluminum (Al) – 0.37 wt% (0.40 at%) : The analysis reveals tiny N concentrations which most likely result from previous layers and surface residue. Its level is negligible.
- Iron (Fe) – 3.26 wt% (1.74 at%) : The XPS spectrum shows evidence of steel substrate material thus implying a weak coating layer or steel metal penetration during production.

The coating mainly consists of TiC accompanied by noticeable amounts of nitrogen which points to its composition as Ti(C,N). The dual-phase structure meets two purposes by creating a stronger and more heat-resistant material. Small concentrations of Fe and Al in the coating analysis highlight substrate interactions and surface impurities that do not affect its main chemical composition. The protective layers demonstrate functional durability which makes demanding working conditions.

**3.3 X-ray diffraction (XRD) Analysis**



**Figure 5:** XRD pattern for the TiC, CrN, and AlTiN coatings

X-ray diffraction (XRD) utilizes the diffraction of X-rays by a crystal lattice to analyze its structure. The incident X-rays interact with the atoms in the crystal, causing them to scatter and interfere, forming a diffraction pattern. This pattern reveals information about the material's crystalline structure, including phase identification, crystallite size, and lattice strain.

**Phase-Specific Analysis**

- The XRD pattern for TiC exhibits distinct peaks at 36.2° (111) and 42.5° (200) which confirms the presence of cubic TiC phase.
- The XRD pattern CrN: shows two peaks at 37.5° for the (111) plane and 43.8° for the (200) plane which confirm the cubic structure of CrN.
- The XRD pattern of AlTiN: appear in a range that overlaps with TiC and CrN because these crystal structures show similar characteristics during X-ray analysis within 36°–44°.

The nearby TiC(111) and CrN(111) peaks near 36.2° and 37.5° create an overlap that causes the pattern to become broadened or shows shoulder features.

The sample contains higher amounts of specific phases when their peaks appear stronger during analysis. The 36.2° dominant peak indicates an important amount of TiC in the sample.

The total peak absence of TiC(220) could stem from preferred orientation combined with low crystallinity together with data collection limitations.

Small peak position differences compared to standard values can originate from three main factors which include residual stress along with compositional variations and instrumental factors.

**4. Microhardness Values of AISI H13 Tool Steel and Surface Treatments**

A Vickers microhardness tester evaluated microhardness parameters across the cross-sections of prepared samples. Actual force was set to 100 grams while the sample received 10 seconds of dwells. A regular indentation protocol across the coated and nitrided layers enabled the creation of hardness data from the surface to the interior. Average surface hardness values allowed for,

evaluation of mechanical enhancement across coated samples through calculation and comparison of test data.

**Table 1: Micro hardness Values**

Surface Treatment	Microhardness Range (HV)
Base Material (H13)	242
Nitrided H13	1062
CrN-Coated H13	2105
AlTiN-Coated H13	2811
TiC-Coated H13	2717

- Base Material (H13) – 242 HV: The untreated AISI H13 tool steel showed 242 HV as its microhardness value. The initial hardness of 242 HV indicates the presence of normally tempered martensitic microstructure that H13 steel develops through standard heat treatment procedures. The material structure gives good toughness and thermal fatigue resistance yet its low surface hardness reduces performance in rigorous and hot operating conditions. Further surface treatment stands necessary to boost tribological performance for die casting and hot forging operations because of the low original surface hardness of AISI H13 tool steel.
- Nitrided H13 – 1062 HV: Gas nitriding resulted in AISI H13 steel achieving 1062 HV surface hardness. The Surface compound layer together with the nitrogen-enriched diffusion zone near the surface contributes to this increased hardness because they both contain iron nitrides ( $\gamma'$ -Fe<sub>4</sub>N and  $\epsilon$ -Fe<sub>2-3</sub>N). The material maintains its mechanical core strength and exhibits enhanced bearing loads as well as superior resistance to wear after nitriding. Surface engineering starting treatments benefit strongly from nitriding because it creates a solid base especially when used in duplex procedures.
- CrN-Coated H13 – 2105 HV: The PVD application of Chromium Nitride (CrN) coating onto AISI H13 produced surface material with a microhardness value of 2105 HV. CrN phase delivers the significant microhardness boost due to its natural hardness and its superior resistance toward wear along with corrosion and high-temperature oxidation. The adhesion properties between CrN coatings and the nitrided substrate help increase performance under mechanical forces as well as thermal stress.

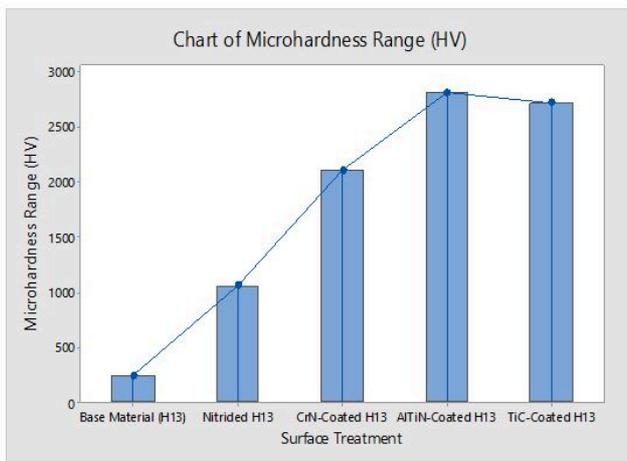
The treatment provides excellent benefits for tooling applications that need a combination of hardness and wear protection alongside mechanical toughness.

- **AlTiN-Coated H13 – 2811 HV:** Surface hardness of AlTiN-coated H13 specimens reached 2811 HV which made them the most durable among all tested treatment groups. Aluminum Titanium Nitride (AlTiN) coatings yield superior thermal stability along with excellent chemical resistance because addition of aluminum and titanium nitride phases creates synergistic effects. The implementation of the AlTiN layer provides optimal protection during applications at elevated speeds and elevated temperatures for cutting tools and hot forming procedures. Advanced multi-element nitride coatings demonstrate excellent performance results when combined with nitriding procedures.
- **TiC-Coated H13 – 2717 HV:** The microhardness achieved by Titanium Carbide (TiC) coated H13 samples reached 2717 HV making them perform similarly to AlTiN. The outstanding properties of TiC result from its covalent bond network in the carbide which produces both exceptional hardness alongside outstanding abrasion wear resistance. This coating shows excellent thermal resistance which enables its usage in heavy-duty applications that involve high friction. While AlTiN is marginally harder than TiC it establishes itself as a strong coating solution for machinery used in demanding mechanical wear environments.

Through surface treatment applications AISI H13 tool steel achieves an improved microhardness property which makes it appropriate for demanding industrial uses. The AlTiN coating stands out for having the most strenuous surface hardness among all investigated treatments because of its effectiveness for rigorous tribological situations demanding maximal temperature resistance and minimum wear levels. Both TiC and CrN coatings delivered exceptional results because they consolidated desirable traits such as surface hardness with material toughness and resistance to wear and corrosion. The single process of gas nitriding produces better surface hardness than non-coated applications through the formation of a hard nitride layer which provides cost-efficient solutions for various industrial requirements. The experimental data demonstrates how duplex surface treatment particularly when using nitriding together with high-performance coatings significantly lengthens the operational lifespan of H13 tool steel components.

## 5. Conclusions

- Surface property improvement of AISI H13 tool steel becomes possible when these materials undergo duplex treatment involving gas nitriding followed by hard coating deposition. All coating samples deliver much higher microhardness compared to untreated or nitrided AISI H13 tool steel. The AlTiN coating demonstrates superior surface hardness of 2811 HV which qualifies it as the best choice to endure harsh tribological environments.
- The coatings TiC and CrN showed combined advantages of 2717 HV and 2105 HV hardness thus providing an ideal balance of wear resistance and material toughness. Gas nitriding by itself produced 1062 HV surface hardness through the formation of tough nitride layers that enhance steel load capacity.
- The XRD and EDS and SEM results verified the proper development and structural characteristics of all coatings. Using gas nitriding together with high-hardness coatings proves to be an efficient technique that boosts H13 tool steel surface durability and operational reliability in severe operational settings.



**Figure 6:** micro hardness Comparisons

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