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INFLUENCE OF THE NITRIDING AND TiAIN/TiN COATING THICKNESS IN THE MECHANICAL PROPERTIES OF A DUPLEX TREATED H13 STEEL[1]

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Abstract

AISI H13 die steel substrates were low pressure gas nitrided in three different nitriding cases. In the nitriding case A, the surface hardness was around 12 GPa and the nitriding thickness was around 40 µm. In the nitriding case B, the hardness was the same as in case A, but the nitriding thickness was around 70 µm. Finally, in the nitriding case C, the nitriding thickness was the same as in case B, but hardness profile showed a different behavior. In case C, the surface hardness was the same as case A and B. But the hardness increases as one move away from the surface showing the highest hardness at 15 µm from the sample surface. The XRD results showed that the nitriding cases microstructure is composed mainly by the diffusion layer with small amount of Cr_2N precipitates. These nitrided samples were subsequently coated with TiAlN using cathodic arc evaporation in two thicknesses of 3 and 7 µm. These samples were characterized with respect to phase chemistry, adhesion, hardness, elastic modulus and scratch tests. The phase chemistry determined through XRD revealed that coating was mostly $Ti_{0.7}Al_{0.3}N$ with some peaks of TiN which comes from the adhesion layer that was deposited prior to the deposition of TiAlN. The instrumented hardness performed in the coated samples showed that the coating system hardness changes with the nitriding cases when the coating thickness is 3 µm. On the other hand, the nitriding characteristics do not influence the coating hardness with thickness of 7 µm. In addition, the 7 µm thick coating is harder than the 3 µm thick coating. In the last part of this work, TiAlN was deposited in the AISI H13 substrate without nitriding; it was found that the hardness in this condition is higher than the nitrided/coated samples. The worn area, probed by the scratch test, was smaller for the TiAlN deposited over AISI H13 without the nitriding layer.

Key words: Duplex Treatment; TiAlN/TiN; Mechanical Properties; PVD.

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1 INTRODUCTION

The duplex treatment has been widely applied in die casting, cutting and forming tools. It consists of a nitriding process followed by a coating deposition. A wide number of coatings systems such as TiN, TiAlN, CrAlN and CrN have been applied on tool steel such as AISI H13, AISI D2 and low to medium carbon steels.[1-4] The most important function of the nitriding layer is to improve the coating load bearing capacity through the formation of a subsurface diffusion zone, while the coating serves to reduce abrasive and corrosion wear.[5]

It is known that a nitrided intermediate layer diminishes the properties mismatch between the coating and the steel substrate, improving the coating adhesion.[6] However, this is achieved only when the nitriding process provides hardening cases without the compound or white layer. The presence of the compound layer in the nitriding cases decreases the coating adhesion because the compound layer is thermally unstable, so it tends to decompose during the coating deposition process.[7] There are basically three industrial nitriding technologies that are currently applied: gas nitride which uses ammonia (NH_3) as a nitrogen source and conveyor; low pressure or vacuum nitriding that uses N_2O and NH_3; and plasma or ion nitriding that works with a mixture of N_2 and H_2. In the last two nitriding techniques the formation of a compound layer can be easier controlled or even avoided.

Concerning the duplex treatment, Huang et al.[8] studied the effect of the nitriding cases on the coating properties, and found that the hardness of TiN coating on plasma nitrided AISI M12 steel was increased due to the improvement on the load carrying capacity of the duplex treated surface. Similar results were found by Ma et al.,[9] where the hardness of TiN presented higher values when deposited over a nitrided surface than over AISI H13 steel without nitriding treatment. On the other hand, Ding et al.[10] found that the substrate hardness does not affect the coating hardness, friction coefficient and wear rate, but it does affect the coating/substrate adhesion. As one can realize it is not very clear if a nitriding treatment increases the coating mechanical properties or simply improves coating/substrate adhesion. In order to clarify the above mentioned issue, it was investigated the mechanical and wear properties of the TiAlN coating with two different thicknesses deposited on three different nitrided cases, and compared with the coating deposited directly on AISI H13 substrate.

2 EXPERIMENTAL DETAILS

AISI H13 substrate samples were quenched and tempered to a hardness of 47 HRC. Prior to nitriding treatment, the samples were ground and polished to a surface finish of Ra = 0.05 µm. The nitriding process was carried out in an industrial unit using a low pressure gas nitriding process. The nitriding process atmosphere consisted of nitrogen and ammonia mixture. The nitriding temperature was fixed at 520°C while the nitriding time varied from 5 to 9 h, to obtain three different nitrided cases.

Prior to the deposition of the coating system, the samples were again polished to a surface finish of Ra = 0.05 µm. The coating system that was deposited over the AISI H13 steel was composed of TiN, where the main purpose was to act as an adhesion layer, and a TiAlN outer layer, which is the working layer. The coating system was deposited by employing a PVD cathodic arc process, according to the Oerlikon

Balzers Balinit Futura process. The TiN/TiAlN coating system was deposited on nitrided and non-nitrided AISI H13 specimens in two different total thicknesses: 3 and 7 µm. In the TiN/TiAlN with 3 µm of total thickness, the TiN thickness is around 0.3 µm while in the TiN/TiAlN with 7 µm total thickness, the TiN thickness is around 0.7 µm.

The nitriding cases and the TiN/TiAlN coating system were characterized in terms of phases chemistry by X-ray diffraction (XRD) using a Shimadzu XRD-7000 diffractometer in a θ – 2θ scan mode. It was used CuKα radiation (40 kV, 20 mA) with continuous scan and scan speed of 1 degree/min.

The nitriding cases hardness profiles were obtained by performing indentation using a Berkovich diamond tip with a load of 50 mN spaced by 5 µm. These profiles were used to determine the depth of the nitrided case and nitrided surface hardness.

The mechanical properties of the coating system were investigated by means of instrumented indentation measurements (Nanoindenter XP, MTS) using a Berkovich diamond tip. A set of sixteen indentations with ten loading/unloading cycles was made on each sample surface. The maximum applied load was 400 mN, and the holding time at each load step was 15 s. The hardness and elastic modulus profiles were determined using the Oliver and Pharr method.[11] The Berkovich indenter was calibrated using fused silica following the procedure described in.[11]

Nanoscratch tests were made with a Berkovich diamond tip following the tip edge direction in ramping loads from 0 to 400 mN. The length of nanoscratch was 1 mm. The tip penetration profiles were monitored before, during and after the loading. A profile of the scratch groove was obtained in order to compare the worn area for each condition of nitriding cases/coating thickness.

The adhesion quality of the TiAlN/TiN coating system on H13 steel was evaluated using a Rockwell C Indenter (Wilson-Rockwell) by comparing the indentation pattern with HF patterns.[12]

3 RESULTS AND DISCUSSION

3.1 Nitrided Cases Characterization

Three different nitrided cases were obtained in the low pressure gas nitriding process. Figure 1 shows the X-ray diffraction patterns for the three nitriding cases. The peaks were identified mainly as α-Fe phase. Smaller peaks showed in detail were indexed as Cr_2N, suggesting that the nitrided cases have no compound layer, that is, nitrogen atoms did not form iron nitrides and are located in interstitial sites in the α-Fe lattice and dissolved as a diffusion layer. Moreover, the XRD patterns show that the phases formed in the three nitrided cases are identical.

The instrumented indentation hardness (H_{IT}) profiles of the three nitrided cases are shown in Figure 2. The nitrided cases thickness was defined as the substrate hardness plus 1 GPa. By this criterion the nitrided case A has a thickness of about 40 µm while the cases B and C have a thickness of about 70 µm. Moreover, Figure 2 also shows two hardness profile behaviors. In cases A and B the hardness decreases with the surface distance while in case C, the highest hardness peak is observed at approximately 15 µm from the surface.

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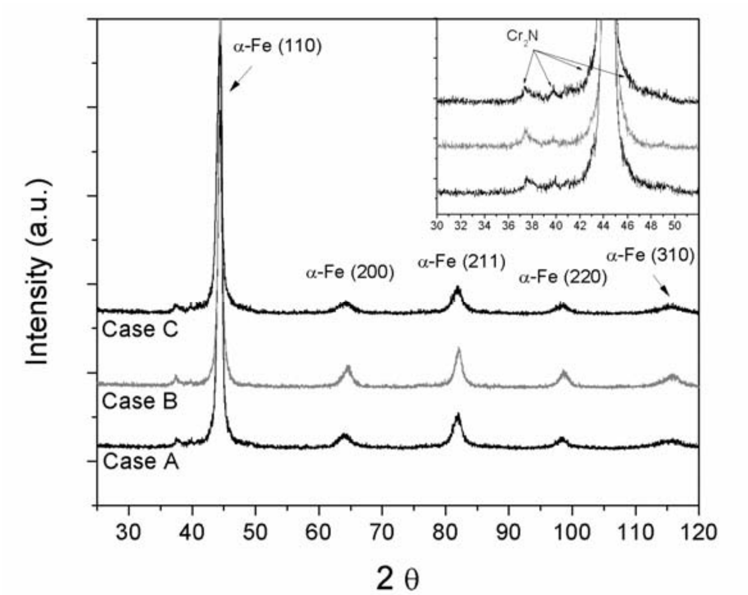

Figure 1. The XRD patterns of the nitrided cases.

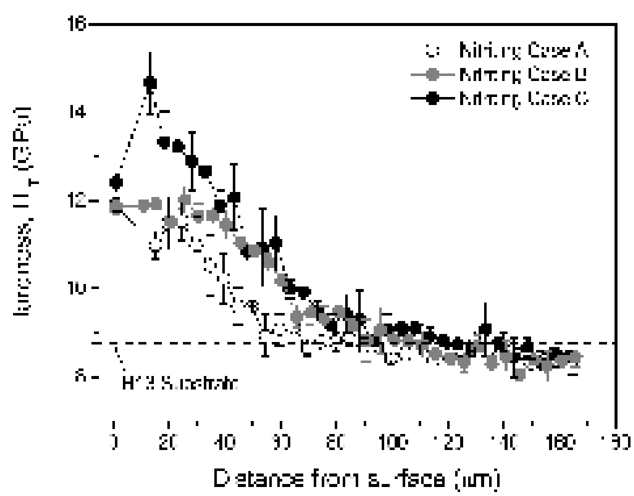

Figure 2. Hardness profile of the nitrided cases.

3.2 Coating System Phases

Figures 3 and 4 show the XRD patterns for the coating system with thickness of 3 and 7 μm deposited over the nitrided cases A, B and C. These XRD patterns reveal that the intermediate layer is indeed TiN and the working layer is TiAlN. Moreover, Ti/Al atomic ratio in the TiAlN layer is about 2.4. By examining Figures 3 and 4 one can realize that the coating thickness and nitrided cases did not alter the coating system stoichiometry, the phases formed in both coating system thickness over the three nitrided cases are practically the same.

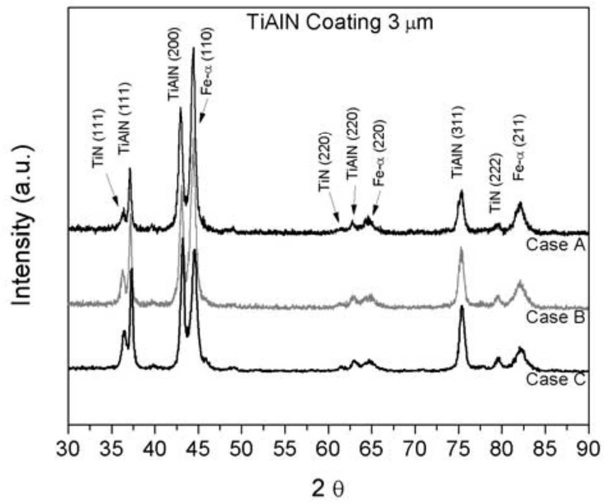

Figure 3. XRD patterns of the coating with thickness of 3 µm deposited over nitrided cases A, B and C.

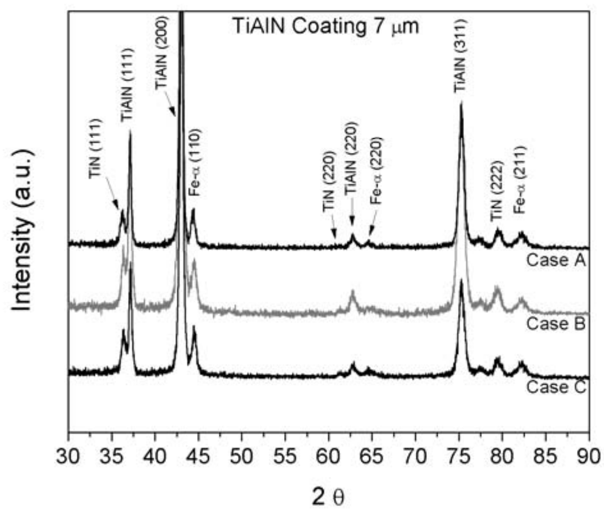

Figure 4. XRD patterns of the coating with thickness of 7 µm deposited over nitrided cases A, B and C.

3.3 Coating System Hardness and Elastic Modulus

Instrumented indentation hardness and elastic modulus of the TiAlN coating on nitrided and non-nitrided AISI H13 steel are shown in Figures 5 and 6. By examining Figure 5, one can notice that the coating surface hardness over nitrided cases is considerably higher for the coating system with thickness of 7 µm. Figure 5 also reveals that the nitrided layer characteristics affect the hardness of the coating when the TiAlN thickness is 3 µm. Moreover, the TiAlN coating with thickness of 3 µm deposited over nitriding case A and B resulted in a coating system with higher hardness than the TiAlN coating system deposited over the nitrided case C. This observation suggests that the thickness of the nitrided layer does not affect the coating hardness, but the nitrided layer hardness. In the coating system with thickness of 7 µm, the coating hardness is not affected by the nitrided layer characteristics. These observations suggest that the thicker the coating the higher the residual stresses and the hardness. In addition, the absence of a nitrided layer between the AISI H13 and the coating system produces higher residual stresses and consequently higher coating hardness.

Figure 6 shows that there is no significant difference among the elastic modulus of tempered and quenched H13 steel and the nitrided H13 substrate. But TiAlN elastic modulus changes with the coating thickness, the thicker the coating the higher the elastic modulus. In addition, the elastic modulus of the coating with thickness of 3 μm is influenced by the nitrided case characteristics while the elastic modulus of the coating with 7 μm is not influenced. It is also observed that the elastic modulus of the coating deposited on the non-nitrided surface is higher than the nitrided ones. Finally the elastic modulus of the coated samples tends to reach the elastic modulus of the substrate as one move away from the surface. Again these observations suggest that the residual stress, which is a function of the coating thickness, plays a major role in determining the coating system elastic modulus.

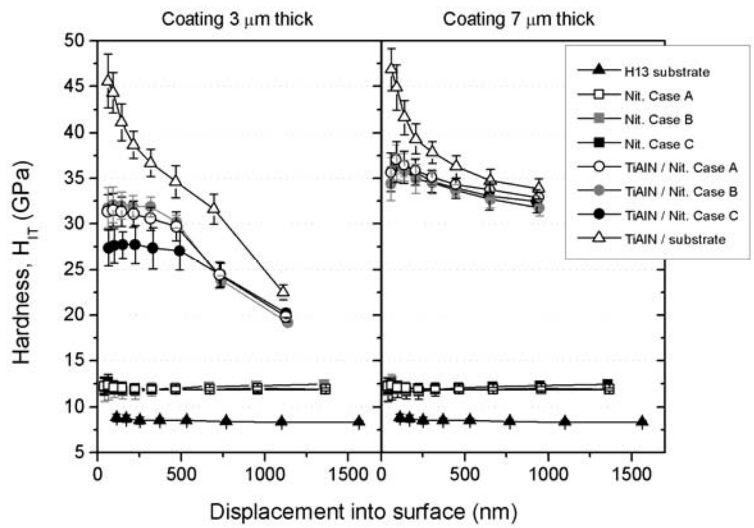

Figure 5. Instrumented Hardness of nitrided and non nitrided AISI H13 coated with TiAlN.

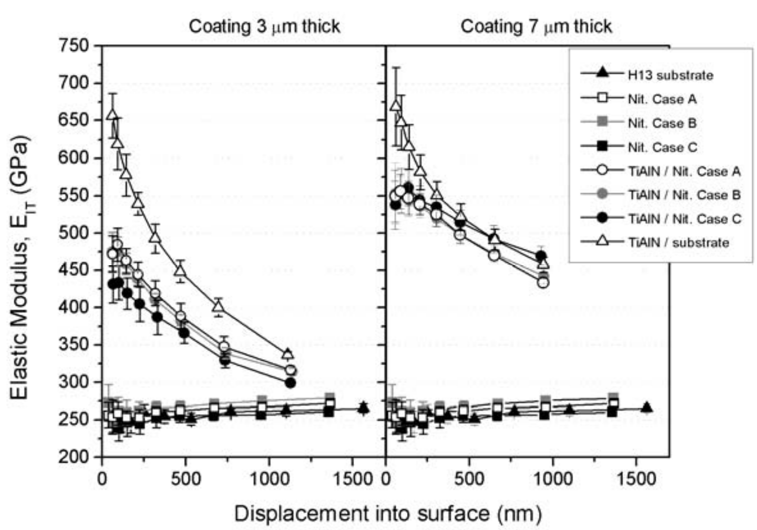

Figure 6. Elastic modulus of nitrided and non nitrided AISI H13 coated with TiAlN.

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3.4 Scratch Tests

The coatings wear resistance was measured through the groove area obtained by the cross-profile topography of the scratches from the scratch test. Figure 7 shows the worn area for both coating thicknesses on nitrided and non-nitrided surfaces. The results show that different nitriding conditions do not affect the wear resistance of the thickest coating, however, it strongly affects the thinnest coating. It has also been observed that the worn area was the smallest when the TiAlN was deposited over AISI H13 without nitriding. By carefully examining Figure 7 and comparing it with Figure 5, it is possible to notice that there is a strong relation between the coating worn area and the coating hardness.

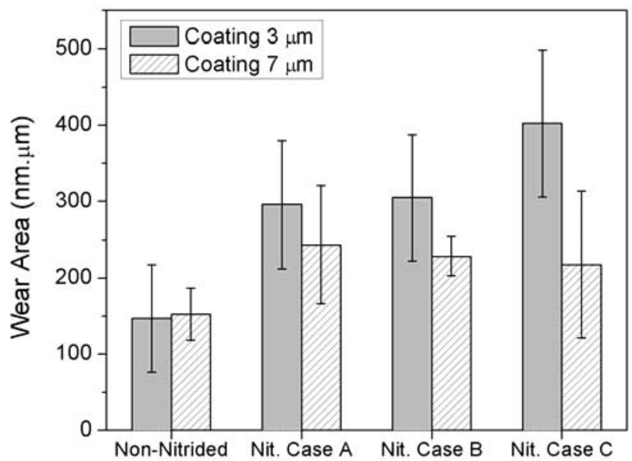

Figure 7. Worn area of nitrided and non nitrided AISI H13 coated with TiAlN.

3.5 Coating Adhesion

Figure 8 shows some typical images of the Rockwell indentations of the coatings on nitrided and non-nitrided steel. It was observed that after high load indentation, chipping took place for the 3 µm coating on non-nitrided steel surface. For coatings on nitrided surfaces, radial cracks are observed, suggesting a good adhesion of the coating. Although presenting higher hardness and elastic modulus, and higher wear resistance, the coating on non nitrided surface presented poor adhesion.

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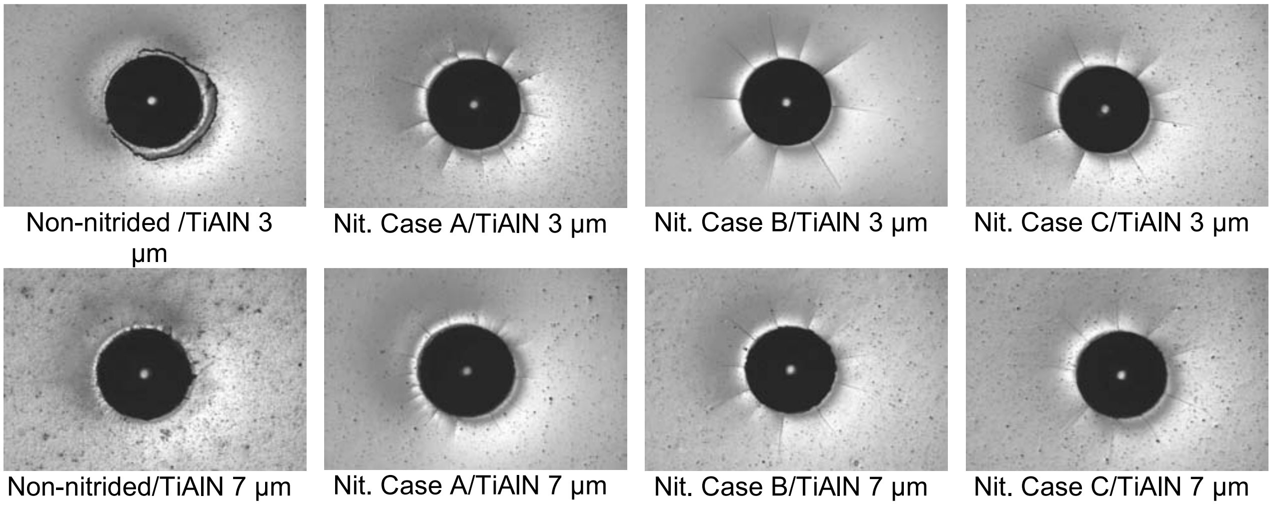

Figure 8. Rockwell C indentation aspect of nitrided and non nitrided AISI H13 coated with TiAlN.

4 CONCLUSIONS

Based on the results it is possible to draw the following conclusions: The hardness of the coating is higher when the AISI H13 steel surface is not nitrided for both coating thickness investigated in this work. Moreover, the hardness of the coating system deposited over nitrided samples changes with the nitrided cases when the coating thickness is 3 μm, but there is no change in the coating system hardness deposited over nitrided cases when the coating system thickness is 7 μm. The coating elastic modulus is influenced by the coating thickness. The elastic modulus of coating with thickness of 3 μm is influenced by the nitrided case characteristics. In addition, the coating deposited over non-nitrided AISI H13 surfaces presented smaller worn area in comparison with nitrided surfaces. Among the nitrided H13 surfaces, the one with 7 μm coating thickness presented a smaller worn area. Finally, it has been observed that the higher the hardness the lower the worn area produced by scratch test.

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