Evaluation of Mechanical, Tribological and Microstructural properties of coatings on non-nitrided and nitrided AISI4140

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Abstract— **Deposition of surface coating materials is one of the most important approach in improving friction and wear properties of surface. There is growing demand for low friction coatings such as TiN, TiAlN, CrN, WC, DLC, etc. that allows coating surfaces to rub against each other with reduced friction and wear. The use of low friction coating is to improve the Tribological properties of tools for metal cutting, forming and machine elements. It is therefore important to do research on materials that have low friction so that they can be used as surface coatings. Today, 4140 is the most commonly used of the high tensile steels with a wide range of applications. In the present work, evaluation of Mechanical, Tribological and Microstructural properties of coatings on non-nitrided and nitrided AISI4140 steel with a view of forming application is done. On AISI 4140, TiN and AlCrN PVD coatings are applied. Various tests such as Microhardness, surface roughness, Pin on Disc and Scratch tests are been performed to find the properties such as hardness, scratch resistance, wear resistance, surface roughness and microstructural properties of different types of coatings. Performance of tools can be increased by nitriding. It is seen that coatings improve the Tribological properties of steel. Duplex treated i.e nitrided as well as coated samples showed good results compared to that of nitrided and coated ones.**

Keywords: **AISI 4140, Low friction coatings, Microhardness, Pin on disk, Scratch tests, surface roughness.**

I. INTRODUCTION

IN industrialized societies there is an increasing demand to reduce or control friction and wear of engineering materials. The principal impetus behind this is related to economical and environmental aspects. The needs to extend the efficiency of machinery, to optimize energy consumption, to conserve scarce material resources and to reduce the use of hazardous lubricants, are just a couple of examples.[1]

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Surface engineering, including surface treatments and coatings, is one of the most effective and flexible solutions for tribological problems. Coatings change tribological systems by inducing residual compressive stresses, decreasing the friction coefficient, increasing the surface hardness, altering the surface chemistry, changing the surface roughness.

Many classes of composites exist, most of which are addressing improved mechanical properties such as stiffness, strength, toughness and resistance to fatigue. Coating composites (i.e. *surface engineered* materials) are designed to specifically improve functions such as tribological, electrical, optical, electronic, chemical and magnetic.[2]

Several other positive effects of applying coatings are:

- The improved wear resistance of coated metal cutting tools is usually utilised to increase the cutting speed and thereby the productivity, rather than to give a prolonged tool life.
- Reduced friction often means reduced energy consumption. In some cases, a lowered friction may permit the exclusion of lubrication or of cooling stages.
- Increased or controlled friction may be a beneficial effect in other applications such as brakes, bolted joints and safety connectors.
- Reduced tendency to sticking and material pick up from the counter surface is crucial to the performance of forming tools and many sliding applications. Anti sticking agents may be omitted in forming tool applications.
- Components of reduced weight can be designed by application of coatings. Reduced weight means e.g. an increased ratio of power to weight of car engines, which in turn may give lower fuel consumption.[2]

The challenging task of improving the properties of hard protective coatings, has gained much attention in recent years leading to a new method called duplex treatment, due to thermochemical pre-treatment of the substrate [3]. A combination of plasma nitriding and physical vapour deposition (PVD) processes has been found to be very attractive methods of duplex treatment, not only for increasing the load carrying capacity, but also for improving fatigue strength, temperature and chemical resistance as well as the tribological behavior of steel surfaces, particularly under high loads.[2][3]

II. LITERATURE REVIEW

Bojan Podgornik, Vojteh Leskov [4] reviewed the wear mechanisms encountered in forming processes, as well as various surface-engineering techniques designed to improve the wear resistance and the anti-sticking properties of forming tools. The possible benefits and restrictions of different surface-engineering techniques were presented for the example of sheet-metal forming, fine blanking and forging.

B. Podgornik and S. Hogmark [5] investigated the potential of using hard PVD coatings on forming tools. Tribological evaluation of TiN, TiB2, TaC and DLC coatings deposited on a coldwork tool steel was carried out in a loadscanning test rig and compared to the behaviour of different uncoated forming tool steels. Experimental results indicate that introduction of a proper hard coating will lead to an improved wear resistance and a longer lifetime of the forming tool. Furthermore, by using hard low-friction coatings excellent anti-sticking properties can be obtained.

Frank Hollsteina,, Renate Wiedemann, Jana Scholz [6] stated that there are limititions on the use of magnesium based light metal alloys due to low mechanical strength and the low corrosion resistance. The industrially very important layer systems TiN, CrN, TiAlN, NbN-(TiAl)N, CrN-TiCN and the multi-layer composite AlN/TiN are discussed in detail. Furthermore, the protective effect of CrN/NbN superlattices has been studied. Both the mechanical behaviour and the corrosion resistance of the specimens have been studied after coating. The chemical composition of the thin films was analysed by GDOES. A special emphasis of the investigation was laid on searching for appropriate stripping procedures due to the fact that the substrate material is very reactive.

J.K. Chen, [7] et. al. used cathode arc evaporation to coat TiAlSiN, TiN, and AlTiN multilayer films on tool surfaces. The specimens were examined by scanning electron microscopy to characterize film structures, thickness and compositions. Wear tests and nanoindentation tests were also performed to evaluate their coefficients of friction and thin film adhesion. The different natures of three coatings can affect their performance and thus their applicability for cutting of different materials.

R.J. Rodrıguez, et. al. [8] reported a comparative study of the tribological properties of the most employed hard coatings like TiN, TiCN, TiAlN, CrN and ZrN. In this study, microhardness tests were carried out by using a microindenter. Friction and wear tests were carried out by using a ball-on-disk tribometer and an optical profilometer. AUGER and XPS techniques were employed to measure the stoichiometry and thickness of the different films. All the coatings showed a very smooth topography, due to the very few and small droplets on the coated surface. It was also seen how the universal hardness (hardness under load) for these coatings from down to up was CrN < TiCN < ZrN < AlTiN <TiN. Friction tests on all the different coatings showed a decrease in friction coefficient, especially at high humidity levels. The best results were obtained for the TiCN (0.2–0.4), and for the ZrN (0.3–0.35) coatings.

B. Warcholinski, et. al.[9] characterized the tribological properties of thin Cr-N coatings, both monolayer Cr_2N, CrN coatings and multilayer Cr/CrN, Cr_2N/CrN coatings, deposited by cathodic arc physical vapour deposition (CAPVD). Structure of the coatings were investigated using the scanning electron microscopy (SEM). The XRD examination was carried out to specify the phase structure, EDS to define the chemical composition of the coatings. The investigation also included microhardness, roughness tests, adhesion, friction coefficient and wear rate.

O. Alpaslan et. al. [10] examined the characterisitics of arc PVD- CrN coatings formed on plasma nitrided and as-received surfaces of an hardened AISI 4140 steel before and after nitiriding by microhardness, adhesion and wear tests. CrN coating deposited on the nitrided surface exhibited remarkable advanced properties as compared to the CrN coating deposited on the as-received surface.

III. PROBLEM DEFINITION

A. Problem Statement

. There is growing demand for low friction coatings such as TiN, TiAlN, CrN, WC, DLC, etc. that allows coating surfaces to rub against each other with reduced friction and wear. The use of low friction coating is to improve the Tribological properties of tools for metal cutting, forming and machine elements. It is therefore important to do research on materials that have low friction so that they can be used as surface coatings. Therefore the present study aims to evaluate the Mechanical, Tribological and Microstructural properties of TiN, AlCrN coatings on nitrided and non-nitrided AISI4140 and selection of the coating with perspective of general forming applications is done.

B. Objectives

1. To find the mechanical properties and surface topographical properties of coatings such as hardness and surface roughness.
2. To conduct tribological tests to find the scratch resistance, wear resistance.
3. To perform the microstructural analysis.
4. To conduct the comparative analysis of properties for various specimens coated with different types of coatings as well as nitrided and non nitrided specimens.

C. Methodology

1. Selection of substrate material for experiment.
2. Selection of coatings to be coated on substrate.
3. Preparation of substrate samples for each test as per the given standards.
4. Nitriding and coatings application on prepared substrate samples.
4. Conducting mechanical tests such as Hardness and surface profiliometry.
8. Conducting Scratch test and wear test to find the tribological properties.
9. Carrying out microstructural analysis of weared samples.
10. Carrying out comparative analysis.
11. Report completion and other activities.

D. *Substrate selection: AISI 4140*

4140 is a 1% chromium - molybdenum medium hardenability general purpose high tensile steel. This material was chosen because it can be nitrided to a high surface hardness without losing its toughness, which makes it suitable for highly loaded machine parts. 4140 is used extensively in most industrial sectors for a wide range of applications. Chemical composition, mechanical and thermal properties of AISI4140 are given in Table I, II and III.

TABLE I
CHEMICAL COMPOSITION OF AISI4140 ALLOY STEEL.

C	Cr	Mn	Si	Mo	S	P
0.380 - 0.430	0.80 - 1.10	0.75 - 1.0	0.15 - 0.30	0.15 - 0.25	0.04	0.035

TABLE II
MECHANICAL PROPERTIES OF AISI 4140 ALLOY STEEL

Properties	Tensile strength	Yield strength	Bulk modulus	Elastic modulus	Shear modulus	Poisson's ratio	Elongation at break (in 50 mm)	Hardness, Brinell
Metric	655 MPa	415 MPa	140 GPa	190-210 GPa	80 GPa	0.27-0.30	25.70 %	197

TABLE III
PHYSICAL AND THERMAL PROPERTIES OF AISI 4140 ALLOY STEEL

Properties	Density	Melting point	Thermal expansion co-efficient (0-@100°C/32-212°F)	Thermal conductivity (@ 100°C)
Metric	7.85 g/cm^3	1416°C	12.2 µm/m°C	42.6 W/Mk

E. *Coatings Selection*

The TiN and AlCrN PVD coatings with approximately 4µm thickness were applied on AISI 4140 and nitrided AISI 4140. The coatings were applied at Oerlikon Balzer's Coatings Pvt. Ltd. Pune. Some of the coating characteristics given by Oerlikon Balzer's are shown in Table 4.

TABLE IV
COATING CHARACTERISTICS GIVEN BY OERLIKON BALZER'S

Coating	TiN	AlCrN
Colour	Golden yellow	Bright grey
Coating temperature (°C)	400	<500
Maximum service temperature	600	1100
Structure	Columnar	Monolayer

IV. EXPERIMENTAL

A. *Microhardness*

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. Microhardness test was carried out using Vickers microhardness tester as shown in fig.1 using ASTM E10-01^{e1}.[18]

Fig.1. Vickers Microhardness Tester. (Make-Reichert Austria. Load -100 grams)

Sample preparation

Samples prepared for microhardness testing were of size, diameter= 30mm and thickness= 18mm with both surfaces flat and polished to 0.4µm. Three samples were salt bath nitrided at temperature around 450°C and two of these were PVD coated and one AISI 4140 without nitrided and one with nitriding was prepared.

B. *Coating Thickness*

Thickness of coatings was calculated using X-Ray Fischeroscope at Oerilikon Balzer's Coating Pvt. Ltd.
Sample Details: Samples used for measurement of coating thickness were each of length 38mm and diameter 13mm.

C. *Surface Topography*

A quantitative complimentary method to examine the surface morphology is obtained by analysing the surface roughness using surface profilometry. The surface roughness test was carried out using Mitutoyo SJ-210 Surface roughness tester shown in fig.2

Fig.2. Mitutoyo SJ-210 Surface roughness tester

Sample preparation:

For surface roughness testing two square rods of dimension 204mm*24mm*11mm of AISI 4140 were prepared and the surfaces were extremely smooth finished with Ra approximately 0.4µm. The specimens were salt bath nitrided and after nitriding they were coated with TiN and AlCrN respectively.

a. TiN coated nitrided AISI4140

b. AlCrN coated nitrided AISI4140

c.

Fig.3. a.TiN b.AlCrN coated nitrided AISI4140

D. *Friction and wear test:*

A pin on disc tribometer is the equipment used to determine the sliding friction coefficient and wear resistance of surfaces. Pin-on disc friction and wear monitor TR-20LE-PHM-400 shown in fig.3 was used to carry out tests.

Fig.4. Pin on Disc apparatus

Specimen Preparation:

Wear testing requires two materials one for the pin and other for the disc. The typical pin specimen is cylindrical or spherical in shape. Typical cylindrical or spherical pin specimen diameters range from 2 to 10 mm. The typical disk specimen diameters range from 30 to 100 mm and have a thickness in the range of 2 to 10 mm.

The cylindrical pins are made of AISI 4140 material. The pins are made of diameter 6mm and length 30mm. Both the ends of the pins are made perfectly flat with surface roughness Ra less than 0.4µm. The pins are salt bath nitrided and the PVD coated with the coating thickness of approximately 4µm. Two pins of AISI4140 are directly coated. The Disk is made up of EN 31 with 60 HRC having diameter 165 mm and thickness of 8 mm which was electroplated. Test is carried as per ASTM G-99 04 [19]. Test was carried for the constant load of 50N with 500 rpm constant speed and the sliding distance 500m. These parameters are selected with respect to forming tool application.

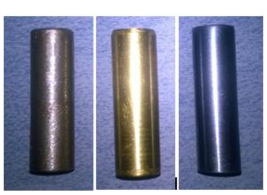


Fig.5. AISI4140 pin(left),AISI4140+TiN coated(middle), AISI4140+AlCrN(right).

E. *Scratch Resistance Test*

Scratch test was carried on a scratch resistance test rig as shown in fig.6 below. A simple 500g load was applied on a diamond tip indenter which rests on the specimen and specimen is pulled gently so that the specimen is been scratched.

Fig.6. Scratch resistance test rig.

Sample preparation:

Samples used were same as those used for surface roughness measurement test.

IV. RESULTS AND DISCUSSIONS

A. *Hardness:*

TABLE V
MICROHARDNESS (HV)

Sample	Hv
AISI 4140	278
AISI 4140+ Nitriding	692
AISI 4140+ Nitriding+ TiN	1041
AISI 4140+ Nitriding+AlCrN	1650

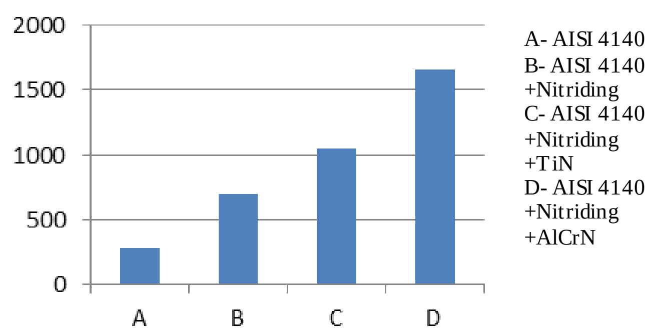


Fig.7. Microhardness

Vickers microhardness test performed on coated specimens, showed improved composite hardness obtained by nitriding of the substrate. This was observed for both coatings. (fig.7). By increasing the hardness of the substrate by nitriding, it improves the load carrying capacity of the substrate and provides good support for the coating as well as reduces the large hardness gradients at the coating-substrate interface. AISI 4140 nitrided and coated with AlCrN showed the highest hardness which is nearly about six times the hardness of only substrate i.e AISI 4140. So AlCrN coating is preferable.

B. *Coating thickness:*

Coating thickness for both the coatings was found to be nearly same and was approximated to 4 microns.

TABLE VI
COATING THICKNESS

Coating	n1 (µm)	n2 (µm)	n3 (µm)	n4 (µm)	Mean (µm)	Std. deviation(µm)
TiN	3.740	3.841	3.779	3.837	3.799	0.049
AlCrN	3.523	3.497	3.570	3.513	3.526	0.032

C. *Surface roughness*

Stylus profilometry was used to measure the change in the surface roughness caused by nitriding and by the coating deposition. After nitriding, the average roughness value *R*a of the original ground surface increased as given in table VII. As coatings were deposited on substrate of Ra 0.4µm the surface roughness of TiN coated sample was found higher than AlCrN. The coatings with less Ra are preferred so considering the surface roughness AlCrN coating is preferable.

TABLE VII
SURFACE ROUGHNESS

Sr.no.	Sample	Surface roughness Ra (µm)			Change in Ra
		Reading 1	Reading 2	Average $(Ra)_c$	$(Ra)_c$- 0.4
1.	TiN Coated	0.742	0.697	0.719	0.319
2.	AlCrN coated	0.562	0.442	0.502	0.102

D. *Friction and wear test*

Friction and wear test results obtained from pin on disk test for constant load of 50N, speed 500rpm and sliding distance of 500m are as tablated in table VIII below.

TABLE VIII
WEAR AND COF

Sample	Wear (µm)	COF
AISI 4140	46.77	0.087
AISI 4140 + Nitriding	22.48	0.106
AISI 4140 + TiN	18.96	0.253
AISI 4140 + AlCrN	17.75	0.125
AISI 4140 + Nitriding + TiN	5.74	0.107
AISI 4140 + Nitriding + AlCrN	2.80	0.212

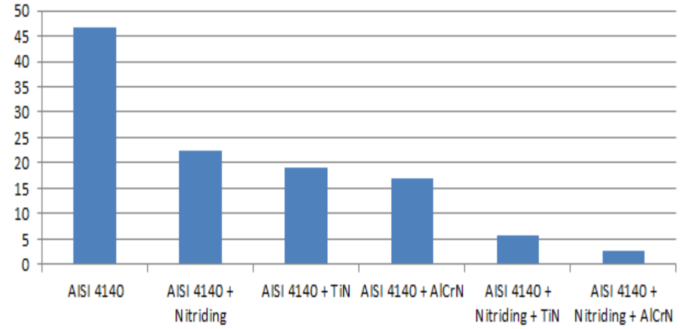


Fig.8. Wear of samples in µm

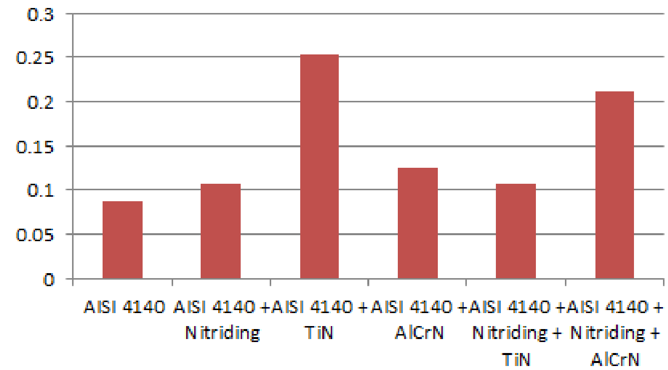
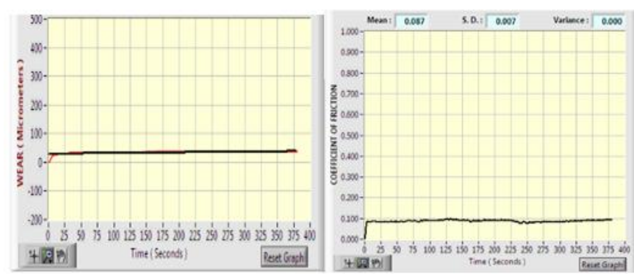
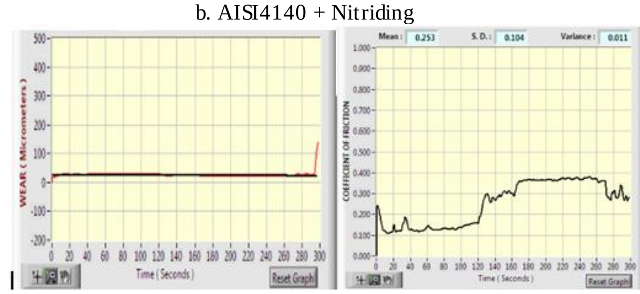


Fig.9. Coefficient of friction (COF)

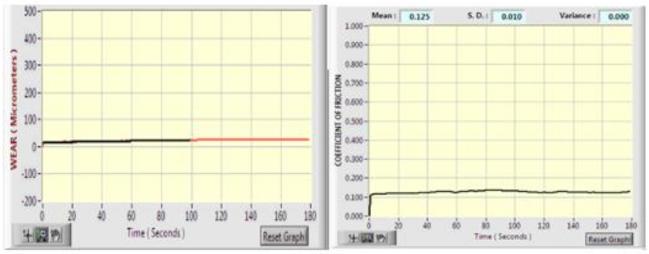


a. AISI4140 Only

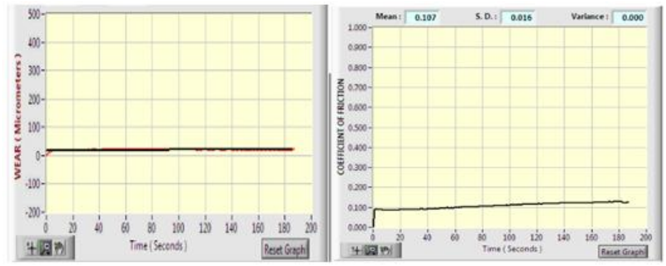
b. AISI4140 + Nitriding



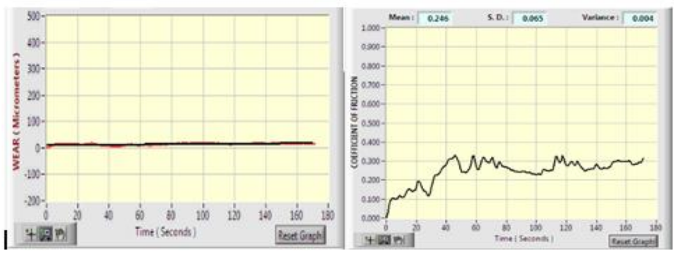
c. Aisi4140 + TiN



d. AISI4140 + AlCrN



e. AISI4140 + Nitriding + TiN



f. AISI4140 + Nitriding + AlCrN

Fig.10. Wear and COF graphs for various samples given in Table VIII

The coefficient of friction of surface-treated AISI 4140 steel with TiN or AlCrN coatings increased from 0.08 for only substrate to 0.253 for TiN coated substrate as shown in fig 9. From fig. 10 a. and b. it can be seen that the sliding COF for substrate and nitrided substrate is nearly constant throughout the time period and is around 0.1. For TiN coating deposited on substrate directly, the COF has the highest value i.e 0.253 and the value increased further with sliding distance as show in fig.10c, whereas TiN applied on nitrided sample showed very less increase in COF shown in fig. 10e. AlCrN coated on nitrided substrate showed the variation of sliding COF between 0.25-0.35 throughout the cycle as shown in fig. 10f.

The wear values in µm are tabulated in Table VIII and the comparison is shown in fig.8.The variation of wear with respect to time for each sample is shown in fig.10. From fig.8 it can be seen that wear of substrate is higher around 48µm, which is reduced to the lowest by applying nitriding and AlCrN coating and the respective value is 2.80 µm. It can be seen that deposition of the wear protective coating (TiN, AlCrN) decreased the pin wear rate significantly. The wear of the investigated coatings decreased with increased substrate hardness and nitriding. Compared to coated substrates, the nitrided and coated specimens showed improved sliding wear resistance. This can be due to a higher substrate hardness and improved coating–substrate adhesion.

E. Scratch resistance

Scratch marks were observed for both the coatings but scratch haven't penetrated upto the bear material.

F. Future Scope

Microstructural analysis will be done using Scanning Electron Microscope. The weared surfaces of pin-on-disk specimens will be analyzed. The cross section of pins will be taken and analyzed under SEM.

V. CONCLUSIONS

Microhardness test performed on various specimens showed the improvement of composite hardness of nitrided and coated samples. By increasing the hardness of the substrate by nitriding, it improves the load carrying capacity of the substrate and provides good support for the coating as well as reduces the large hardness gradients at the coating-substrate interface. AISI 4140 nitrided and coated with AlCrN showed the highest hardness (1650 hv) which is nearly about six times the hardness of only substrate i.e AISI 4140. The decreasing hardness order is: (Nitrided and AlCrN coated)> (Nitrided and TiN coated) > (nitrided AISI4140)>(substrate AISI4140).

The surface roughness of TiN coated sample was found higher than AlCrN. Compared to coated substrates, the nitrided and coated specimens showed improved sliding wear resistance. The wear resistance can be arranged in order as: (Nitrided and AlCrN coated)> (Nitrided and TiN coated) > (AlCrN coated) >(TiN coated) >(substrate AISI4140). From friction tests it can be concluded that though the COF of coated specimens is more than that of uncoated and nitrided ones the increase is not much and these increase in COF can be neglected since the other Mechanical and Tribological properties of coated and nitrided samples are improved a lot compared to uncoated and non-nitrided samples. Morever AlCrN can sustain the high temperatures upto 1100^{o}C.

Therefore it can be said that AlCrN coating applied on nitrided AISI4140 will provide suitable solutions to problems related to formig tool applications.

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