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CRYOGENIC TREATMENT AND PVD COATINGS TO AUGMENT WEAR RESISTANCE OF TOOL STEELS

ZASTOSOWANIE OBRÓBKI KRIOGENICZNEJ I POWŁOK PVD DO ZWIĘKSZENIA ODPORNOŚCI NA ŚCIERANIE STALI NARZĘDZIOWYCH

Słowa kluczowe

powłoki PVD, stale narzędziowe, obróbka kriogeniczna

Key words:

PVD coatings, tool steels, cryogenic treatment

Summary

The influence of deep cryogenic treatment and PVD coatings on the wear resistance and toughness of D2, S7, H13 and M4 tool steels were studied. Three different PVD coatings were applied after heat treatment: AlCrN, TiAlN, TiCrN. In order to research the wear resistance, the ball-on-disk tribological test was used per ASTM G-99; material toughness was evaluated through the

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Charpy-V test. Wear scars were examined through optical microscopy to calculate the volume loss. It is found that cryogenic treatment improved wear resistance and toughness up to 63% and 12% respectively. The material with the best combination of wear resistance properties and toughness was S7 with cryogenic treatment and TiAlN coating.

INTRODUCTION

Materials selection is highly important when manufacturing tools for forming, cutting, extruding, etc., since each process has specific parameters of speed, temperature, and pressure. D2 steel is one of the most commonly used tool steel due to its impact toughness, wear resistance, hardness, and low cost **[L. 1]**. For these reasons 35% of the total tool steel sales come from D2 steels **[L. 2]**. However, D2 is recommended for cold working, and as shown in previous studies is not the ideal candidate for cutting applications **[L. 2]**. In our study, the following steels were selected: H13, S7, and M4 processed by powder metallurgy. H13 is usually used for hot forging dies **[L. 3]** but have also been applied for cuttings blades **[L. 4].** High speed tool steel M4 may also be used for cutting tools due to their high abrasion resistance **[L. 5]**, and shock resisting steel S7 is a good candidate for guillotine blades for cutting steel sheets **[L. 5]**.

A cryogenic treatment and coatings deposited by means of Physical Vapour Deposition (PVD) were applied in order to increase the wear resistance of selected tool steels. Quenching increases wear resistance of steels by a microstructure transformation from austenite to martensite. However, one issue with this heat treatment is the amount of retained austenite (γ_R). Cryogenic treatment increases wear resistance by achieving the transformation of γ_R into martensite, and it precipitates fine secondary carbides with dimensions of about 5 µm **[L. 6].** This process consists in slowly cooling the material to -196ºC for 20 to 40 hours, followed by a tempering at 150°C **[L. 7].** PVD coatings are known to increase tool life by decreasing the coefficient of friction (COF) and increasing the wear resistance of the coated element **[L. 8]**. Studies have shown a considerable decrease in fatigue wear **[L. 9]**, scuffing wear, and tribo-corrosion **[L. 10]** with the application of PVD coatings. This method consists in depositing thin and hard coatings at temperatures of 200-500°C, which is lower than the temperature required for structural transformations on steel. The coatings are usually nitrides of titanium, chromium, and/or aluminium with higher hardnesses than carbides **[L. 11]**, and thicknesses ranging from 0.5–4 µm **[L. 12]**.

EXPERIMENTAL: MATERIALS AND TESTING

The H13, S7, and M4 specimens were selected and compared to D2. Chemical composition analysis was done by a Spectromaxx spectrometer **(Table 1)**.

Table 1. Chemical composition of tool steels
Tabela 1. Skład chemiczny stali narzędziowych

Steel	%C	%Cr	%V	%Mn	%Si	%Mo	%W
D2	1.54	11.92	0.80	0.32	0.31	0.70	–
H13	0.43	5.02	1.00	–	0.79	1.31	–
M4	1.77	4.06	6.04	–	–	4.25	4.08
S7	0.53	3.29	0.27	0.62	0.37	1.50	–

The materials were quenched and tempered according to the supplier's specification **[L. 14]**. The temperatures for heat treating each tool steel are shown in **Table 2**. After quenching, materials were cryogenically treated at *Cryosa Servicios SA de CV* **[L. 13]** in order to transform γ_R into martensite. Materials were cooled at a rate of 1°C/min to -196°C and were kept at this temperature for 20 hours. Materials were tempered at 150°C.

Table 2. Temperatures for quenching and tempering D2, H13, M4, and S7 tool steels [L. 14]
Tabela 2. Temperatury ulepszania cieplnego stali narzędziowych D2, H13, M4 i S7 **[L. 14]**.

Steel	Quenching Temperature (°C)	Tempering Temperature (°C)
D2	980	550
H13	995	540
M4	1220	550
S7	950	380–400

PVD coatings of AlCrN, TiAlN, and TiCrN were deposited by *Oerlikon Balzers* on heat-treated samples due to their low COF **(Table 3).**

Table 3. Properties of selected PVD coatings [L.12]
Tabela 3. Właściwości wybranych powłok PVD **[L. 12]**

Properties	*AlCrN*	*TiAlN*	*TiCrN*
Vickers Hardness (HV 0.05)	3,200	3,400	2100
Service Temperature (ºC)	1100	900	700
COF Against Steel (Dry)	0.35	0.30–0.35	0.5
Thickness (µm)	1 – 6	1 – 6	1 – 6

Impact toughness was tested by a Chapy-V type test with a Tinius Olsen pendulum. The sample dimensions were 55 *x* 10 *x* 10 mm according to ASTM standard E 23 **[L. 15]**, and five samples were tested for each of the different materials with and without cryogenic treatment**.** Only base materials and cryogenically treated samples were analysed with this test, since the effect of the thin PVD coating on impact toughness is negligible.

The wear resistance of selected tool steels was characterised by the T-11 tribotester with a ball-on-disk setup **(Fig. 1)** according to G99 **[L. 16]** and G133 **[L. 17]** ASTM standards. Disk samples had a diameter of 25.25 mm and the thickness of 6.35 mm. The ball test material was AISI 52100 steel, with a diameter of 6.3 mm and a hardness of 59 HRC. Tribological tests were performed under conditions of 50N load, a rotational speed of 125 rpm, a humidity level of 50%, a wear track radius of 10mm, and 10min of test duration. At least 3 tests for each material (quenched, cryogenic, PVD coated) were done, according to the Dixon probabilistic methodology to generate statistically reliable results.

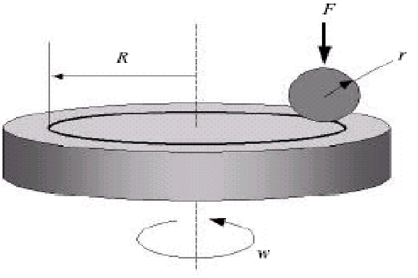
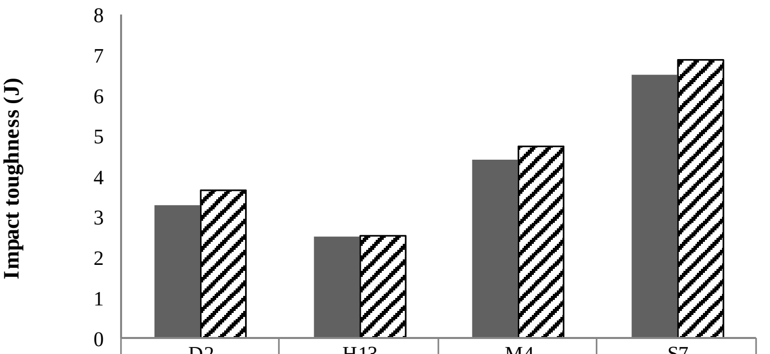


Fig. 1. Arrangement of ball-on-disk test [L. 16, 17]
Rys. 1. Schemat węzła tarcia urządzenia kula-tarcza **[L. 16, 17]**

RESULTS

Charpy V-test results showing the impact toughness in J for D2, H13, M4 steels are shown **Fig. 2**.



	D2	H13	M4	S7
Quenched	3.26	2.50	4.40	6.50
Cryogenic	3.66	2.54	4.75	6.90

Fig. 2. Impact toughness of D2, H13, M4 and S7 steels quenched and cryogenically treated
Rys. 2. Wyniki pomiarów udarności stali narzędziowych D2, H13, M4 i S7 poddanych hartowaniu i obróbce kriogenicznej

The application of cryogenic treatment showed a small effect on impact toughness, with improvements ranging from 2 to 11% **(Fig. 2)**. Test results showed that H13 had the lowest impact toughness at 2.5 J and 2.54 J for quenched and cryogenic treated samples, respectively. The material with the highest impact toughness S7, with 6.5 J when quenched, and with 6.9 J when cryogenically treated.

For the tribological tests, wear tracks and depth profiles were measured by an optical microscope and a rugosimeter–perfilometer. Wear volume loss for disk and ball materials were calculated using the relationship given in test method G-99 **[L. 16]**:

$$\textbf{Disk volume loss} = 2\pi R\,[r^2 \sin^{-1}(d/2r) - (d/4)(4r^2 - d^2)^{1/2}]$$

Where: R = wear track radius, and
d = wear track with.

$$\textbf{Ball volume loss} = (\pi h/6)\,[3d^2/4 + h^2]$$

Where: $h = r - [r^2 - d^2/4]^{1/2}$
d = wear scar diameter, and
r = ball radius.

According to the G-99 **[L. 16]** and G-133 **[L. 17]** ball-on-disk test methods, three different wear situations can be presented depending on the material's properties **(Fig. 3)** as follows: (a) only the ball material shows significant wear, (b) only the disk shows significant wear, and (c) both the disk and the ball materials show significant wear.

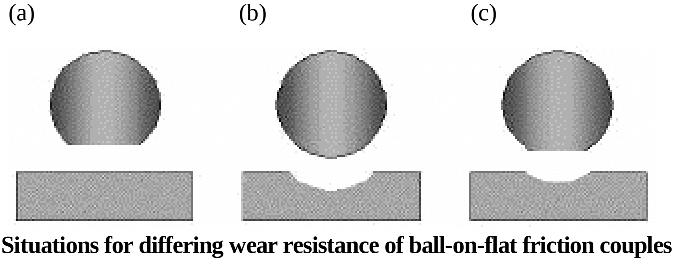


Fig. 3. Possible Situations for differing wear resistance of ball-on-flat friction couples [L. 16, 17]
Rys. 3. Schematy zużycia par ciernych w skojarzeniu kula–płaska powierzchnia

For quenched and cryogenically treated materials significant wear was observed for both the disk and ball, similar to the wear situation presented in **Fig. 3c**. For PVD coated samples only the ball showed significant wear (**Fig. 3a)**, due to their high hardness.

Wear tracks for quenched, cryogenically treated, and PVD coated H13 steel are shown in **Fig. 4**, as measured by optical microscope and rugosimeter–perfilometer.

(a)

(b)

(c)

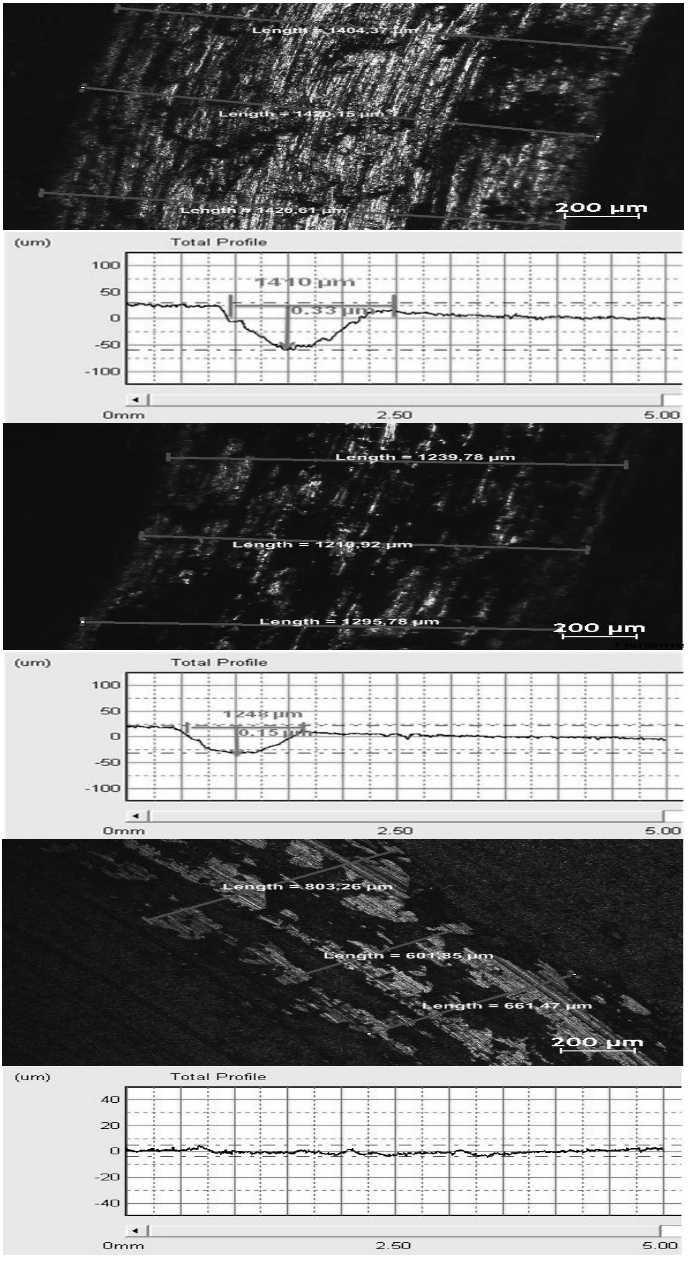


Fig. 4. Wear tracks and roughness profiles of H13 disks: (a) quenched, (b) cryogenically treated, (c) TiAlN PVD coated

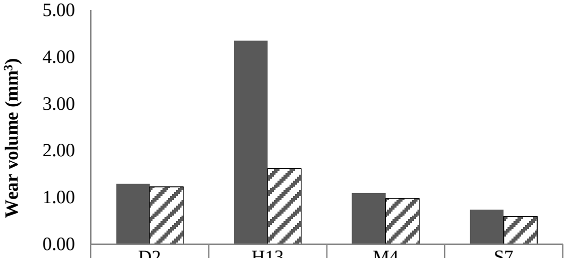
Rys. 4. Zdjęcie ścieżki tarcia i profil chropowatości tarczy ze stali narzędziowej H13: a) hartowanej, b) po obróbce kriogenicznej, c) z powłoką TiAlN

Both the quenched and cryogenically treated samples showed a wear track with a significant depth (**Fig. 4a** and **4b)**; however, the PVD coated sample did not show a wear depth, thus the apparent wear track may be attributed to the adhesion of ball material on the surface of the disk during the test **(Fig. 4c).** Similar results were observed for all PVD coated samples.

Wear volume loss of quenched and cryogenically treated disks is shown in **Fig. 5**.

According to the results, S7 steel showed the lowest wear volume loss with 0.74 mm^3 and 0.59 mm^3 with quenching and cryogenic treatment, respectively. The application of cryogenic improved wear resistance of D2, H13, M4, S7 steel a 4.7%, 63%, 10%, and 20%, respectively.

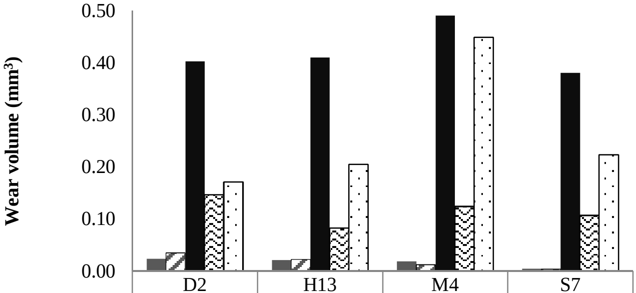
The wear volume loss of ball materials used as counterface for quenched, cryogenically treated, and PVD coated steels is shown in **Fig. 6**.



	D2	H13	M4	S7
Quenched	1.29	4.35	1.09	0.74
Cryogenic	1.23	1.61	0.98	0.59

Fig. 5. Wear volume loss of disk samples of quenched and cryogenically treated D2, H13, M4, and S7 tool steels

Rys. 5. Zużycie objętościowe tarczy ze stali narzędziowej D2, H13, M4, i S7 poddanej hartowaniu i obróbce kriogenicznej



	D2	H13	M4	S7
Quenched	0.0239	0.0214	0.0191	0.0044
Cryogenic	0.0350	0.0222	0.0123	0.0040
TiAlN	0.4027	0.4101	0.4905	0.3810
AlCrN	0.1466	0.0826	0.1242	0.1071
TiCrN	0.1711	0.2051	0.4490	0.2234

Fig. 6. Wear volume loss (mm^3) of balls used as counterfaces for the ball-on-disk test

Rys. 6. Zużycie objętościowe kuli (mm^3) użytej w teście kula-tarcza jako przeciwpróbka

In the case of ball specimen, the higher the volume loss, the higher is the wear resistance of the samples (disks). Ball materials used for quenched and cryogenically treated disks had the lowest wear volume loss, as the disk samples showed the highest wear. On the other hand, balls used as counterfaces for PVD coatings showed the highest wear, as the disk did not showed a measurable volume loss for the test conditions. The coating with the highest volume loss of ball material for all base materials was the TiAlN coating.

CONCLUSIONS

This investigation presented the effect of cryogenic treatment on the impact toughness and wear resistance of D2, H13, M4, and S7 tool steels. Overall, the application of this treatment had a small effect on impact toughness. Tribological tests showed a decrease of a wear volume loss of 63% when applying cryogenic treatment to H13. The application of PVD coating showed the highest wear resistance when applied on the selected steels, since only counterface ball materials showed any wear volume loss.

For applications of high impact toughness and wear resistance, cryogenically treated S7 is recommended. This material showed an improvement of 110% and 54% in wear resistance and impact toughness as compared to D2 steel, a material generally used in industry for cutting operations. The application of a TiAlN PVD coating is also recommended, because it showed the highest wear volume loss of ball counterface materials.

Future experiments should be performed with a ball or pin counterface material with a higher hardness than the applied PVD coatings in order to be able to better compare the results of all coated and uncoated materials.

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Streszczenie

W pracy przedstawiono wyniki badań wpływu obróbki kriogenicznej i powłok PVD na odporność na ścieranie oraz udarność stali narzędziowych D2, S7, H13 i M4. Zastosowano trzy różne powłoki PVD: AlCrN, TiAlN, TiCrN, które wcześniej poddano obróbce cieplnej. W celu zbadania odporności na zużycie przeprowadzono badania z użyciem testera kula-tarcza zgodnie z wymogami normy ASTM G-99. Udarność została oceniona z wykorzystaniem młota Charpy'ego. Zużycie objętościowe zostało zmierzone za pomocą mikroskopu optycznego.

Stwierdzono, że obróbka kriogeniczna polepszyła odporność na ścieranie o 63%, a udarność o 12%. W wyniku przeprowadzonych badań stwierdzono, że materiałem o najlepszych właściwościach na ścieranie i udarność jest stal narzędziowa S7 poddana obróbce kriogenicznej i pokryta powłoką TiAlN.