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Energy-Efficient Application of CrN Coating on Low-Alloy Tool Steel: Comparative Analysis of Technological Processes

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Abstract

This research examines the technological processes of applying CrN coating on low-alloy tool steel, focusing on the comparison between hardening-tempering-coating (HTC) and hardening-coating (HC) processes, with an emphasis on energy savings. The study investigates the chemical composition, microstructure, mechanical properties, fractography, residual stress, and corrosion resistance of the coated tool steel. Notably, the results indicate no significant differences in the microstructural, mechanical, and corrosion properties between the HTC and HC processes, suggesting that tempering may be excluded without compromising the quality. This study introduces a novel approach to tool steel coating, which improves energy efficiency while maintaining high-quality outcomes. The findings highlight potential improvements in industrial applications, offering an energy-efficient alternative that does not sacrifice the performance or durability of the tool steel. This advancement could lead to significant improvements in manufacturing efficiency and sustainability.

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1. Introduction

Unalloyed, low and medium alloyed or high-speed tool steels are used for cutting, slicing and chopping. Low-alloyed tool steels are alloyed with Mn and Cr, which increase hardenability, Si increase ultimate tensile strength, the addition of V reduce the grain size. These steels are usually hardened at temperature of 730°C – 820°C and tempered at temperatures of 180°C – 250°C, when the smallest deformations occur (Skočovský et al., 2015; Fremunt et al., 1994). High requirements must be met by tool steels, hardness, cutting property, wear resistance, hardenability, constancy of dimensions and high service life. The one of the possibilities to meet these requirements is coating, the formation of a thin layer of hard material in the order of micrometers with increased resistance to wear. High-quality coating can increase the life of tools up to twice (Grainger and Blunt, 1998). The coating methods used are chemical (CVD – Chemical Vapour Deposition) or physical (PVD – Physical Vapour Deposition). The CrN coatings are coatings deposited with PVD method. These coatings have a fine-grained structure, low internal stresses, high hardness and adhesion to the surface of the substrate, low COF,

excellent corrosion resistance. They are used on tools for cutting, cold forming, they have also found application e. g. in the plastic industry and in wood processing as coatings on forms (Grainger and Blunt, 1998; Kusý et al., 2007; Hazlinger and Moravčík, 2013).

2. Literature review

The CrN deposition process can be performed at the temperatures in temperature range 180°C to 200°C or 400°C – 500°C. In the work authors, Navinsek and Panjan, 1995, the CrN (PVD) coatings were applied on the AISI tool steels (AISI H11, AISI D2 and D3) at 200°C. Increased tool life, a higher quality of surface of the products and high reliability of CrN coated tools in production were observed during long-term performance tests. In the study (Gählin et al., 1995) is shown, that mechanical and tribological properties of the low-temperature CrN coating (200°C) are comparable with the standard temperature CrN coating although difference in microstructure and chemistry were observed. The CrN coatings are widely used because of their good wear resistance and good oil retention in oil lubricated



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applications (Holmberg and Matthews, 1994). The good adhesion of the CrN/TiN coatings deposited onto a high-speed steel at the temperatures of 130°C – 200°C was confirmed by the authors, Rabadzhiyska et al., 2018. In investigations, Navinsek et al., 1995 and Navinsek et al. 1997, the authors state that CrN coating prepared at 200°C can be used as a hard and oxidation resistant coating in industrial practice incl. working temperature at 750°C and that CrN coatings used in moulds operating at temperatures of 180°C – 220°C provided high quality in practical use. The authors, Aharonov et al., 1993, presented results about CrN coatings deposited at 200°C – 230°C (low temperature) and 480°C – 540°C (high temperature) onto high-speed steel. Both coatings exhibit good adhesion and a dense structure morphology, higher hardness was observed at coating deposited at lower temperatures. The authors, Zhang et al., 2013 and Zhang et al., 2013, report that the highest mechanical properties (hardness and bond strength) of Cr coatings were achieved at a temperature of 300°C. Milosev et al., 1996, proved that the CrN coatings with thickness of 0.34 µm deposited on the steel substrate at 200°C were dense, smooth, and homogeneous.

The reducing in the energy consumptions of technological processes is a desirable goal of individual branches of industry (Kukielka, 2016; Ulewicz, 2015, Szataniak et al. 2014). This reality must also be respected in the area of processes of heat treatment and coating of tool steels. The process of CrN coating of tool steels is carried out in the chain of hardening, tempering and coating, while tempering and coating temperatures are identical for low temperature coating. With regard on this fact, the aim of the work is to experimentally verify the technological process of CrN coating only in the hardening and coating chain (excluding tempering).

3. Experiments

The base materials used in the experiments was low-alloy 90MnCrV8 tool steel. This steel was obtained in the form of a bar with 30 mm in diameter, in soft annealed state. Verification of the chemical composition, chemical analysis, was performed by the spark emission on a SPECTROMAXx device. The experimental material was subsequently heat treated in a two-chambered vacuum furnace ROVA-8.10.6,5/13G (firm Voestalpine HPM, Martin, Slovakia) by the processes: austenitization at 820°C for 40 minutes, cooling in the oil at 120°C, low-tempering at 180°C for 120 minutes, followed by cooling in air. After the above-mentioned heat treatment, the steel was sandblasted and CrN coating was applied. The CrN coating was prepared by arc evaporation from Cr target (130 mm in diameter, 99.5 at. %) in STATON PD300-G2 deposition equipment (company STATON s. r. o., Turany, Slovakia). Material was placed on the heated biased rotatable holder. The distance of the materials from Cr target was set to 200 mm. Prior to deposition, chamber was evacuated to the base pressure of 2×10^{-3} Pa, followed by heating for 4000 s to reach temperature of 180°C. The material surface was pre-treated using Ar etching and metal ion etching process. After plasma etching CrN coating was deposited by using one Cr target in a nitrogen atmosphere of 0.3 Pa for 2300 s. The material bias was

at the potential -200 V during the deposition. The total time of coating together with cleaning process was 120 minutes (this process, Hardening + Tempering + Coatings is marked as HTC). In the second case, the technological process consisted only of the above described hardening and coating, but without tempering (this process, Hardening + Coating is marked as HC).

The microstructure after HTC and HC was studied with the use of standard light microscopy (Carl Zeiss Axio Observer Z1m) and by a scanning electron microscopy (TESCAN VEGA-II). The retained austenite was determined by RTG diffractometric analysis (Proto iXRD device using the CrK α radiation, $\lambda = 2.2910 \times 10^{-10}$ m, collimator with an irradiated area 2 mm², Average Peak Method). After HTC and HC the specimens for the tensile tests and Charpy tests were machined. The round cross-sectional specimens of diameter $d = 6$ mm were used per the EN 10002-1 standard. Three specimens for tensile tests were used, INSTRON testing machine and testing parameters, $T = 20^\circ\text{C} \pm 3^\circ\text{C}$, loading range $F = 0 - 20$ kN, the strain rate $\epsilon_m = 10^{-3}\text{s}^{-1}$. For Charpy test, three specimens per the EN 10045-1 were used. PSW 3000 Charpy equipment and testing parameters, $T = 20^\circ\text{C} \pm 3^\circ\text{C}$, nominal energy 300 J. The fracture surfaces were evaluated by SEM. The Rockwell hardness HRC, was measured on an RR-10/AQ hardness tester, average value from five measurements is given. The Vickers hardness HV1, was measured on a microhardness Zwick/Roell ZH μ tester in the direction from the surface to the center of the samples. The microhardness H_{IT} and Young's modulus E_{IT} of CrN coating were measured using nanoindenter (Anton Paar NHT2) equipped with a Berkovich diamond tip by applying a load of 2 mN. In order to avoid a material influence on hardness, an indentation depth was set to 10 % of the film thickness. H_{IT} and E_{IT} were determined via Oliver and Phaar method. Presented results are averages from total of 16 measurements (4x4 pattern).

The residual stresses state (HTC and HC) was evaluated by the X-ray diffraction measurement. The ProtoiXRD device was used for the measurements, using the CrK α radiation with an irradiated area of 1 mm². The diffraction signal from $\{222\}\alpha$ planes was collected at $2\theta = 156.9^\circ$. The measurements were carried out using the $\sin^2\psi$ method, with nine inclinations between $\pm 39^\circ$. The measurements were carried out in axial ($\varphi = 0^\circ$) and tangential ($\varphi = 90^\circ$) direction. To obtain the depth profile of the residual stresses distribution, the surface was gradually removed by electrolytic polishing.

Potentiodynamic polarization (PD) tests were employed in order to monitor corrosion resistance of base material and investigated coatings with standard 3-electrode connection. Measurements were performed using Biologic VSP-300 laboratory potentiostat (Kajánek et al., 2017). Samples were connected as working electrodes, Pt mesh as counter electrode and saturated calomel electrode served as reference electrode (Kajánek et al., 2017; Džiková et al., 2021). PD tests were carried out in 3.5% NaCl solution after 15 minutes of open circuit potential (OCP) stabilization. Samples were polarized in range of -200 to +500 mV vs. OCP with step rate of 1 mV/s. Measured data were analysed using Tafel analysis using EC-Lab V11.50 software providing values of corrosion potential

(E_{corr}), corrosion current density (i_{corr}), Tafel slopes (β_a , β_c) and corrosion rate was calculated (r_{corr}) (Jambor et al., 2020).

4. Results and discussion

The chemical composition, Tab. 1, proved that experimental base material was low-alloyed tool steel in accordance with the material standard (STN 41 9313, DIN EN 90MnCrV8). From the results of tensile tests (Fig. 1), it can be seen that no significant differences were noted between the technological processes of HTC and HC. The mechanical properties, Table 2, correspond to the values stated in the works (Fremunt et al., 1994; Beneš, 1968).

Table 1. Chemical composition of experimental base material (in wt. %)

Steel grade	Chemical composition			
	C	Si	Mn	Cr
Exper. material	0.949	0.289	2.25	0.236
STN 41 9313	0.8 - 0.9	0.15 - 0.35	1.75 - 2.1	0.2 - 0.4
DIN EN 90MnCrV8	0.85 - 0.95	0.10 - 0.40	1.9 - 2.1	0.2 - 0.5
Steel grade	P	S	V	Fe
Exper. material	0.0153	0.0079	0.0798	balance.
STN 41 9313	max. 0.035	max 0.035	0.1 - 0.2	balance.
DIN EN 90MnCrV8	max. 0.035	max 0.035	0.05 - 0.15	balance.

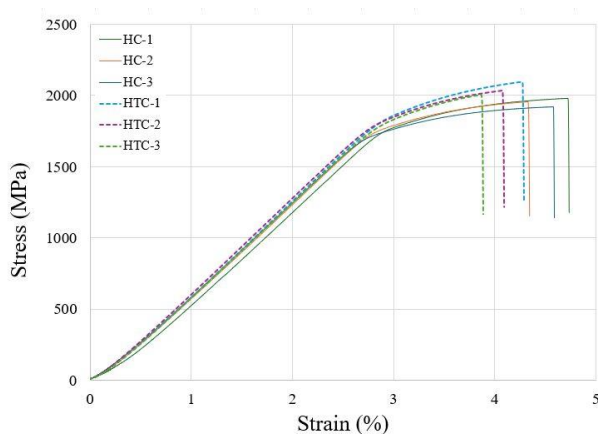


Fig. 1. Tensile testing results of HTC and HC technological processes

Table 2. Mechanical properties of the experimental base material, HTC and HC process

Process	Yield point (MPa)	UTS (MPa)	Elongation (%)	Impact toughness (J)	Hardness HRC
HTC	1879	2045	1.02	6.7	59
HC	1788	1951	1.56	6.6	59

The results (average values) of the HRC (59 HRC, HTC, HC) and HV1 (727 HV1 – HTC, 715 HV1 – HC) hardness measurements are listed in Tab. 2 and shown in Fig. 2. The

hardness values HTC and HC processes are practically the same, correspond to the use of steel and are in accordance with the author's works (Skočovský et al., 2015; Fremunt et al., 1994; Beneš, 1968). The Vicker's method is more accurate, more sensitive, and the results correspond well with the results of mechanical properties, Tab. 2.

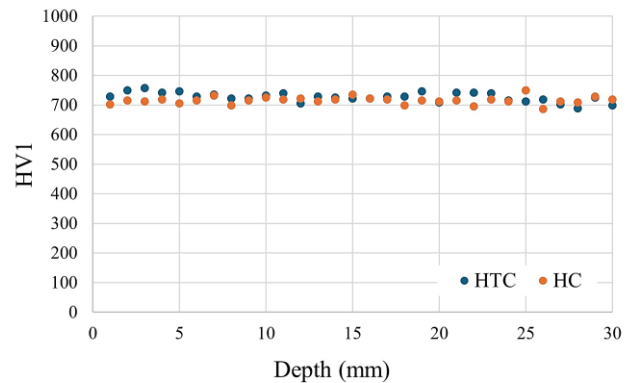
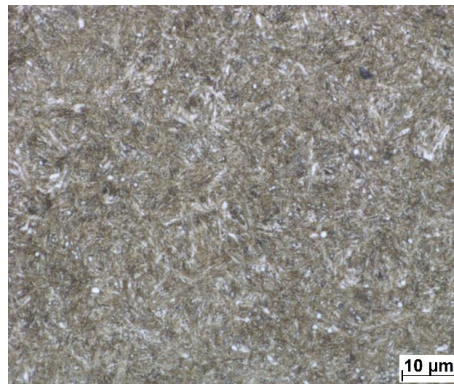


Fig. 2. Hardness HV1 after HTC and HC technological processes

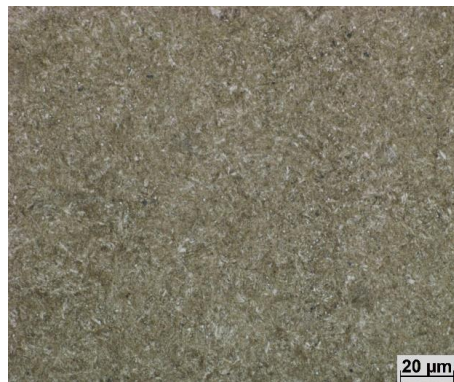
The microstructure of the experimental base material after the HTC and HC technological processes was created by low-tempered martensite, with fine, evenly distributed globular carbides and retained austenite, Jech, 1983, Fig. 3. The detected content of retained austenite in both cases was less than 5 %. The stated content of retained austenite is satisfactory, because retained austenite is a soft phase and is undesirable for most tools. With the growth of retained austenite in the matrix, the hardness and yield point decrease (Fremunt et al. 1994). The applied CrN coating had a thickness 0.3 μm , hardness value $H_{\text{IT}} = 20 \pm 2$ GPa and the elasticity modulus, $E_{\text{IT}} = 264 \pm 20$ GPa. The characteristics of the CrN coating are in accordance with the works (Milosev et al. 1996; Vicen et al. 2023) and due to the high values H_{IT} , E_{IT} represent good wear resistance.



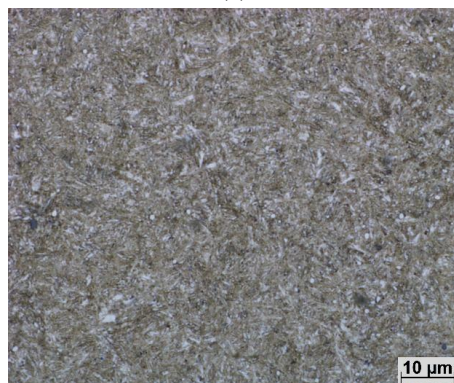
(a)



(b)



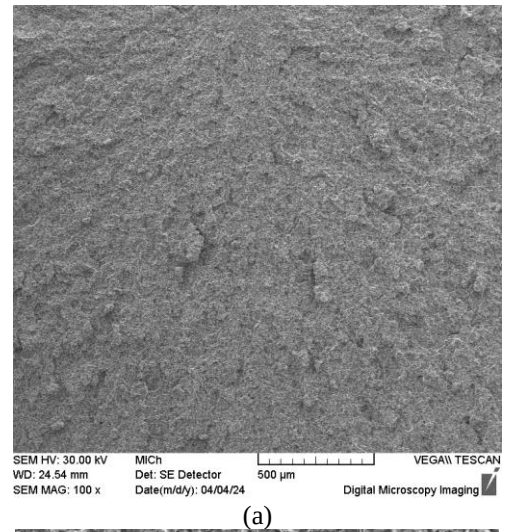
(c)



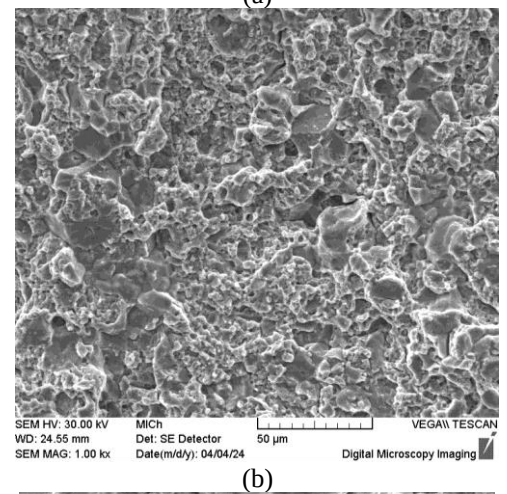
(d)

Fig. 3. Microstructure of experimental base material after HTC (a), (b), and HC (c), (d) technological processes, etc. 2 % Nitral

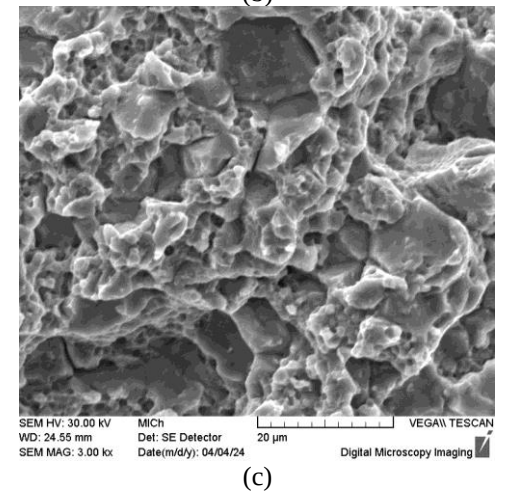
The fracture surfaces after carried out the Charpy test are shown in Fig. 4. The fracture surfaces show the same type of failure, mixed transcrystalline (60 %) and intercrystalline (40 %) failure. Surfaces are dominated by typical signs of failure with dimple morphology, the remaining approx. 40 % is intercrystalline brittle fracture. The mentioned type of failure is characteristic for the vast majority of heat treatment of tool steels (Fremunt et al., 1994).



(a)



(b)



(c)

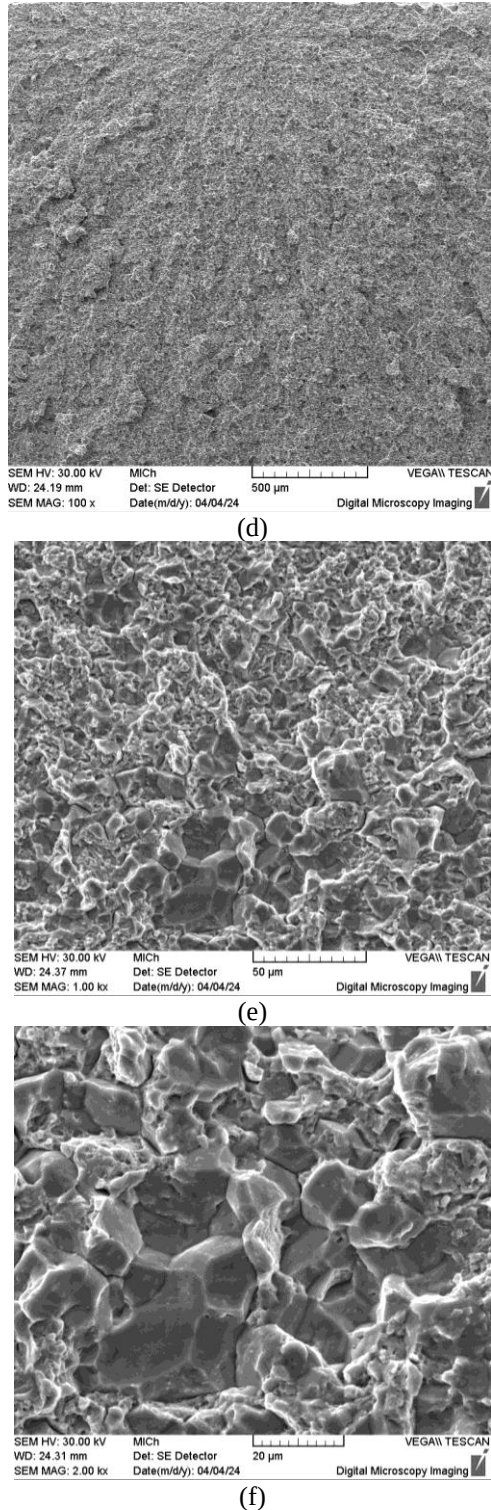


Fig. 4. Fracture surface after HTC (a), (b), (c) and HC (d), (e), (f) technological processes, SEM

The near-surface residual stresses values after HTC and HC technological processes are indicated in the Fig. 5. As it can be seen from the results, the values of residual stresses are generally very low (<100 MPa) in both HC and HTC process. Also, there is no significant difference in the axial and tangential direction of the stresses with respect to the specimen's

axis, thus the near-surface residual stress state after both processes can be considered as identical (Trško et al., 2020; Vicen et al., 2020; Vicen et al., 2022 and Vicen et al., 2023).

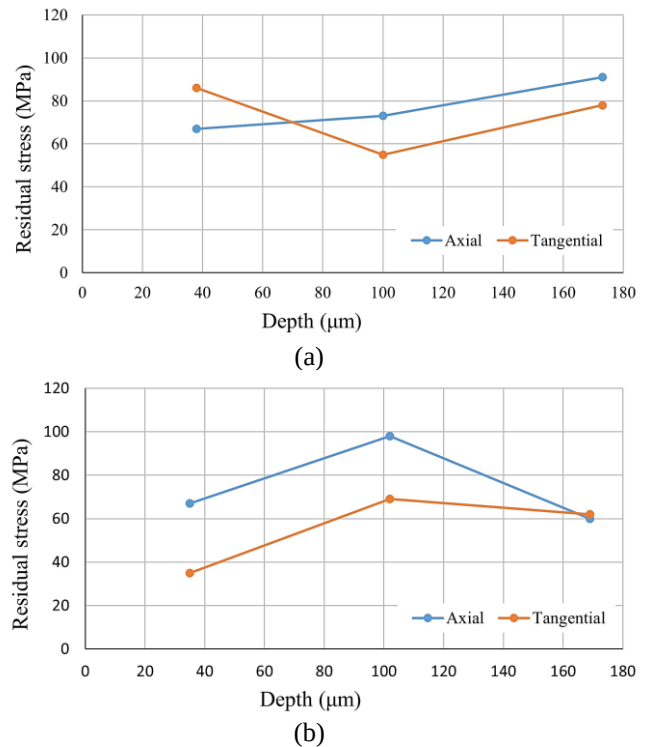


Fig. 5. Residual stresses after HTC (a) and HC (b) technological processes

Resulting electrochemical characteristics measured in 3.5 % NaCl obtained by Tafel analysis of potentiodynamic curves clearly showed improvement of corrosion resistance for both coatings compared to non-coated low-alloy 90MnCrV8 tool steel counterparts Fig. 6 and Tab. 3. Shift of corrosion potential E_{corr} towards positive direction, more notable in case of HTC, is observed indicating improved thermodynamic stability of both created coatings (Sovik et al., 2023).

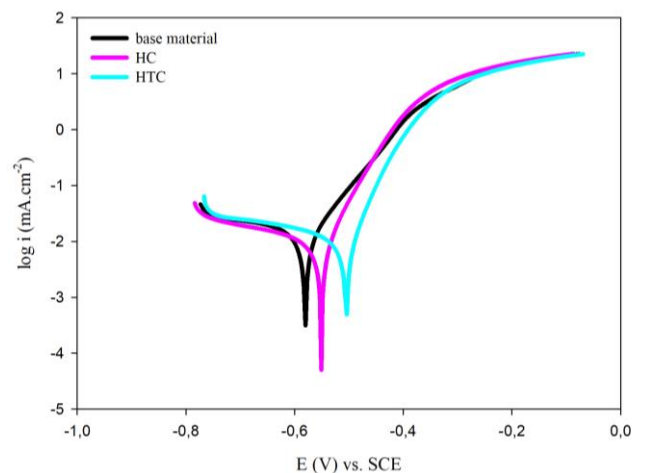


Fig. 6. Potentiodynamic curves measured in 3.5 % NaCl

Table 3. Electrochemical characteristics measured in 3.5 % NaCl

Pro- cess	E_{corr} (mV)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	β_a (mV/dec.)	β_c (mV/dec.)	r_{corr} (mm/y)
base	-568	13.9	85	568	0.07
HC	-551	9.4	65	453	0.04
HTC	-504	10.5	55	454	0.05

Additionally, corrosion current density i_{corr} was reduced for both coatings pointing to suppressed kinetic of corrosion processes occurred on the surface. Moreover, presented i_{corr} values show that there is no significant difference between created coatings (Vicen et al., 2023). Since the i_{corr} values are in direct relation with corrosion rate r_{corr} , it can be summarized that corrosion rate of low-alloy 90MnCrV8 tool steel was decreased by preparation of examined coatings. Results of electrochemical measurements can be explained by sufficient anti-corrosion barrier demonstrated by both coatings which subsequently inhibited penetration of aggressive Cl^- ions towards low-alloy 90MnCrV8 tool steel. No significant difference between coatings means that application of coating right after hardening procedure can fully substitute process of tempering without compromising the corrosion resistance of the low-alloy 90MnCrV8 tool steel.

5. Conclusion

1. **Energy Efficiency:** The research demonstrates that the hardening-coating (HC) process is a viable energy-efficient alternative to the traditional hardening-tempering-coating (HTC) process for applying CrN coatings on low-alloy tool steel. By eliminating the tempering phase, significant energy savings can be achieved without compromising the quality of the coating.
2. **Quality Maintenance:** Comparative analyses of microstructural, mechanical, and corrosion properties reveal that the HC process maintains high-quality standards similar to the HTC process. Both methods produce a low-tempered martensitic structure with fine, evenly distributed carbides and minimal retained austenite, ensuring the tool steel's desirable hardness and toughness.
3. **Mechanical Properties:** The mechanical properties, including tensile strength, hardness, and impact toughness, are consistent across both processes. The hardness values measured in both HTC and HC processes indicate excellent wear resistance, essential for extending the tool's service life.
4. **Corrosion Resistance:** Potentiodynamic polarization tests confirm that the CrN coatings applied via the HC process provide comparable corrosion resistance to those applied via the HTC process. The corrosion

potential shifts positively, indicating an improvement in corrosion resistance for both coating methods.

5. **Structural Integrity:** Fractographic and residual stress analyses indicate that both HTC and HC processes produce fracture surfaces and residual stress profiles that are largely similar, with no significant differences in the axial and tangential directions of stress. This suggests that the structural integrity of the tool steel is maintained in both processes.
6. **Sustainability:** This study contributes to sustainable manufacturing practices by presenting an energy-efficient method that does not compromise the tool steel's performance. The HC process aligns with the industry's goals of reducing energy consumption while maintaining high-quality standards in production.
7. **Practical Application:** The findings support the practical application of the HC process in industrial settings, providing a cost-effective and environmentally friendly alternative to traditional coating methods. The adoption of this process can lead to significant improvements in energy savings and overall production efficiency. The hardening-coating process presents a promising avenue for enhancing the sustainability of tool steel manufacturing, achieving substantial energy savings without sacrificing the quality and performance of CrN coatings. This approach aligns with the industry's ongoing efforts to improve production efficiency and reduce environmental impact.

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