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INFLUENCE OF CORROSION ON THE FATIGUE RESISTANCE OF A CARBON STEEL WITH Ti AND Cr NITRIDES BASED COATINGS

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Abstract: The quality of components and structures is directly related to degradation processes such as corrosion and fatigue that are concomitant phenomena during their operation. One of the options for increasing the quality of components and structures is the use of appropriate surface coating. In this paper is presented an experimental investigation of the corrosion influence on the fatigue resistance of the carbon steel with coatings based on Ti and Cr nitrides. The experimental work included determination of the chemical composition of the base material, heat treatment, microstructure evaluation, mechanical properties measurement, coating depositions, corrosion test and the fatigue tests by rotation bending of the deposited layers. The corrosion and fatigue tests have shown that the coating provides improvement of the corrosion resistance and that there was no reduction of the fatigue resistance.
Keywords: carbon steel, coatings, corrosion, fatigue resistance

1. INTRODUCTION

Degradation mechanisms, such as fatigue, corrosion and wear, are typically initiated at the surface of components and structures. Therefore, when assessing the reliability, safety, and service life of those components, the increased attention must be paid to their surfaces' properties. One of the effective approaches of improving the functional performance of components and structures is the application of suitable coatings to their surfaces. Technologies, based on physical and chemical deposition of coatings, are commonly used to produce the protective layers.

The main feature of physical deposition processes (PVD – Physical Vapour Deposition) is the condensation of vaporized coating material onto the surface of the substrate. The resulting coatings, such as TiN, CrN, or multilayer systems like CrN/TiN, are characterized by high hardness, excellent wear resistance, chemical and thermal stability, and improved resistance to fatigue and corrosion (Madej et al., 2015, Moravčík and Hazlinger, 2017, Grainger and Blunt, 1998). An important group of PVD layers are the diamond like coatings (DLC), which significantly decrease the coefficient of friction and thus reduce friction-related losses (Madej et al., 2014, Piotrowska et al., 2020). In the study (Shiozawa and Oshima, 1990), authors investigated the fatigue behavior of steel coated with a TiN layer. Rotating bending fatigue tests were conducted on round specimens made of 0.37% carbon steel (JIS S35C). The results revealed a 16–23% increase in fatigue strength for the TiN-coated specimens compared to the uncoated base metal. This improvement was attributed to the retardation of crack initiation, caused by the restriction of dislocation movement near the substrate surface due to the presence of the hard thin TiN film. Similarly, (Wilson et al., 1993) carried out the rotating bending fatigue tests on smooth specimens in air at a speed of 5700 rpm, for up to 10^6 cycles or until failure. Their findings showed that TiN, CrN, and duplex coatings not only did not reduce the fatigue resistance, but in fact enhanced the S–N curves and increased the endurance limit. The coatings were deposited on both steel and grey cast iron substrates. According to (Baran et al., 2016), the fatigue resistance of coated materials increased by approximately 10 % as a result of the coating process, and this improvement was only marginally affected by variations in deposition parameters. In another study, authors examined the influence of a 1.4 µm thick TiN coating on the fatigue properties of AISI 316L stainless steel, (Puchi-Cabrera et al., 2004). The results demonstrated a 22 % (60 MPa) increase in the fatigue limit compared to the uncoated substrate. It has been concluded that the increase in fatigue properties of the coated substrate is associated mainly with the compressive residual stresses present in the coating. In the study by (Kim et al., 2003), the influence of the intrinsic properties of coatings was investigated, specifically of TiN, TiCN, and TiAlN films influence on the fatigue behavior of Cr-Mo-V rotor steel. The results demonstrated that the fatigue strength of coated specimens exceeded that of the uncoated substrate, particularly in the high-cycle fatigue region. This improvement was primarily attributed to the retardation of crack initiation in the substrate, caused by the presence of hard coating layers. Similarly, (Su et al., 1998) conducted experiments using an axial tension–compression testing machine to evaluate the tensile and fatigue performance of TiN-coated materials with coating thicknesses of 3.5 µm and 7.0 µm, compared to uncoated samples. The application of TiN films produced a positive effect, resulting in an enhancement of the endurance limit by 20 to 40 MPa. In the work (Park et al., 2007), a novel duplex coating process, combining plasma carburization with CrN coating, was applied to Ti-6Al-4V alloy to study its effect on the fatigue life. The duplex-coated specimens exhibited significantly improved fatigue life compared to untreated samples. The authors attributed this enhancement to the retardation of fatigue crack initiation due to the presence of hard and strong surface layers. According to (Bouzakis et al., 2008), the film fatigue endurance stress of the investigated coatings was found to be adequate for practical applications. Furthermore, (Yonekura et al., 2015) deposited the Cr/CrN multilayer coatings, with varying numbers of bilayers, on Ti-6Al-4V alloy substrates and performed the tension–tension fatigue tests. Their findings revealed that the fatigue strength of multilayer coatings was higher than that of the single-layer CrN coatings, confirming the beneficial effect of multilayer structures on fatigue performance.

In addition to improving the wear resistance, thermal stability, hardness, and overall mechanical properties, TiN, CrN, and CrN/TiN coatings are applied to carbon steels as well, to enhance their corrosion resistance. Titanium nitride (TiN) is a hard, ceramic, golden-colored material widely used as a wear-resistant coating in industrial, biomedical, and aerospace applications. However, a limitation of TiN coatings is their susceptibility to porosity, which varies depending on the deposition technique employed. Such porous defects, including pinholes and microcracks, can enable penetration of electrolyte, leading to localized corrosion (typically pitting or crevice corrosion) at the steel-coating interface, particularly in aggressive environments (Souto and Alanyali, 2000, Datta et al., 2018). The authors of (Lou et al., 2021) reported that the TiN coating process, performed by physical vapor deposition (PVD), resulted in coatings with low thickness and a high density of defects, which in turn led to reduced resistance to localized corrosion, as confirmed by electrochemical impedance spectroscopy (EIS) measurements. In contrast, (Fazel et al., 2024) demonstrated that the use of cathodic arc deposition can eliminate or significantly reduce the coating defects, such as pits and macro/microparticles, thereby producing TiN coatings with superior corrosion performance. Chromium nitride (CrN) is a dense, hard, and corrosion-resistant ceramic material commonly used as a protective coating in high-wear and high-temperature applications, such as in the mold and die industry, automotive components, food processing equipment, aerospace, and marine environments. The excellent corrosion resistance of CrN coatings is primarily attributed to their dense microstructure, which limits electrolyte penetration, and to the formation of a protective chromium oxide (Cr_2O_3) passive layer, which enhances the oxidation resistance and provides protection against chemical attack. The superior resistance of CrN coatings to chloride-induced localized corrosion has been confirmed by (Toshioka and Nishimoto, 2020; Subramanian and Jayachandran, 2011) through electrochemical measurements, including potentiodynamic polarization and electrochemical impedance spectroscopy (EIS), conducted in a 3.5 wt.% NaCl solution. The CrN/TiN multilayer coating combines the advantages of both constituent materials, exhibiting enhanced mechanical strength and toughness, improved adhesion and crack resistance, as well as greater durability and a reduced risk of coating failure under mechanical stress (Choi et al., 2009; Barkovskaya et al., 2011). The excellent pitting corrosion resistance of CrN-based coatings is attributed to their dense microstructure, which minimizes the penetration of aggressive solutions, particularly when the outer CrN layer forms a protective Cr_2O_3 passive film (Choi et al., 2009; Ou et al., 2016). According to (Fazel et al., 2024), the outstanding corrosion performance of CrN/TiN multilayer coatings can also be ascribed to the large number of interfaces and the smooth surface morphology with few macroparticles, which together enhance the barrier effect against the corrosive media. Furthermore, (Rabadzhiyska et al., 2018) confirmed the suitability of this multilayer coating for biomedical applications through electrochemical testing in physiological solutions.

Degradation processes of materials caused by corrosion and fatigue are dominant in the field of durability of machine components, their safety and reliability. There are not many scientific publications dealing with the effect of corrosion on fatigue resistance of carbon steels with Ti and Cr nitrides coatings. Therefore, the aim of the experiments was to determine the influence of corrosion of the high-quality, medium carbon steel with coating based on Ti and Cr nitrides on its fatigue resistance in the area of high number of loading cycles.

2. EXPERIMENTAL METHODS

The base material (substrate), used in this investigation, was the high-quality, medium carbon, C55 steel obtained in the form of bars of 16 mm in diameter. Verification of the chemical composition, i.e., the quantitative chemical analysis, was performed by the spark optical emission spectroscopy on a SPECROMAXx device. The experimental material was subsequently heat treated as follows: austenitization at 820 °C ± 5 °C, 30 minutes; cooling in the Durixol V70 oil to 20 °C ± 5 °C; tempering at 450 °C ± 5 °C, 120 minutes, followed by cooling in the air. After the heat treatment, the microstructure was studied by a NEOPHOT 32 light microscope with the NIS Elements 4.20 evaluation system, and by VEGA TESCAN electron microscopy. Hardness of the specimens was determined by the Rockwell hardness test (HRC) on an RR-10/AQ tester. The specimens for the tensile tests were machined. For the tensile tests, the round cross-section specimens of a diameter of 10 mm were used according to the EN 10002-1 standard. The tensile tests were carried out on a ZWICK Z050 testing machine at an ambient temperature T of 20 ± 5 °C, with the loading force F in interval of 0 to 20 kN and the strain rate ε_m of 10^{-3} s^{-1}. The coatings were deposited to a polished surface of the specimens. The surface of the specimens was ionically cleaned before the coating to remove impurities. The deposition of the coatings on the base material, on the round cross-section specimens of a diameter of 6 mm, was executed by a combined plasma process of plasma enhanced chemical vapor deposition and physical vapor deposition, particularly the DC magnetron sputtering (Drábik et al., 2018). After the coating, the specimens were placed in a 3.5 % NaCl solution for 72 hours (Fig. 1). After that period, the specimens were removed from the solution and rinsed with water and ethanol. Subsequently, the specimens were subjected to the fatigue test.

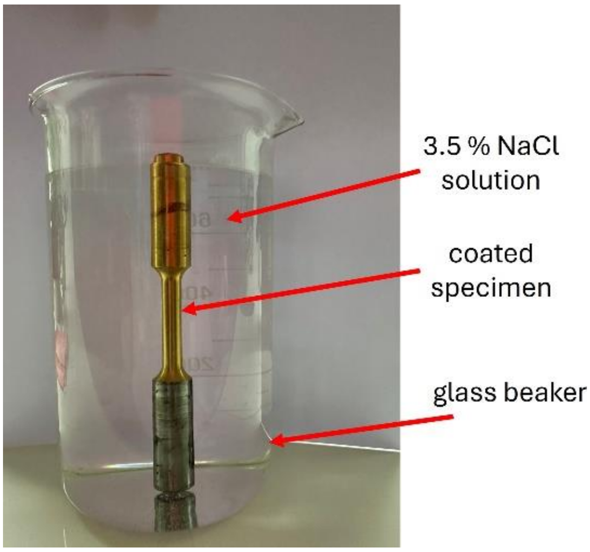
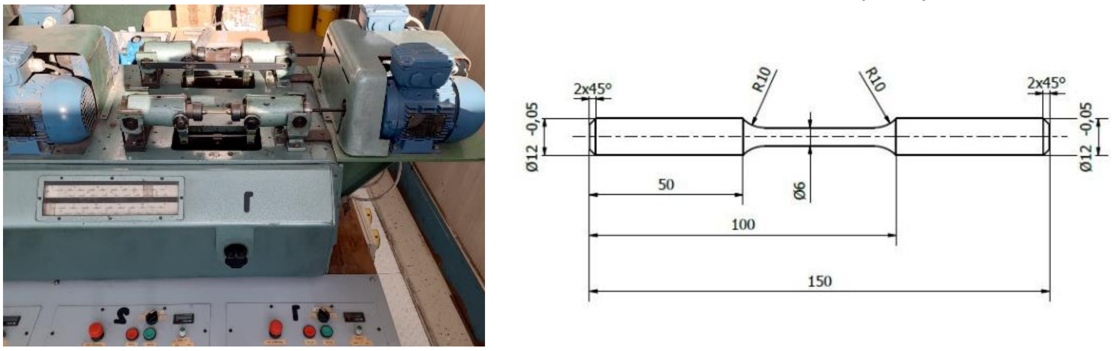


Fig. 1. Immersion corrosion test, 3.5 wt. % NaCl solution

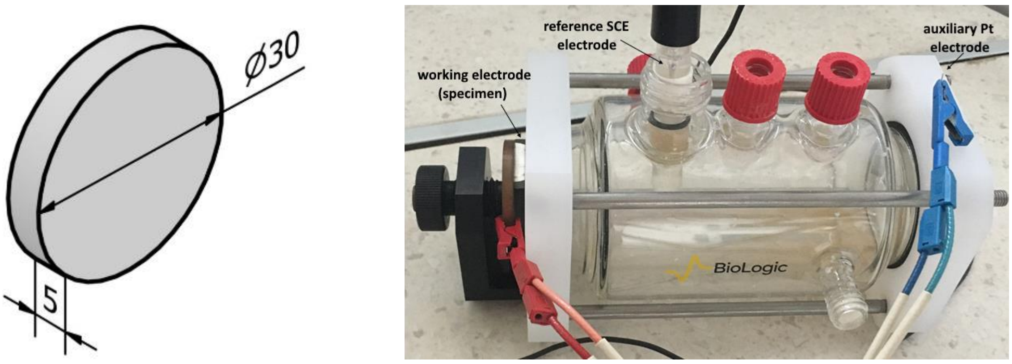
Fatigue tests were performed on a ROTOFLEX testing equipment, by rotating bending, at a loading frequency of 30 Hz, room temperature of 20 ± 2 °C, Fig. 2a. The number of loading cycles N, needed to determine and compare the fatigue properties of C55 carbon steel with and without coatings, was 2.5×10^6 cycles (for the base material also $N > 10^7$ cycles). With regard to the recommended number of 3 specimens for fatigue resistance at stress levels (Moravčik and Hazlinger, 2017), 5 specimens (Fig. 2b) were used for the base material and for the base material with each type of coating.



a) b)

Fig. 2. ROTOFLEX testing equipment, a) rotation bending loading, b) specimens for fatigue tests

The electrochemical properties of the coated surfaces and the as received surface were evaluated, by potentiodynamic polarization (PP) carried out at the temperature of 20 ± 2 °C in 3.5 wt. % sodium chloride solution on circular specimens (diameter 30 mm, thickness 5 mm), Fig. 3a. The test was performed in the conventional three-electrode cell system with a saturated calomel reference electrode (SCE) and a platinum auxiliary electrode (Pt) using BioLogic corrosion measuring system, with PGZ 100 measuring unit, Fig. 3b. The time for potential stabilization between the specimen and the electrolyte was set to 10 min. The exposed area of a specimen was 100 mm^2. The PP measurement was recorded at the sweep rate of 1 mV/s, a potential scan range applied was between -0.3 and 1.0 V, vs. open circuit potential (OCP). At least three experiments were repeated for each surface condition (as received; TiN; CrN; CrN/TiN coatings) and the representative curve was selected.



a) b)

Fig. 3. PP measurement, a) specimen, b) the three-electrode corrosion cell

3. RESULTS AND DISCUSSION

The chemical composition and mechanical properties of experimental base material, high quality, medium carbon, C55 steel, are presented in Tables 1 and 2, respectively. The microstructure after the heat treatment (quenching and high tempering) was sorbitic, formed by a ferritic matrix and globular cementite in accordance with the work (Jech, 1983).

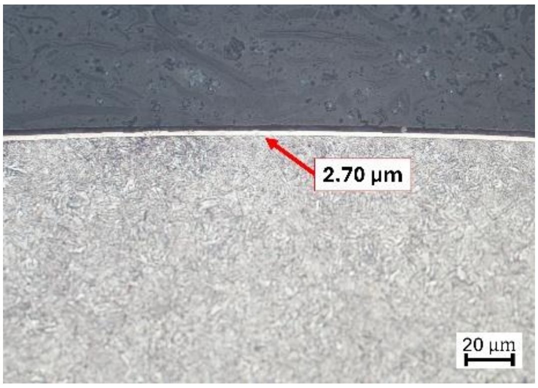
Table 1
Chemical composition of experimental material (wt. %)

C	Si	Mn	Cr	P	S	Mo	Fe
0.597	0.398	0.860	0.345	0.037	0.014	0.193	ball.

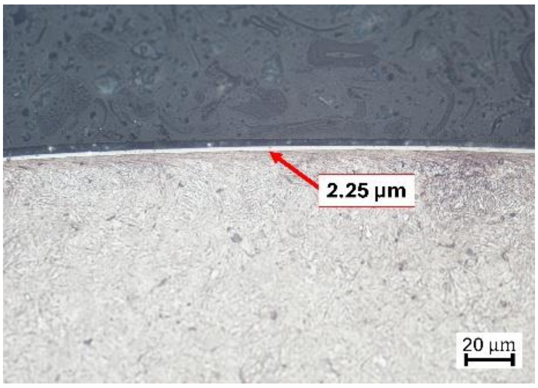
Table 2
Mechanical properties of experimental material

Ultimate tensile strength (MPa)	Yield point (MPa)	Elongation (%)	HRC (-)
1316	1150	8	42

The TiN, CrN and CrN/TiN coatings, deposited on the base material, observed on the transversal section through the coatings, are shown in Fig. 4. The thickness of the TiN coating was 2.70 µm, CrN coating 2.25 µm, and in the case of the CrN/TiN coating 2.55 µm.



a)



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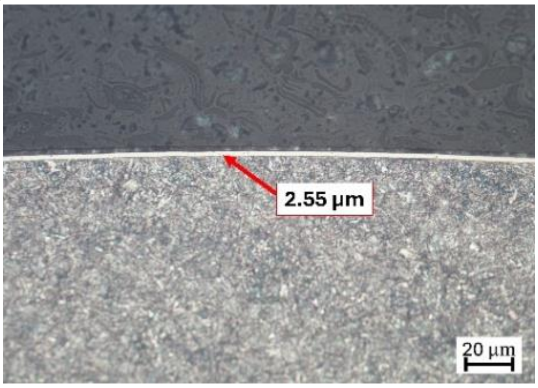

c)

Fig. 4. The thickness of the coatings, a) TiN, b) CrN, c) CrN/TiN deposited on the base material, etch. 3 % Nital

The representative potentiodynamic polarization (PP) curves for the various coated C55 steel surfaces (TiN, CrN, CrN/TiN coatings), as well as for the as received surface, are presented in Fig. 5. All the investigated parameters, i.e., E_{corr} (corrosion potential), i_{corr} (corrosion current density) and v_{corr} (corrosion rate) were obtained as a result of the Tafel extrapolation of the representative curves, using the EC-LAB software. The corrosion potential E_{corr} is the equilibrium potential at which the rate of oxidation is equal to the rate of reduction. On the polarization curve, E_{corr} can be localized as the potential of the transition from the cathodic to the anodic branch of the curve. The E_{corr} expresses the level of the thermodynamic stability of a material in the applied solution. The higher E_{corr} value means the higher thermodynamic stability of the corrosion system (Hadzima and Liptáková, 2008). The corrosion current density i_{corr} reflects the kinetic aspect of the corrosion process. That is the current density reached at the corrosion potential (when the

current density of the oxidation equals the current density of the reduction) (Khoshnaw and Gubner, 2020). The corrosion rate v_{corr} is in the direct quantitative relationship with corrosion current density based on Faraday's law (the higher i_{corr} means the higher v_{corr}). The values of PP parameters are listed in
Table 3.

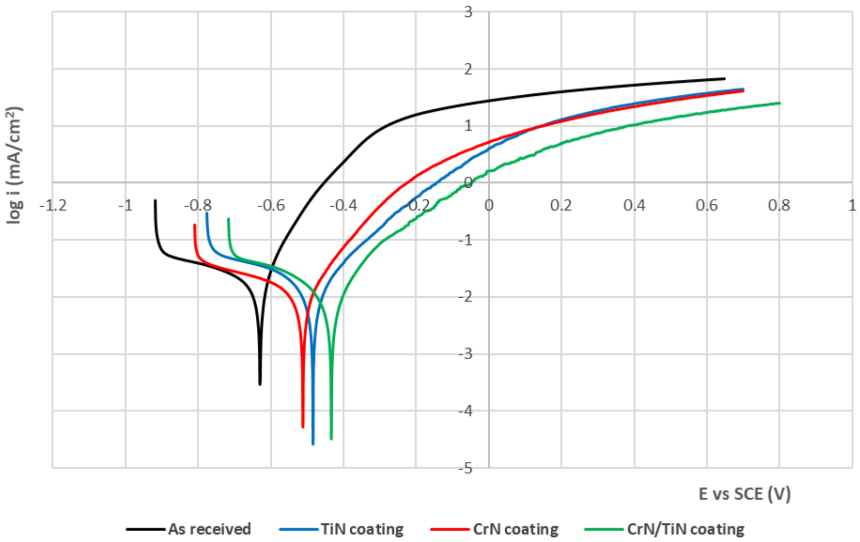

Fig. 5. Potentiodynamic polarization curves in 3.5 wt. % NaCl solution

Table 3
Values of the PP parameters of the tested surfaces in 3.5 wt. % NaCl solution

Type of surface	**Corrosion Potential E_{corr} (V vs SCE)**	**Corrosion current density i_{corr} (μA/cm^2)**	**Corrosion rate v_{corr} (mm/year)**
As received	-0.630	14.428	0.168
TiN coating	-0.484	10.197	0.118
CrN coating	-0.512	10.557	0.123
CrN/TiN coating	-0.435	10.261	0.119

As can be seen from Fig. 5, all the presented PP curves have a similar shape, but with significant differences in the PP values, especially between the as received and the coated surfaces. All the tested coatings caused the marked improvement to the corrosion resistance, both from thermodynamic (E_{corr}) and from the kinetic (i_{corr}, v_{corr}) point of view. According to the E_{corr} value, the highest thermodynamic stability was reached for the combined CrN/TiN coatings. This result could be related to their dense microstructure preventing a penetration of the aggressive solution into the coating as well as to the smooth surface. These two features both led to a combining of CrN and TiN coatings advantages (Fazel et al., 2024; Choi et al., 2009). Similar behavior of CrN/TiN coating in potentiodynamic polarization (in 3.5 wt. %) was confirmed also by (Ou et al., 2016). The i_{corr} and the corresponding v_{corr} values of the coated surfaces are close (v_{corr} from 0.118 to 0.123 mm/year) what points to substantially higher corrosion resistance compared to the uncoated C55 surface (v_{corr} = 0.168 mm/year).

The results of the fatigue tests are shown in Fig. 6 (only for the selected, comparative number of N = 2.5x10^6 cycles). Experimentally obtained fatigue test results are in accordance with the works of (Baran et al., 2016; Puchi-Cabrera et al., 2004; Bouzakis et al., 2008). For the TiN, CrN coatings, as compared to the base material (Fig. 6), σ_o was higher by 25 MPa. Those authors reported the σ_o values of 20 to 60 MPa or 10 to 23 % increase. In the case of the duplex CrN/TiN coating, σ_o was significantly higher with respect the base metal, for 150 MPa.

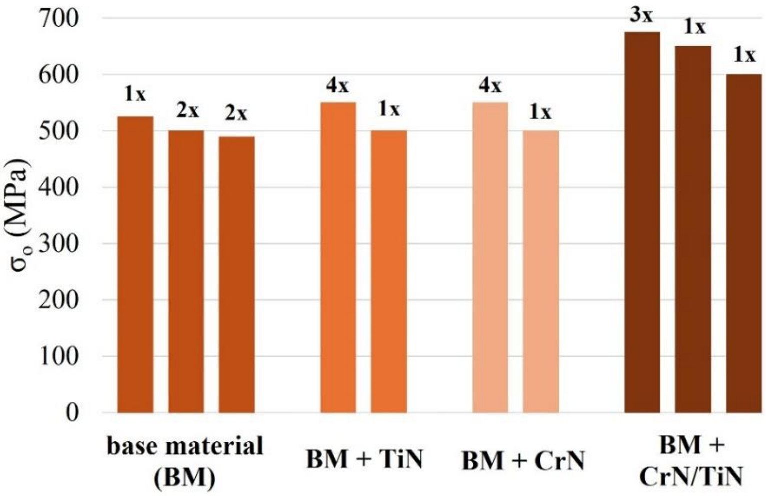

Fig. 6. Comparison of fatigue results base material (BM) and BM with TiN, CrN and CrN/TiN coatings, σ_o at number of N = 2.5x10^6 cycles

The authors (Wilson et al., 1993; Yonekura et al., 2015) reported a positive effect of multilayer coatings on fatigue resistance. In general, it can be concluded that the mentioned coatings, with their properties, inhibit the development of degradation fatigue mechanisms, the initiation on fatigue cracks in the base material. The fatigue test results demonstrated significant corrosion resistance of the coatings (Datta et al., 2018; Lou et al., 2021; Fazel et al., 2024).

4. CONCLUSIONS

Based on the experimental work carried out to determine the influence of corrosion attack on high-quality, medium, C55 steel with coating based on Ti and CrN nitrides on its fatigue resistance, it is possible to draw the following conclusions:

- The tested TiN, CrN and CrN/TiN coatings provided significant improvement in corrosion resistance, compared to uncoated C55 steel.
- The obtained values of the corrosion rate V_{corr} were the following: for the TiN coating - 0.118 mm/year; for the CrN coating - 0.123 mm/year; for the CrN/TiN coating - 0.119 mm/year and for the uncoated C55 steel – 0.168 mm/year.
- The fatigue test results of the coated C55 steel were higher as compared to the uncoated material.
- The obtained fatigue resistance σ_o values of the TiN and CrN coatings were higher by 25 MPa, as compared to the base material.
- The multilayer CrN/TiN coating exhibited the higher fatigue resistance, as compared to the TiN and CrN coatings.
- The fatigue resistance σ_o values for the CrN/TiN coating was higher for 150 MPa.

- The corrosion attack of C55 steel with TiN, CrN and CrN/TiN coatings did not reduce its fatigue resistance.
- The obtained results are important from the point of vies of application in engineering practice, in areas of applications as shafts, gears, and highly stressed parts of motor vehicles.
- The following research, related to engineering applications, will focus on the effect of corrsion of carbon steels with Ti and Cr nitride coatings on fatigue resistance in the ultra-high number of loading cycles range.

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