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# WEAR AND SOLDERING PERFORMANCE OF BARE, NITRIDED AND PVD COATED HOT-WORKING TOOL STEEL IN CONTACT WITH Al-ALLOY CASTING

**Pal TEREK[1,*], Lazar KOVACEVIC[1], Vladimir TEREK[1], Zoran BOBIC[1], Branko SKORIC[1], Marko ZAGORICNIK[1], Aljaz DRNOVSEK[2]**

[1] Faculty of Technical Sciences, University of Novi Sad, Novi Sad, Serbia

[2] Jožef Stefan Institute, Ljubljana, Slovenia

*Corresponding author: palterek@uns.ac.rs

***Abstract:*** *In recent years the application of high-pressure die casting (HPDC) technology for mass production of complex aluminium alloy components is expanding. As a result, the requirements for the efficacy and wear resistance of HPDC tool elements have also increased. Therefore, applying hard coatings and protective layers on HPDC tool surfaces provides great potential in this regard. The performance of protective layers heavily depends on their surface topography, growth defects in the coating, and the variable nature of surface chemistry, whose effects are still not fully understood. This investigation evaluated EN X37CrMoV5-1 steel, plasma nitrided steel, CrN, and TiAlN PVD coatings deposited in form of duplex layers. All kinds of samples were prepared to the same degree of surface roughness, except the coated samples, which were prepared with one additional degree of surface roughness. Soldering and wear behaviour in cast Al-Si-Cu alloy was evaluated by a laboratory ejection test performed in two configurations, conventional (CS) and delayed (DS) alloy solidification. Before and after the experiments 3D profilometry and different microscopy and spectroscopy techniques were employed to characterize the sample surfaces. In DS experiments, the steel samples exhibited severe soldering and very high ejection forces. Plasma nitrided samples behaved significantly better but spallation of the surface layer occurred in DS experiments. In both experimental configurations, PVD coatings outperformed steel and nitrided layers and exhibited no reaction with the cast alloy. The main drawback of PVD coatings is that decreasing the roughness considerably increases the ejection force from the CS castings. However, the highest ejection force recorded for both post-deposition polished PVD coatings decreased in DS tests. This is due to the formation of an oxide layer on both CrN and TiAlN coatings, which significantly reduced chemical interaction with the cast alloy and friction. To achieve optimal coating performance, it is essential not only to select the appropriate coating type but also to consider surface morphology and the transformations of coatings during their use.*

***Keywords:*** *HPDC, Al-alloy casting, hot-working tool-steel, PVD coating, plasma nitriding, galling, soldering*

## 1. INTRODUCTION

HPDC is a technology used for the mass-production of complex-shaped components of light alloys. In contemporary industry, the importance of this technology for producing lightweight components is ever-increasing. However, maintaining the casting quality and production efficiency at high production rates is a challenging task due to the tool wear [1,2]. The HPDC tools suffer from soldering (sticking), erosion, thermal cycling fatigue, and adhesive or abrasive wear. To combat this, the critical tool elements are protected by diffusion layers and coatings with high properties: adhesion, hot hardness, toughness, inertness, and oxidation resistance [1–3].

Nowadays, the cast alloy soldering wear remains one of the greatest challenges to be solved [4]. It affects casting properties like dimensional and shape tolerances, surface finish, and chemistry, but also production efficiency and HPDC tool integrity [1]. To solve this problem, layers inert to molten Al-alloys and less susceptible to the adhesion of the alloy are required [1]. Development of coatings requires testing their performance in laboratory conditions that can closely resemble the real industrial conditions [3].

The mechanisms of soldering wear are divided into mechanical and metallurgical soldering. Mechanical soldering (sticking) includes galling and adhesion of cast alloy [1,4]. Metallurgical soldering combines the effects of mechanical interaction with the chemical interactions and diffusion of the cast alloy and the tool materials [1,4].

The performance of protective layers strongly depends on their surface topography, the presence growth defects in the coating, and on the nature of the chemistry of the top layer that is brought in contact with the cast alloy. In our previous studies [1,5], we evaluated the performance of different materials, with different surface finishes by the ejection test. The chosen configuration of the test predominantly simulated mechanical soldering wear [1]. To explore the effects of more severe HPDC conditions on the behaviour of developed layers a test with enhanced effects of metallurgical soldering wear is required.

In this investigation, we examined the soldering wear od hot-working tool steel, plasma nitrided steel, and CrN and TiAlN duplex coated steel. For these purposes we used an ejection test with conventional and delayed casting alloy solidification.

## 2. MATERIALS AND METHODS

This investigation concerned quenched and tempered H11 hot-working tool steel (EN X27CrMoV51), plasma nitrided steel, CrN and TiAlN duplex layers produced on plasma nitrided H11 steel. Cylindrical samples used for this investigation were ϕ15×100 mm. Final surface finish was obtained by fine grinding and polishing using 6 and 3 μm diamond paste.

Pulsed plasma nitriding was performed by ION-25I (IonTech) unit in a 12-hour processing cycle. Before the coatings deposition plasma nitrided samples were subjected to polishing. The CrN coating was deposited by the thermionic arc ion plating in BAI730 (Balzers) system and the TiAlN coating was deposited by an unbalanced magnetron sputtering in CC800/7 (CemeCon) unit.

All kinds of samples were prepared with two degrees of surface roughness but the coated samples were prepared with one additional degree of surface roughness. The finest surface finish of coated samples was obtained by post deposition polishing with 3 μm diamond paste. Denotations of used samples are presented in Table 1.

**Table 1.** Samples denotations

| | **H11** | **PN** | **CrN** | **TiAlN** |
|---|---|---|---|---|
| **Smooth (S)** | H11 | PN | CrN | TiAlN |
| **Post polished (PP)** | - | - | CrN-PP | TiAlN-PP |

The ejection test was employed to evaluate the cast alloy soldering tendency. In this test, pin sample is used as a core for production of a simple casting with a hole (Figure 1). By this method, a sample-casting assembly is obtained. A tensile testing machine ZDM 5/91 (VEB) was used for the ejection of the samples from castings. During the ejection process, a load-displacement curve was recorded (ejection curve). This test simulates the process of a core removal from the casting produced by HPDC technology. The load recorded during the test contains information about the soldering tendency of cast alloy toward sample material. Production of sample-casting assemblies was performed in two types of experiments, i.e. by conventional and delayed casting solidification.

The casting experiment was performed by gravity melt pouring of EN AC-46200 aluminium alloy, at temperature of 730 °C. The cast metal pouring is performed into a specially designed steel die (Fig. 1), preheated to a temperature of 320 °C, and it this configuration is called a conventional casting solidification (CS). The experiment with delayed solidification (DS) was conducted using the same die, which was preheated to 600 °C (Figure 1). Upon the filling of the die cavity, the die was placed into the furnace heated to 700 °C for 20 minutes in to prevent casting solidification. After the predetermined time, the die was taken out and the casting was allowed to solidify. This experimental procedure has been developed to enhance the corrosion processes between the casting and the sample. The CS experiments were conducted with three repetitions, whereas the DS experiments were carried out without any repetitions.

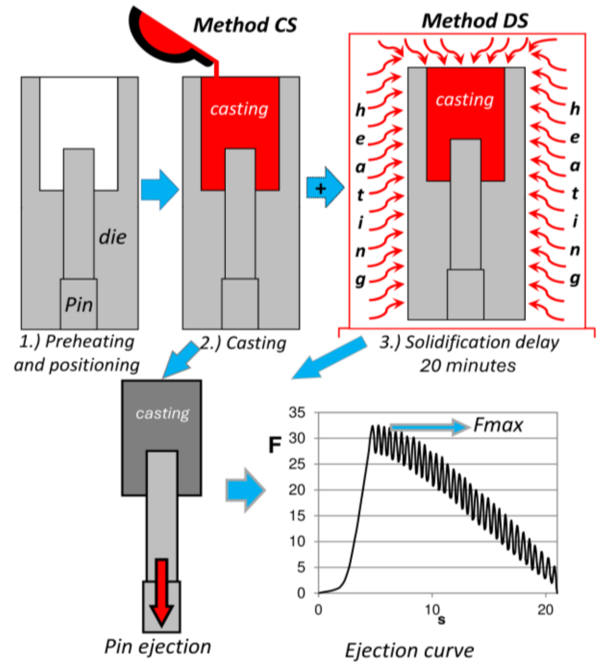


**Figure 1.** Schematic representation of the employed experimental casting methods

3D stylus profilometery (Talysurf, Taylor Hobson ) was employed for the evaluation of the samples’ surface topography. Mechanical properties were evaluated by an instrumented hardness tester H100C (Fischerscope). A load of 100 mN was applied for these measurements.

After the ejection tests, the samples` surface morphologies were examined by confocal optical microscope (CFM) Axio CSM700 (Zeiss). Surface characterization by Focused ion beam (FIB) was performed using Helios Nanolab 650i (Fei) and Scanning electron microscopy (SEM) was performed by TM2000 (Hitachi) device, both equipped with energy dispersive spectroscopy (EDS).

## 3. RESULTS AND DISCUSSION

The properties of the investigated materials are presented in Table 2. The mechanical properties of H11 steel are typical representatives of HPDC tool elements. The thickness and hardness of all investigated layers are sufficient to increase the material's bearing capacity and wear resistance for application on HPDC tool elements.

**Table 2:** Properties of investigated materials and surface layers

| | H11 | PN | CrN | TiAlN |
|---|---|---|---|---|
| **Hardness [$HV_{kgf}$]** | 455 $HV_{30}$ ±41 | 300 $HV_{0.01}$ ±76 | 2575 $HV_{0.01}$ ±208 | 3600 $HV_{0.01}$ ±655 |
| **Thickness [μm]** | - | 100 ±10 | 2.7 ±0.2 | 3.4 ±0.2 |

The mean arithmetic roughness (Ra) of the investigated samples is jointly presented with the values of the ejection force in Figure 2. The surface roughness of smooth samples (S) belongs to the range of fine surface finishes characteristic for HPDC tool parts employed in the production of high-quality castings. However, the roughness of the post polished samples (PP) belongs to the group of very fine surface finishes.

The appearance of sample surfaces after the ejection tests are shown in Figure 2. All samples are covered with a cast alloy built-up layer, the soldering layer. Also, soldering craters and initial sample surfaces can be observed. The soldering observed on samples from CS experiments results from pure sticking and galling phenomena.

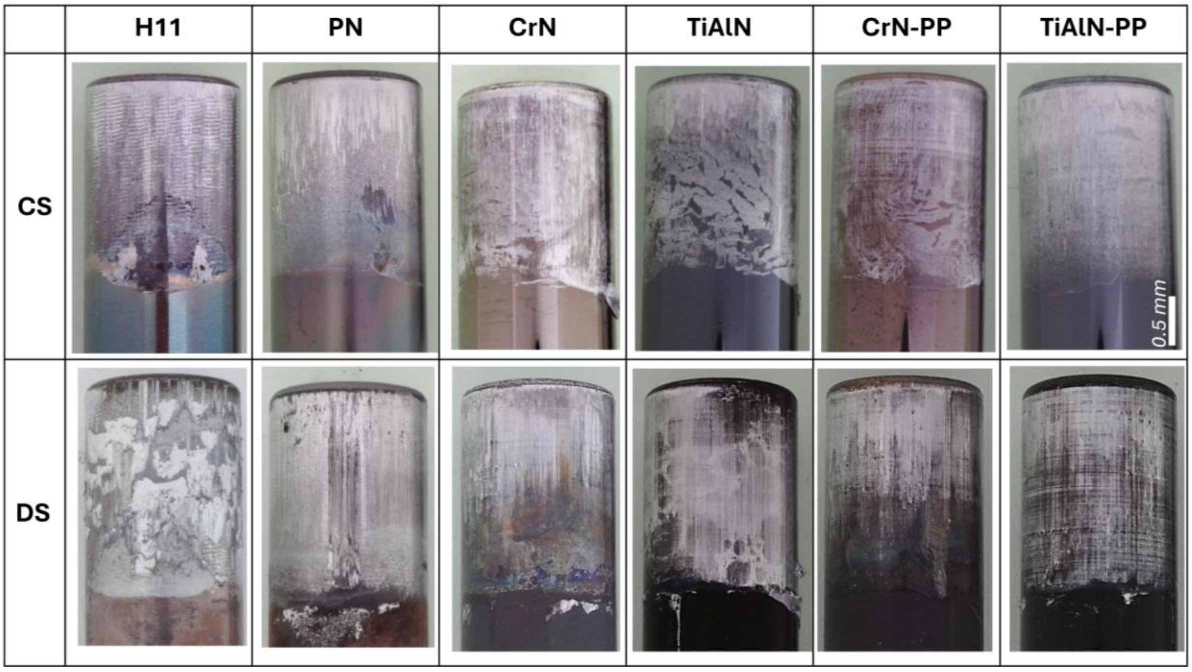


**Figure 2.** Appearance of the samples' surfaces exposed to the molten alloy after the ejection tests

Such soldering phenomena chemically do not affect the pin material, but induce effects of adhesive wear [1].

After the DS tests, the most severe soldering layers (white in the image) were observed on the surfaces of H11 samples, together with the presence of deep craters (intermediate grey). Such features indicate metallurgical soldering. The untreated surfaces of PN, CrN, and TiAlN samples have a darker colour than the initial sample surface, which indicates oxidation. The soldering layer has the same morphology as the one on the samples subjected to CS tests. However, in the top regions of PN sample, very small delaminated areas can be seen.

To reveal the wear and soldering mechanisms that operate in the investigated process, a more detailed analysis of the sample surfaces after the tests was performed by SEM, FIB and EDS. SEM and EDS analysis of H11 sample, presented in Figure 3. and Table 3., reveals an occurrence of metallurgical soldering. On the initial, oxidised surface (1) an intermetallic feature (2) formed between the cast alloy and sample material. Given that these features have weak adhesion, they are easily torn out during the sample ejection. In this way, a soldering crater is formed (3). Such phenomena occur on a much larger scale, which can be seen as an intermediate grey colour on the samples shown in Figure 2. The presence of oxide on H11 steel increases the soldering resistance [6], but it does not stop it. The observed soldering is similar to that reported in the literature [3,4].

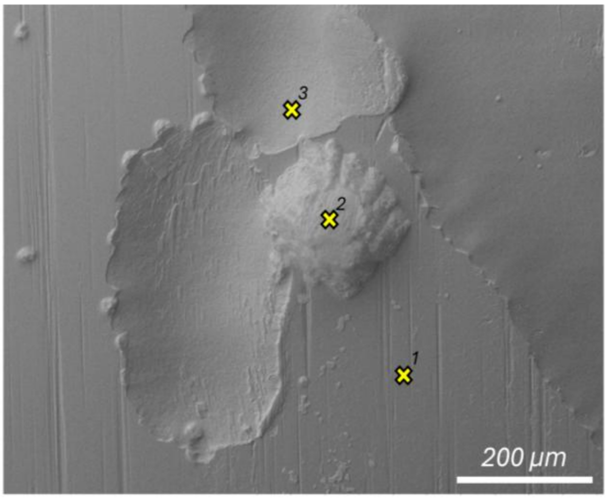


**Figure 3.** SEM analysis of H11 surface after DS experiment, cross marks indicate the locations of EDS analysis on typical areas, and these are: 1-initial surface, 2-cast alloy soldering, 3-soldering crater

**Table 3:** Results of EDS on H11 sample

| % | Fe | Al | O | Si | C | Cr | ... |
|---|---|---|---|---|---|---|---|
| **1** | 64.0 | 8.0 | 20.2 | 1.3 | 4.6 | - | 1.8 (Mg) |
| **2** | 13.5 | 36.2 | 14 | 36.3 | - | - | - |
| **3** | 56.3 | 3.6 | 23 | 2.6 | 4.8 | 9.0 | 0.6 (S) |
| **4** | 72.0 | 1.5 | 13.6 | 1.6 | 4.9 | 5.3 | - |

The surface of PN sample subjected to DS test is covered by a thin layer of cast alloy and it is characterized by multiple delaminated layers, Figure 4 and Table 4. The initial surface (1) is oxidised during the sample preheating stage, which is expected for heating of plasma nitrided layer to 600°C [7]. The cast alloy soldered to the oxidised surface by its reduction and formation of Al-Si-Fe intermetallic layer (2). Given that this layer has good bonding to the casting, during the sample ejection, it transfers the shear stress below, which tears off the oxide top layer and the one beneath it. This process leaves sporadic areas of delaminated material stretching to the diffusion layer beneath the compound nitriding layer (4). Such a wear mechanism of nitrided HPDC tool is not reported in the literature so far.

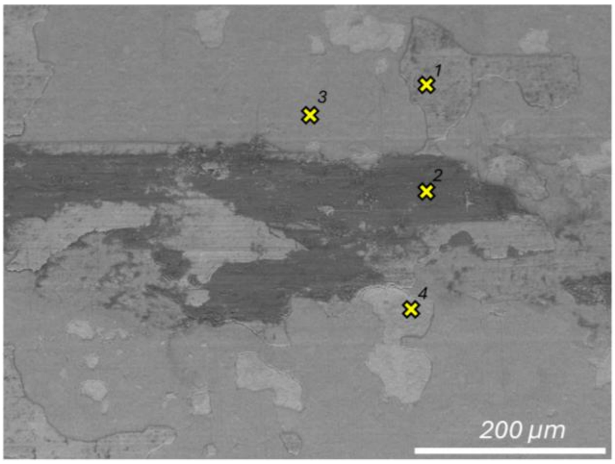


**Figure 4.** SEM (backscattered electron) image of PN surface after DS experiment. Cross marks indicate the locations of EDS analyses on typical areas, which are: 1-initial surface, 2-cast alloy soldering, 3-soldering crater, 4-area beneath compound layer

Figure 5 shows typical image of the CrN and TiAlN samples' surfaces covered with the cast alloy, observed after DS tests. On both samples, the cast alloy, remained in thin, soldering layers that sporadically cover the surface. Delamination, cracking and corrosion of the coatings is not observed. The soldering layer seems firmly bonded to the surface. As a consequence of the ejection process, in some regions the built-up is smeared on the surface.

**Table 4:** Results of EDS on PN sample

| | Fe | Al | O | Si | C | Cr |
|---|---|---|---|---|---|---|
| **1** | 79.6 | 1.7 | 12.5 | 1.43 | | 4.8 |
| **2** | 8.4 | 36.5 | 17.7 | 20 | 17.5 | |
| **3** | 85.3 | 7.2 | | 3 | | 4.5 |

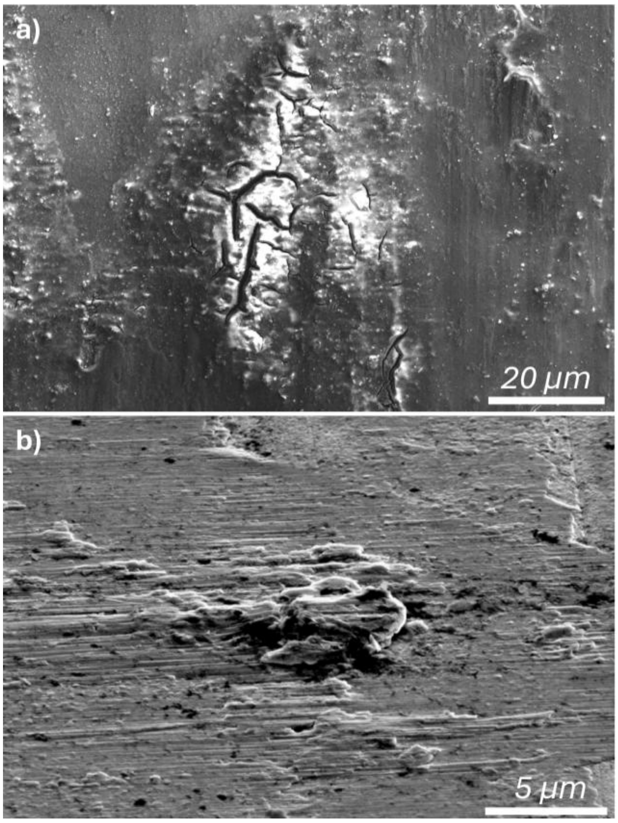


**Figure 5.** SEM analysis of a) CrN and, b) TiAlN sample surfaces after DS experiment

The coatings' integrity and inertness were evaluated on the cross section of the coating exposed to cast alloy. FIB-SEM and EDS line analysis of CrN coating are presented in Figure 6. The cross section is performed on a location with a nodular defect in the coating. On the cross section, changes in the top layer and on the layer close to the substrate are observed. On the top of the CrN coating a 50 nm thick oxide layer formed. Such layer is not detected on the sample subjected to CS test. $Cr_2O_3$ formed during the preheating stage of the sample before the casting to 600°C, which is in line with the literature data [8]. Beneath the oxide layer lies a layer rich in N. Its formation is attributed to the process of substituting the N by O and formation of a diffusion barrier by Cr-O layer [9]. The bottom layer enriched in N formed because of N diffusion from the nitrided layer into the under-stoichiometric CrN layer which caused its saturation [9]. The formation of compounds between the cast alloy and the coating material were not detected. Generally, the CrN coating underwent some changes in its chemical and microstructural properties, whose impact on its mechanical properties remained unknown.

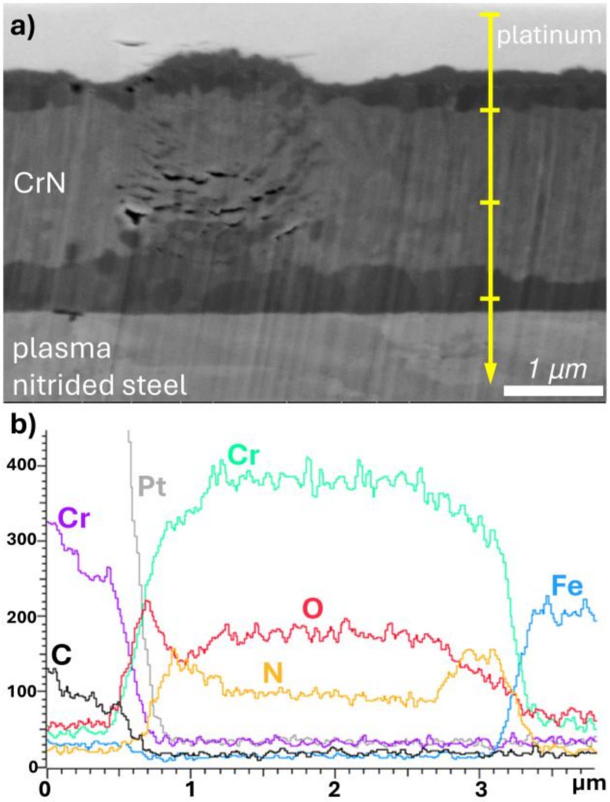


**Figure 6.** a) FIB-SEM cross-sectional analysis of CrN-PP sample after DS experiment, b) EDS line analysis performed on yellow line in image

The formation of a thin oxide layer on the top of the coatins is also detected on the FIB cross-section of TiAlN coating, not shown here. Additionally, we did not found any other changes in the coating layer. This indicates that this coating is more stable than the CrN coating. The presence of oxides on both coatings is also confirmed by X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectroscopy (SIMS), which are not included here.

Given that a thin layer of the cast alloy is detected on the surfaces of PVD coatings, it is indicated that some sticking tendency of cast alloy exists. However, metallurgical reasons for such behaviour are not found in this study.

The maximal ejection force of different materials, subjected to CS experiments, is presented in Figure 7. The values of the ejection force of the samples with similar roughness are quite comparable between the samples. However, the ejection force of CrN-PP and TiAlN-PP samples are higher by 50 and 70 %, respectively.

These results indicate that in the investigated range of surface roughness, the sticking and galling tendency is independent of the surface chemical composition [1]. It is more dependent on the surface roughness. High ejection forces of the post polished samples were induced by high tangential force, which enhances the adhesion effects [10]. These results indicate that the application of highly polished HPDC tool elements is not beneficial in the production cases with dominant mechanical soldering and galling.

Compared to the CS test, the force required to eject the H11 sample from the casting in DS tests increased by ≈ 120 %. This is a consequence of severe metallurgical soldering [4], which was confirmed by SEM and EDS analysis. This process creates strong bonding between the casting and H11 steel, which, upon ejection, changes the surface geometry by forming undercuts.

For PN, and the smooth, coated samples (CrN, TiAlN) the ejection force remained consistent with the CS test or slightly decreased. The observed shearing and partial delamination of observed on PN sample is linked with the values of the ejection force. The soldering layers formed in this process are thinner but have weak bonds with the unaffected sample material. Accordingly, during sample ejection, these layers shear under lower loads but leave shallow delaminated areas. These findings indicate that the PN samples exhibited higher resistance to metallurgical soldering than the untreated H11 steel [3]. Still, the observed wear is detrimental to the tool's integrity. Low ejection force values observed for smooth CrN and TiAlN samples align with the fact that they did not undergo serious galling, layer delamination, and metallurgical soldering. Their performance remained similar to that observed in CS test.

A pronounced decrease in the ejection force is observed for the post-polished PVD coatings, which is 47% and 42% for CrN and TiAlN coating, respectively. The formation of oxide layers on both investigated coatings reduced the ejection force. These oxide layers are more inert to molten aluminium alloys than the nitride coatings and as such, they impede galling and adhesive wear [11,12]. Additionally, these oxides have a lower coefficient of friction in contact with the oxides on the aluminium alloys [13]. These findings indicate that the oxidation of CrN and TiAlN coating is beneficial in the improvment of the casting ejection performance. Deposition of $Cr_2O_3$ and $Al_2O_3$ coatings for the reduction of tool soldering and ejection improvement is suggested in previous works [2] as the most appropriate approach. However, deposition of such layers on tool elements is quite expensive and problematic from the adhesion point of view [2]. Therefore, controlled oxidation of HPDC tool elements coated with nitride hard coatings could be an cost-effective alternative with better mechanical properties and endurance.

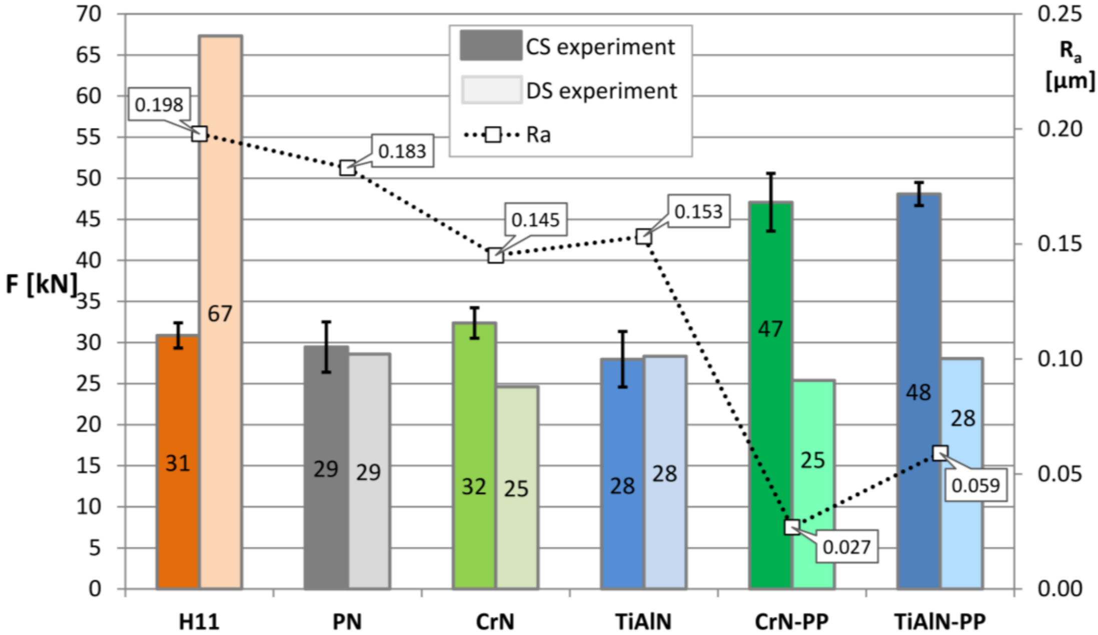


**Figure 7.** Values of the maximal ejection force obtained for all tested samples in both experimental configurations. Ra roughness parameter is given for different sample groups. Error bars represent ±1 confidence interval

## 4. CONCLUSION

In this study, the soldering wear performance for H11 steel, plasma nitrided H11 steel, and CrN and TiAlN duplex coatings is evaluated. The soldering tendency is evaluated by the ejection test performed with conventional and delayed cast alloy solidification. From the conducted investigation, the following conclusion can be drawn:

- In a conventional solidification test, a large influence of the surface roughness on the values of the ejection force was revealed. For PVD coated samples, a decrease in surface roughness induced an increase in the ejection force.
- The ejection test with the delayed casting solidification better simulates the metallurgical soldering effects, combined with cast alloy galling, than the conventional casting solidification.

- In delayed solidification tests, H11 steel displayed severe soldering in aluminium alloy, formation of pits, and undercuts.
- Plasma nitrided H11 steel displayed better resistance to the soldering wear than bare H11. The soldering wear of the plasma nitrided layer manifests in delamination of thin layers that occur during the ejection process.
- In the delayed casting solidification tests CrN and TiAlN did not display any reaction with the cast alloy or any kind of mechanical damage to the coating layer.
- Oxidation of CrN and TiAlN coating significantly improved the coating ejection performance without compromising the soldering and corrosion tendency.

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## ORCID iDs

Pal TEREK 0000-0001-9994-6039
Lazar KOVACEVIC 0000-0002-4087-4373
Vladimir TEREK 0000-0003-0798-456X
Zoran BOBIC 0000-0002-0843-4984
Branko SKORIC 0000-0002-3232-2544
Marko ZAGORICNIK 0000-0002-4764-3172
Aljaz DRNOVSEK

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