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EVALUATION OF SOLDERING RESISTANCE OF CrAlN COATINGS INTENDED FOR APPLICATION ON HIGH PRESSURE DIE CASTING TOOLS

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Abstract: *During high-pressure die casting (HPDC) of aluminum alloys the tool surfaces are exposed to extreme conditions as high temperatures, molten metal impact at high velocities, high pressure, corrosive environment, alternate heating and cooling cycles and abrasive wear. In such conditions tool surfaces suffer from thermal fatigue, erosion and soldering wear. As a result of tool wear, machine downtime and tool maintenance increases which cause significant financial losses. To reduce these losses and to extend the tool life, HPDC tool surfaces are submitted to plasma diffusion treatments and deposition of PVD (Physical Vapor Deposition) coatings. Therefore, in this study, the performance of duplex CrAlN coatings with three different chemical compositions was evaluated. The performance of the coatings was investigated in term of its soldering tendency to Al-Si-Cu alloy, using a detachment test. This test involves formation of a cylindrical casting on flat coated surfaces which is detached after the solidification and a required force is recorded. After detachment the analyses of contact surfaces reveal the soldering mechanisms and a sticking tendency of cast alloy toward investigated surfaces. For these purposes we employed 3D profilometry and different microscopy techniques. The cast alloy is kept in contact with the investigated coatings for four predetermined periods of time. The scanning electron microscopy (SEM), on all coatings, revealed the remnants of cast alloy (solder) after detachment tests and also a prominent coating delamination. The soldered area for different coatings has the same trend for different periods of contact with molten alloy. However, the amount of coatings delaminated area is different and closely correlates with samples detachment force. Higher detachment force is addressed to larger coating delamination area. It was found that the chemical composition of investigated coatings has modest influence on the soldering phenomena.*

Keywords: *HPDC tool, aluminum alloy, duplex treatment, PVD coatings, soldering*

1. INTRODUCTION

HPDC is a mass production process in which molten metal is injected into steel die (tool) at high velocities and employing high pressures. Rapid casting solidification is achieved by cooling lines in the tool walls. The process is used for casting aluminum, magnesium and zinc alloys. HPDC products are applied for wide variety of applications and can be found in everyday life all around us. Typical representatives of casted aluminum are parts of different devices, housings for mobile phones, engine parts, decorative parts, structural car parts, etc.

During the HPDC process, tool is exposed to extreme environment which cause thermal shock, oxidation, erosion, corrosion and soldering [1]. Cast alloy soldering represent one of the greatest issues in modern HPDC industry because it reduces both casting quality and process productivity [2]. In a soldering process cast alloy (casting) “welds” to the surface of the metallic die and forms a firm bond between them [3]. Such process hampers the casting ejection, cause defects on casting surfaces, damages the HPDC tool surfaces and endanger it integrity. All these effects beside the shortening of a die life increase machine downtime due to increased tool maintenance.

The cast alloy soldering is prevented by spraying of a liquid lubricant on die surfaces before each casting cycle. Considering that HPDC is a mass production process, significant amount of lubricant during is used in this production process. The amount of used lubricant takes a serious part in production costs by this technology. Additionally, excessive use of lubricant has harmful influence on the environment.

In order to reduce negative effect of a lubricant, numerous studies have been conducted in the field. One group of investigations directed toward application of permanent protective layers on tool surfaces to improve their corrosion and soldering resistance [1, 4]. For these purposes, different types of surface treatments were investigated. The most successful treatments so far are conducted by plasma processes, such as plasma nitriding, coating deposition by PVD processes and combination of these, the duplex treatments [4].

This study aimed to investigate the interaction of Al-Si-Cu alloy with duplex treated hot-working tool steel surfaces, using a detachment test. Focus was on the bonding (detachment) force achieved between the casting and the sample and on the soldering effects on coated surfaces. It was assumed that different compositions of CrAlN nanolayered coatings would display different performance under soldering conditions.

2. MATERIALS AND EXPERIMENTAL

Disc shaped samples (ϕ30 x 5 mm) used in this experiment were produced of EN X27CrMoV51 hot-working tool steel in a quenched and double tempered condition. Samples (contact) surfaces were submitted to grinding by sandpapers of different grades (grits), from the roughest to the finest (360, 500, 1000 and 2000). After surface preparation, plasma nitriding was carried out in an industrial nitriding unit ION-25I (IonTech) equipped with a pulsed plasma generator. Nitriding was conducted for 12 h at a temperature of 510 °C using a gas ratio of H_2:N_2=3:1, and by applying a 0.6 duty cycle. The compound layer that formed during the plasma nitriding process on the sample contact surface was removed before the deposition of the CrAlN coatings. The compound layer was removed by diamond paste polishing technique using 3 µm paste granulation. The removal was done in order to produce duplex layers with high adhesion and high heat resistance.

Nanolayered CrAlN coating was deposited using an industrial DC-magnetron sputtering system CC800/9 (CemeCon). The coating was produced with three different chemical compositions. Different compositions were obtained employing a special approach with triangular sputtering targets. The whole process of a nanolayered architecture required four targets. Two regular rectangular targets one Cr and one Al, and to triangular-segmental Cr/Al targets. Different composition is achieved at different heights in the chamber. The deposition process was performed using a 2-fold samples rotation. The sample denotations are as follows. The coating with balanced chemical composition is denoted as CrAlN, the coating with higher aluminum content as CrAlN-Al and the coating with higher chromium content as CrAlN-Cr.

The coating thickness was determined using a standard ball cratering test. The coatings adhesion was evaluated by Rockwell HRC test applying a force of 150 N. Surface topography of samples was acquired by 3-D stylus profilometer Talysurf (Taylor Hobson). Topographic measurements were analyzed by Mountainsmap and a Scanning Probe Image Processing software-SPIP (Image Metrology) software.

A laboratory test was employed for soldering evaluation, the detachment test. This test involves formation of a cylindrical casting on flat coated surfaces which is after the solidification detached from the surface. During detachment a force that is needed for casting detachment is measured and it represents a soldering tendency of paired materials.

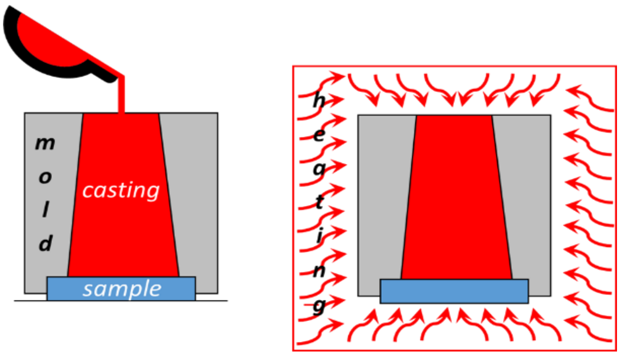


Figure 1. Schematic illustration of **c**asting process

For casting experiment, the mold was made from castable heat-resistant refractory, based on Al_2O_3 ceramic. The schematic of the casting process is given in Figure 1. Before the casting process molds were preheated to 550 °C and samples to 300 °C. Casting process is performed by gravity pouring of molten Al-Si-Cu alloy (EN AB-46000) into the cavity. The cast alloy temperature was chosen to be 750 °C to achieve better flowability. The contact surface between the sample and the molten aluminum was set at ϕ22 mm. Considering the casting solidification, experiments were performed in two setups. In first setup, after pouring one group of samples was left immediately to solidify in ambient air, the so called 0-delay experiments. In second setup, after the pouring, the mold was placed in a furnace (heated to 700 °C), kept there for 10, 30 and 60 min to delay the solidification and afterwards taken out to allow casting to solidify in ambient air. These experiments were named 10; 30; 60 - delay experiments. The second group of experiments basically simulates a larger number of casting cycles which occur in industrial HPDC process.

A ZGIM 500 tensile tester was used to perform the detachment test and to measure the force needed to separate the casting from the coated samples.

After the test, both sample and casting surfaces were analyzed using digital camera D3200 (Canon), light optical microscope (LOM) Orthoplan (Leitz), Scanning Electron Microscope TH3030 (Hitachi). Image analyses of the surfaces with the built-up were also performed SPIP software,

3. RESULTS AND DISCUSIONS

The properties of investigated coatings are presented in Table 1. Although the coatings were deposited in a same production batch they resulted with different thickness. This is attributed to the effect of position (height) in deposition chamber. The number of coating defect varies from sample to sample. In Table 1. the average number of the area that is covered by coating defects and the average number of coating defects are given. The roughness of produced coatings is quite similar while the highest roughness of CrAlN-Cr coating is attributed to the highest density of average coating defects (crater and nodular). Figure 2 shows the results of HRC adhesion test. All produced coatings exhibited very good adhesion. CrAlN-Al coating has the best adhesion achieving HF1 level, while CrAlN coating exhibited HF2 and CrAlN-Cr coating the HF3 level of adhesion. The highest adhesion of CrAlN-Al coating is attributed to higher ductility achieved by thicker AlN nanolayers. The obtained levels adhesion and the observed coating ductility make these coatings appropriate for application on HPDC tools.

Figure 3 presents the values of detachment force recorded for investigated coatings which were subjected to different experimental setups. Detachment (sticking) force should be a measure of bonding strength achieved between casting and coating.

Table 1. Properties of Investigated coatings

Sample	Coating thickness [µm]	Sq [µm]	Coating defects density [num/mm^2]	Area of coating defects [nm^2]
CrAlN	5,7	0,192	185	119.7
CrAlN-Al	4,9	0,193	144	173.5
CrAlN-Cr	7,1	0,269	204	155.5

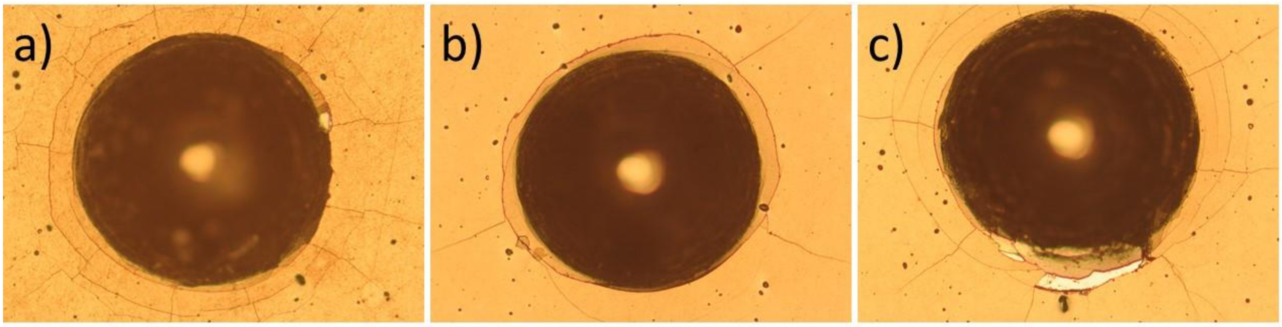


Figure 2. Results of Rockwell HRC adhesion test, a) CrAlN, b) CrAlN-Al and c) CrAlN-Cr coating

However, the sample surfaces indicate few different wear mechanisms which all have their contribution to the value of detachment force. These different components are: the bonding strength achieved between the casting and the coating, cohesive strength of soldered aluminum alloy and coating-substrate adhesion.

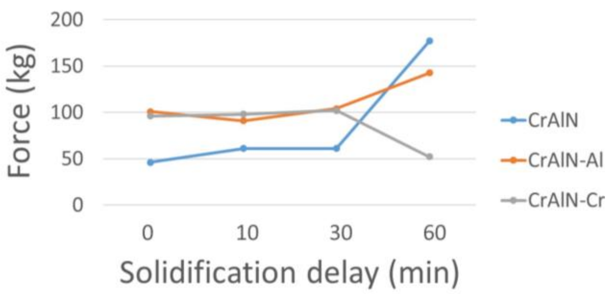


Figure 3. Detachment forces recorded for investigated coatings. Different experimental setups (period of contact with molten alloy) are presented on abscissa, 1= 0 min; 2 = 10 min; 3 = 30 min; and 4 = 60 min

For most of the investigated range of solidification delay periods CrAlN coating displayed the lowest ejection force. In the range of solidification delay from 0 to 30 min the values of detachment force determined for all investigated coatings does not change considerably with delay periods. For the experiment of 60 min solidification delay, the previously established trends completely change. CrAlN exhibit approximately three times higher detachment force, CrAlN-Al a slight increase and CrAlN-Cr coating exhibit almost double decrease of detachment force. Such results are mostly in agreement with results of cast alloy soldered areas and coating detachment areas, that are presented in paper.

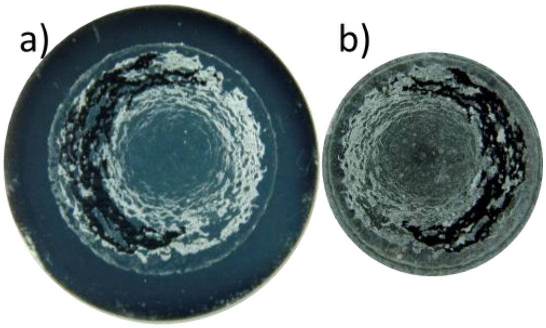


Figure 4. Appearance of CrAlN-Al 30min of solidification delay, a) sample and b) casting contact surfaces after detachment test

The appearance of coated sample surfaces and corresponding castings, after detachment tests, provide additional information about the soldering process. Using this information, the obtained detachment force can be explained and the inherent soldering mechanisms can be revealed. Figure 4 present a macroscopic image of the most representative coated sample (Figure 4 a) and its corresponding detached casting surface (Figure 4 b). The features on both elements correspond to each other. This is obvious when the images are specifically oriented to forms a mirror reflection of each other, see Figure 4. On both surfaces distinctive features can evidence the soldering process. LOM analysis revealed two distinctive soldering mechanisms. The first is that the cast alloy remained on coated surface in form of a soldering layer. The second is the coating detachment that occurred in these tests. Soldering layer can be seen as bright area, and spallation as dark area on the images of Figure 4. Analysis conducted on both coated sample and corresponding casting provide information on soldering processes that have detrimental effects which cause coating detachment from steel substrate surface.

In order to confirm typical wear features detected by LOM, after detachment test the coated samples were submitted to SEM and energy dispersive spectroscopy (EDS) analysis, Figure 5. In Figure 5a, a wider area with different kind of coating damage is presented. In that image a soldered layer can be clearly distinguished from detached coating and the intact coating area. All these features were confirmed by EDS analyses enclosed in the figure. The location shown in Figure 5b is an area with intact coating layer. However, the area nearby is the area where coating detachment occurred. At the area with intact coating, soldering is not detected, which agrees with findings published in literature that ceramic coatings do not wet with liquid aluminum alloy [5]. However, this does not agree with the fact that soldering layer can be found on a considerable area of coated samples, as shown in Figures 4 a and 5 a. The inherent coating delamination and soldering mechanisms were not revealed in this analysis.

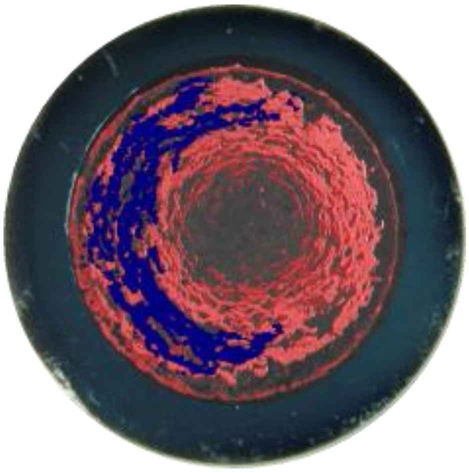
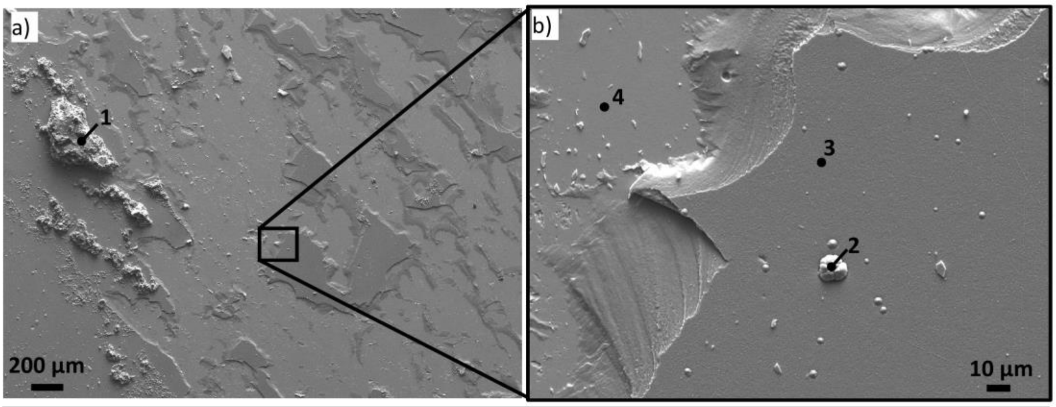


Figure 6. Representative image analysis, CrAlN-Al 30min

In order to quantify the soldering and coating detachment that occurred on the coated samples software image analyses were conducted. Detection of different wear features is helped by coupling the findings from SEM and LOM analysis. In Figure 6, the process of image analysis is presented on example of CrAlN-Al sample in 30 min experiment delayed solidification experiment. In presented image the red color represents the area of soldering and the blue color represents the color of the detached coating area from the substrate.



Spectrum	Chemical composition [%]									
	N [%]	O [%]	Al [%]	Cr	Fe	Si	Mo	V	C	Mn
1	-	3.22	52.81	16.52	15.49	0.91	-	-	8.9	2.13
2	24.58	1.84	8.6	64.61	0.38	-	-	-		-
3	22.13	1.62	9.22	67.04	-	-	-	-	-	-
4	3.19	-	-	5.49	86.51	1.08	1.16	0.33	2.24	-

Figure 5. SEM images of CrAlN-Cr sample subjected to 0 min delayed solidification, a) area with coating detachment and soldering; b) area with coating detachment, table presents EDS composition analysis

Figures 7 and 8 presents the quantitative results of image analysis used in soldering evaluation. There the areal coverage by cast alloy solder and detached (delaminated) coating area are presented for two experimental setups (30 and 60 min). For 30 min solidification delay, the amount of soldering and coating delamination closely correlates with measured detachment force presented in Figure 3. However, the amount of coating delamination has higher degree of correlation. The increase of detachment force is linked with more intensive coating delamination. This is obvious because the bond achieved between the coating and the substrate should be higher than one between the cast alloy and ceramic coating. CrAlN coating displayed the best performance in 30 min delayed solidification experiment.

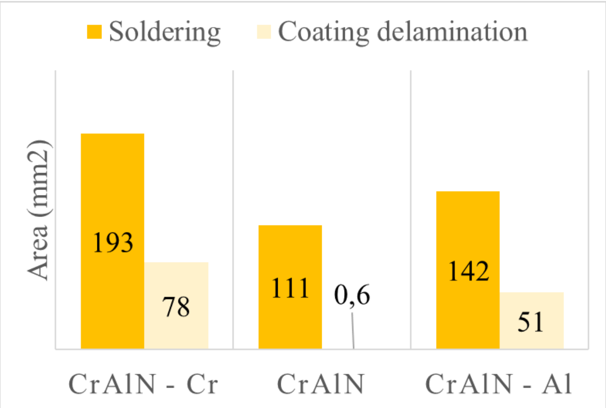


Figure 7. Areal coverage by soldering and area of coating delamination for 30 min delayed solidification

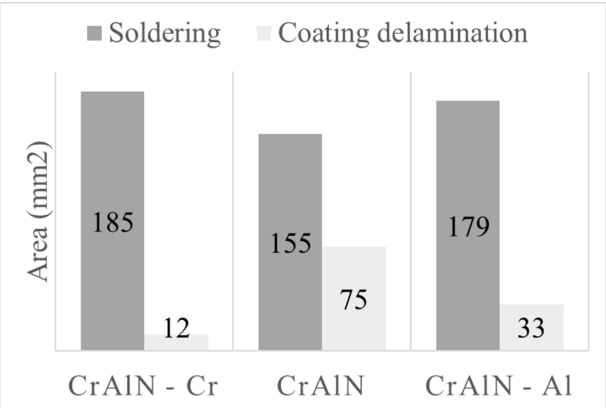


Figure 8. Areal coverage by soldering and area of coating delamination for 60 min delayed solidification

The quantitative results obtained for 60 min delayed solidification (Figure 8) display a similar trend of soldering area which is obtained for 30 min delay (Figure 7), but completely different values of coating delamination. However, once again, the coating delamination area has very good correlation with the detachment force. Such finding undoubtedly confirm that the value of detachment force dominantly depends on amount of coating delamination. CrAlN-Cr coating exhibited the best behavior in 60 min delayed solidification experiments.

A complete change of trends, in detachment force and areal coverage, between different coatings evaluated in 30 and 60 min delay experiments, remained unclear. We believe that such diversity in results can be explained by difference in the amount of coating growth defects between the same kind of samples which are used in different experiments (experimental points). In this investigation the coating defects density was not detailly monitored for every single sample.

4. CONCLUSIONS

From presented study the following conclusions are drawn.

- The investigated nanolayered CrAlN duplex coatings, of all three chemical composition, have appropriate properties for application on HPDC tools.
- For all investigated coatings and all experimental conditions, cast alloy soldering and coating spallation (detachment) was detected.
- The soldering mechanism and mechanisms that led to coatings deterioration (detachment from substrates) were not revealed in this study.
- CrAlN coating with balanced composition of Cr and Al, in most of the experimental conditions (contact durations with aluminum alloy) exhibited the lowest detachment force and smallest areas covered with cast alloy soldering. However, for the most severe experimental condition CrAlN coating with higher Cr content

displayed the smallest ejection force and coating spallation.

- The observed scatter of results obtained for two experimental points (delay periods) are addressed to the effects of coatings growth defects whose quantity were not monitored for every single sample in this investigation.
- The area of coatings delamination during detachment test has good correlation with samples detachment force. Such correlation was not found for the areal coverage by cast alloy solder.

The applied experimental method provides a new prospect on the evaluation of coatings soldering tendency. Its advantages are simplicity and a potential to acquire quantitative data on soldering resistance. However, some of techniques used in method have to be improved.

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