EFFECT OF DUPLEX SYSTEM NITRIDED LAYER – COATING ON THE CHANGE OF SURFACE TEXTURE PARAMETERS OF ALLOY CoCrMo

Ondrej KLANICA[1] – David DOBROCKÝ[2] – Filip ONDERKA[3] – David KUSMIČ[4]*

[1]*Ondrej Klanica, University of Defence, Kounicova 65, 602 00 Brno, Czech Republic. E-mail: ondrej.klanica@unob.cz*
[2]*David Dobrocky, University of Defence, Kounicova 65, 602 00 Brno, Czech Republic. E-mail: david.dobrocky@unob.cz**
[3]*Filip Onderka, University of Defence, Kounicova 65, 602 00 Brno, Czech Republic. E-mail: filip.onderka@unob.cz*
[4]*David Kusmic, Univeristy of Defence, Kounicova 65, 602 00 Brno, Czech Republic. E-mail: david.kusmic@unob.cz*
*Corresponding author E-mail address: david.dobrocky@unob.cz

Abstract
The properties of surface of biomedical implants made from CoCrMo alloy which are used as joint replacements have a fundamental influence on durability and functional properties of these implants. Surface texture parameters Ra and Rt are one of the important parameters which directly influences the durability of the coating and thus whole implant. The values of these parameters are given by standards ISO 7206-2:2011 and ISO 7207-2:2011. Three duplex surface systems were individually deposited to improve mechanical properties on the CoCrMo alloy. As a duplex treatment were used the combination of plasma nitriding process with subsequent depositing thin films of the coatings $nACo^3$®, DLC and ZrN. The process of plasma nitriding of CoCrMo alloy was implemented under these conditions: duration time was 10 hours, the ratio of gases H_2:N_2 = 3:1 and process temperature 450 °C, 500 °C and 550 °C. The surface texture parameters Ra, Rt were measured on the polished surface of the CoCrMo alloy before plasma nitriding then on the polished surface after plasma nitriding and finally on the polished surface after depositing of coating. It was found that the deposition of selected coatings on the surface after application of plasma nitriding influences the surface texture parameters Ra and Rt.

Keywords: CoCrMo alloy, Surface texture, Plasma nitriding.

1 Introduction

CoCrMo alloys are frequently used for the metal-on-metal hip resurfacing due to their high corrosion and wear performance. Thermal treatments are used on these alloys in attempt to alter the microstructure to improve the mechanical properties. However, the effect that this then has on the mechanical properties is in many cases insufficient especially in terms of wear resistance and low hardness. For this reason, the biocompatible coatings on CoCrMo alloys are used [1, 2]. The duplex surface system was applied to improve surface and subsurface properties. The combination of plasma nitriding and subsequent depositing thin films of the $nACo^3$®, DLC and ZrN were used as a duplex treatment. Plasma nitriding is universal chemical-heat treatment process which was used for creation of hard layer on CoCrMo alloy which serves as an underlayer for coatings. These duplex systems have very high hardness and good corrosion resistance. High hardness, chemical inertness and excellent tribological properties of nanocomposite coating of titanium aluminium nitride matrix of silicon nitride ($nACo^3$®), amorphous carbon coatings often called like diamond-like carbon (DLC) coating and zirconium nitride (ZrN) coating are of great interest for technological applications [2, 3, 4]. The surface texture has a decisive effect on the properties and behaviour of spare biomedical implants during their using (e. g. wearing, fatigue properties, connection strength and dynamic binding of surfaces, etc.). In case of coatings creation on implants, they may improve their properties using a duplex system nitriding – coating. Selected surface texture parameters Ra – arithmetical mean deviation of the assessed profile (μm), Rt – total height of the assessed profile (μm), according to [5], quantify the state of the surface before nitriding, after nitriding and after the deposition of coatings. Surface texture parameters Ra, Rt must be lower than Ra = 0.05 μm, Rt = 1 μm for the femoral component of the hip joint with using a wavelength 0.08 mm [6]. Surface texture parameters Ra, Rt must be lower than Ra = 0.1 μm for the femoral component of the knee joint [7]. The finishing treatment of inarticulating areas of metal components of knee joints that may come into contact with soft tissues must be smooth and non-abrasive. The value of surface roughness Ra = 1.5 μm is considered sufficient [8].

2 Experiment

For the preparation of the specimens were chosen cast CoCrMo alloy rollers of labeled B1, B1.2, B1.3 and M1, M1.2, M1.3. A series of specimens B and M differ by method of casting. Analysis of the chemical composition of the surface of the substrate of alloy CoCrMo (ISO 5832-4) was performed on a Noran EDS System SIX/300 and it is listed in Tab. 1.

Table 1 *Chemical composition of CoCrMo alloy*

Chemical composition wt (%)							
Element	Cr	Mo	Fe	Si	P	Ti	Co
Specimen B1	31.46	5.81	-	2.5	-	-	60.23
Specimen M1	30.22	4.81	-	1.47	-	-	63.50

The preparation of specimens consists of several consecutive steps. Each specimen was manually wet fine grained using by six grained papers from company HERMES with decreasing grain size of abrasives 120, 240, 500, 1000, 2500 and 4000. After grinding, the specimens were polished with diamond polishing paste (type POM L) with granularity 1 µm. Subsequently, the experimental specimens were plasma nitride according to the parameters listed in Tab. 2.

Table 2 *Parameters of plasma nitriding process*

Specimen	**B1.3, M1.3**	**B1.2, M1.2**	**B1, M1**
Temperature (°C)	450	500	550
Time/Duration (h)	10	10	10
Flow H_2 (l/h)	24	24	24
Flow N_2 (l/h)	8	8	8

Then plasma nitriding specimens were polished, cleaned and degreased. PVD coatings listed in Tab. 3 were deposited on surfaces thus prepared specimens. Selected surface texture parameters Ra, Rt were measured on the surface of the polished substrate CoCrMo alloy, polished nitride surface and subsequently on the deposited coatings. It was measured on five selected places for qualitative evaluation of the surface. The measurement was carried out by Talysurf CLI profilometer with diamond stylus and inductive gauge. Subsequently surfaces were evaluated by software TalyMap.

Table 3 *The overview of the used coatings*

Specimen		**Deposited coating**
B1	M1	nACo^3®
B1.2	M1.2	DLC
B1.3	M1.3	ZrN

3 Results and discussion

3.1 Arithmetical mean deviation of the assessed profile – Ra

Fig. 1 and Fig. 2 represent results of measured parameters Ra. The individual technological steps in the implementation of duplex system nitride layer – coating are color-coded. The default values of the parameter Ra substrates of experimental specimens B1 – B1.3 did not differ significantly from each other (Fig. 1). After application of plasma nitriding and subsequent polishing, the parameter Ra increased for all three cases of plasma nitriding. This phenomenon represents the increase of surface caused by the absorption (condensation) of nitrides during the process of plasma nitriding and simultaneously at its formation has the proportion even increase in the volume of material due to diffusion of nitrogen during nitriding process, as it is mentioned by other authors [9, 10]. Subsequently, in two cases B1 and B1.3 occurred after the deposition of the coating (B1 – nACo^3®, B1.3 – ZrN) to increase the values of the parameter Ra.

In case of the experimental specimen B1.2 was detected the lowest surface roughness (Fig. 1). The final value of Ra for sample B1.2, after deposition of DLC coating, was 0.039 ± 0.004 µm. The highest value of Ra parameter reached a sample B1.3 namely 0.054 ± 0.006 µm.

In case of experimental specimens M1, M1.2 and M1.3, the results are shown in Fig. 2. The lowest final value of the parameter Ra reached specimen M1, after the deposition of the coating (coating nACo^3®). In this case, the parameter Ra reached 0.042 ± 0.014 µm. Conversely, the highest value was measured on a specimen of M1.3 (ZrN coating) whose surface roughness reached 0.052 ± 0.004 µm.

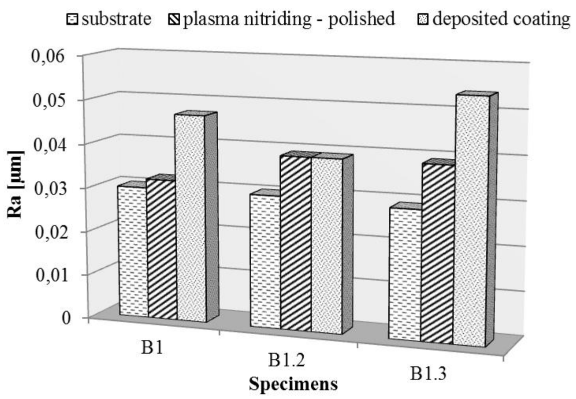

Fig. 1 *The parameter Ra texture of the surface of the specimens B1, B1.2, B1.3*

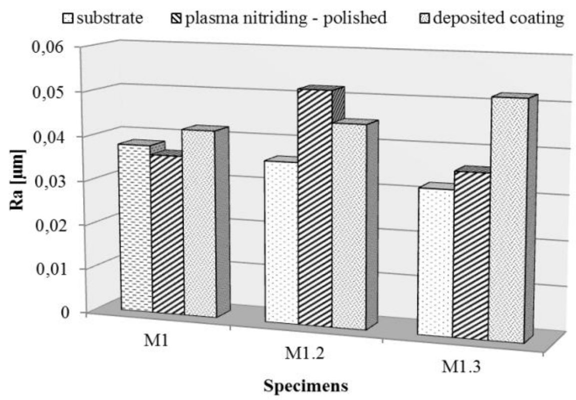

Fig. 2 *The parameter Ra texture of the surface of the specimens M1, M1.2, M1.3*

3.2 Total height of profile – Rt

Another evaluation parameter, which is binding by standard [5], is the parameter Rt. Fig. 3 shows the resulting values of Rt parameters for the set of samples B1 – B1.3. As well as at the parameter Ra, also in case of parameter Rt occurred to increase of its values after plasma nitriding and subsequent polishing. The lowest value of parameter Rt was measured for a specimen B1.2 (DLC coating). Parameter Rt reached value of 0.039 ± 0.004 µm. The highest values of the parameter Rt was achieved for specimen B1.3 (ZrN coating).

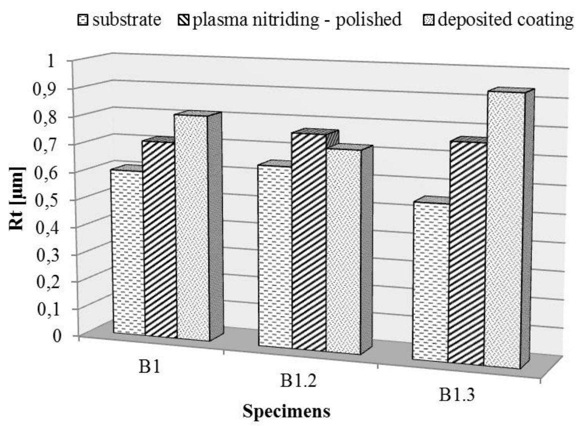

Fig. 3 *The parameter Rt texture of the surface of the specimens B1, B1.2, B1.3*

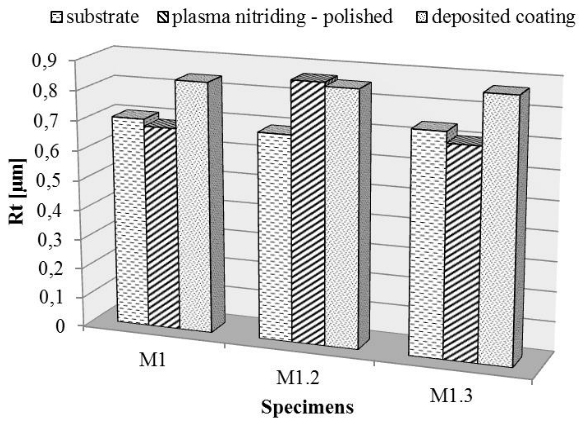

Fig. 4 *The parameter Ra texture of the surface of the specimens M1, M1.2, M1.3*

In the case of sets of specimens M1 – M1.3 occurred in two cases to a decrease parameter Rt after plasma nitriding and subsequent polishing. The final value of the parameter Rt was the lowest for the specimen M1 (coating nACo^3®) and it reached a value of 0.841 ± 0.105 µm. The parameter Rt had the highest value for a specimen M1.3.

4 Conclusion

The measured results point out that duplex system nitriding layer - coating which effect final values of the parameters surface textures. Analysis of changing parameters Ra, Rt of the surface texture was carried out depending on the different temperatures of the nitriding process and different type of the coating (DLC, ZrN, nACo^3®). Maximum standardized values of the parameters Ra and Rt for the production of the femoral component of the hip and knee joint are shown in ISO 7206-2, ISO 7207-2. After the experiments it can be stated that the maximum values given by standards are not exceeding. However, in some cases, some values move closer to the maximum permissible value. From the analysis it can be stated that the duplex surface nitrided layer (500 °C) - DLC coating (sample B1.2) showed the lowest values of parameters Ra = 0,039±0,004 µm and Rt =

0,719±0,101 μm. The best results showed duplex system nitrided layer (550 °C) - nACo^3® coating in terms of the different way of the casting specimens M1 - M1.3. The lower values of the parameters surface textures are associated with less wear and, together with the properties of the surface layer are critical factors for their durability and service life.

Acknowledgments
The work presented in this paper has been supported by the specific research project 2014 at the Department of Mechanical Engineering, University of Defence in Brno.

References

[1] G. Bellefontaine, The corrosion of CoCrMo alloys for biomedical applications, p. 3, University of Birmingham (2010).
[2] Z. Joska at al., Testing of DLC Coating on Plasma Nitrided Austenitic Stainless Steel, in 14th ABAF: Advanced Batteries, Accumulators and Fuel Cells. Brno, 2013, s. 99-100, ISBN 978-80-214-4767-7.
[3] M. Jelinek at al., Laser Physics, vol. 13, 10 (2003).
[4] M. Jelinek at al., Laser Physics, vol. 15, 2 (2005).
[5] ČSN EN ISO 4287. Geometrické požadavky na výrobky (GPS) – Struktura povrchu: Profilová metoda – Termíny, definice a parametry struktury povrchu (1999).
[6] ISO 7206-2. Implants for surgery – Partial and total hip joint prostheses – Part 2: Articulating surfaces made of metallic, ceramic and plastics materials (2011).
[7] ISO 7207-2. Implants for surgery – Components for partial and total knee joint prostheses – Part 2: Articulating surfaces made of metallic, ceramic and plastics materials (2011).
[8] M. Novotný, ČSN EN ISO 21536, Neaktivní chirurgické implantáty – Implantáty pro náhradu kloubů – Specifické požadavky na implantáty pro náhradu kolenních kloubů, (2009).
[9] V. Hrubý and A. Holemář, Iontová nitridace v praxi, SNTL Praha, ISBN 80-0300001-7 (1989).
[10] D. Pye, Practical nitriding and ferritic nitrocarburizing, USA, ISBN 0-87170-791-8 (2003).