BIOCOMPATIBILITY OF THIN LAYERS

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Abstract

Medical tools for minimally invasive surgery are often used in hard environmental conditions. For more effective protection against corrosion of the substrates, caused by steam sterilization loadings, TiN, TiCN, TiAlN, ZrN PVD - layers were used. All of the considered PVD - coatings have shown a sufficient sterilization resistance and the necessary biotolerance, which is required by the legislator for short-term applications of tools. The biocompatibility was estimated by monitoring and evaluating the growth of certain animal cell cultures. Steam sterilization was performed in an autoclave by the sterilization pressure 105KPa in 60 cycles.

Keywords: *PVD coating, medical tools, steam sterilization, biotolerance*

Introduction, experimental details

All these PVD coatings TiN, TiAlN, TiCN, ZrN investigated in this work were deposited in TECHNO-COAT Zittau GmbH on mirror-like polished stainless steels substrates, shaped as 5 mm thick circular discs with diameter of 20mm. We have used three steels X20Cr13, X5CrNi189 (AISI 304) and ČSN 17348 for discs. These steels belong to the most important ones used for tools of minimally invasive surgery. But AISI 304 stainless steels (about 9wt% of nickel) has lost good reputation in some applications of surgical instruments recently. The reason for this is the so-called nickel allergy problem. One of the most frequently asked questions' arising from the surgical tool industry is whether nickel can diffuse into the coating layers. It was demonstrated in [1] even after salt-spray loading above the anchoring region, absolutly no elements of the substrate material including nickel were observed. PVD coatings can protect human body from nickel difusion and as you will find in this paper; these layers can save substrat material from the corrosion and they can be biotolerant.

There were coatings investigated under two entirely different coating conditions in our case; unbalanced d.c.-magnetronsputtering using the so-called arc-bound sputtering method (ABS), and poor arc evaporarion. An HTC 1000/4 ABS engine was utilized as the coating device in all cases FIG. 1. This on cross-section hexagonal designed four-source device is equipped with a coating chamber of approxomately 1.5 m^3 inner volume, where 0.8 m^3 can be used as a loading space. The inner radius of the hexagon is 0.5m and the target shape is rectagular (18 x 60 cm^2). Two kinds of deposition methods can be performed. TiN, TiAlN, TiCN were deposited in so called arc mode (a) and ZrN in mode magnetron sputtering (m). All of the circular pieces to be coated were mounted on a triple-planetry substrate holder. For the experiments described here only two rotation axes were used. The whole batch process for a HTC 1000/4ABS™ device consists of the well-known steps; chamber evacuation, glow discharge (target cleaning and substrate heating/cleaning), ion etching (ion implantation), coating, substrate cooling and chamber venting.

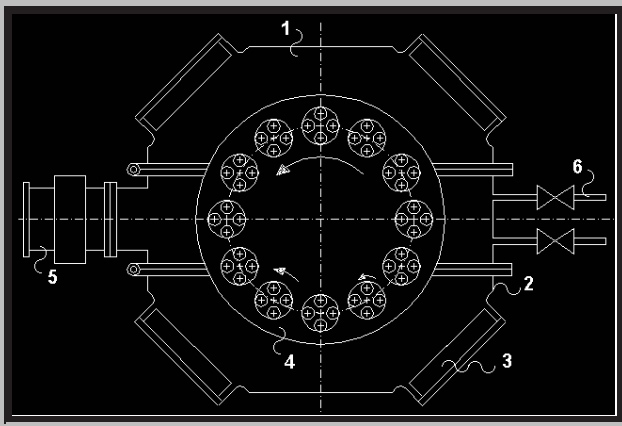

FIG. 1. Cross-section of HTC 1000/4 ABS™ coating device. 1. coating chamber; 2. chamber wall; ABS™ sources; 4. rotation table; 5. vacuum system flange; 6. gas mains.

		Thickness	Hardness	Scratch	CIELAB			Roughness R_z
		µm	Hv	N	L*	a*	b*	µm
Layers								
m-TiAlN	F	3,7	2200	10	47,9	-0,5	-1,8	2,2
	B	1,8	1600	30	48,0	-0,6	-1,5	1,9
m-TiCN	F	1,3	2000	25	68,3	13,4	24,9	3,0
	B	0,6	-	-	-	-	-	-
m-TiN	F	1,1	1400	20	83,9	-0,7	31,4	1,9
	B	0,5	-	-	-	-	-	-
a-ZrN	F	0,6	(1600)	(20)	77,3	2,9	31,7	2,0
	B	0,4	-	-	-	-	-	-
Substrat								
1.4021		0	600	-	81,8	0,2	1,6	1,0
CZ-17348		0	280	-	82,7	0,3	4,0	1,0
1.4301		0	230	-	83,8	0,3	4,3	1,0

TABLE 1. The main features (F = foreside, B = back of the specimen; m = Arc-Bound-Sputtering, a = arc-evaporation).

Results

To characterize the mechanical properties of the layers, the usual measurement methods were utilized, e.g. a kalotest for thickness, a scratch-test for adhesion, Vickers hardness for micro-hardness and Hommel Tester TDL for roughness.

Coating colours have been described by the CIELAB sys-

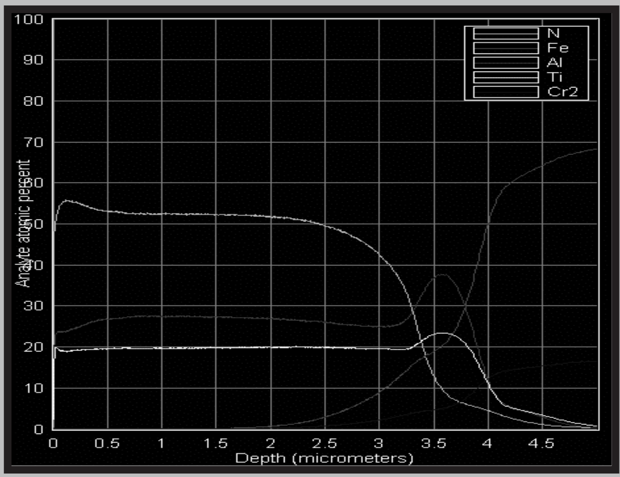

FIG. 2. GDOS Depth profile of the specimen coated TiAlN on 17 348.

tem utilizing a chromameter (TABLE 1). Regarding the three values, L*, a*, b* (lightness, redness-greenness and yellowness-blueness) of the CIE-1976 system, the lightness L* is of particular interest for reflective features of surgical tool. Such a layer's GDO(E)S analyse is down in FIG. 2 regarding the chemical depth profile. Besides some mechanical and optical properties, aspects of biocompatibility and steam sterilization resistance of TiN, TiAlN, TiCN, ZrN on steels were focused on.

Biocompatibility

Tools for minimally invasive surgery have to fulfill the demands of legislators. Simply speaking they have to be at least biotolerant. The observation and evaluation of the growth of certain animal cells is a simple and accredited method how to estimate the biotoleration of a PVD hard coating. The investigations with regard to biocompatibility were performed by Prof. Červinka from UK Faculty of Medicine in Hradec Králové. For this investigation mouse's-cells (cells type EL292) were used with medium MEM and solution 1.5% agar and 0.015% of neutral red (Holborn). Both the coated specimens and the solution were placed into 60 mm plastic tissue culture dish and incubated 24 hours at a body temperature 37°C in an atmosphere of 5% CO_2/95% air FIG. 3. The cells around the speciemen would die of

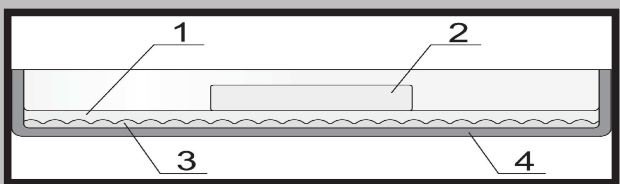

FIG. 3. The test of the layers' toxicity - 1. solution, 2. coated specimen, 3. cells, 4. Petri dish.

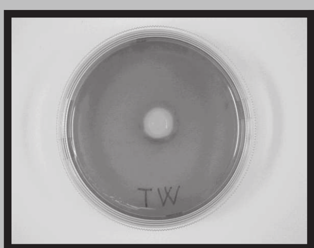

FIG. 4.The control specimen with toxic substans TW.

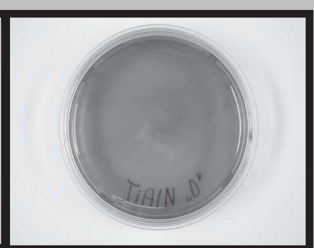

FIG. 5. Specimen coated TiAlN on 1.4021.

interaction with toxic material. Then, the scale of a toxic influence will be the zone of the white (dead) cells FIG. 4. There were no toxic impacts against investigated cells in our case FIG. 5. Such a performance could be interpreted as non-toxicity of the PVD-coatings on stainless steels. Nevertheless, this result is only an indication for "general" non-toxicity. And this test have to be repeated for every application.

Steam sterilization

The word sterilization means complete destruction of all micro organisms such as: spores, viruses, bacteria, etc. The most dangerous of all are the bacteria in the spore state, that must be both humidified and heated, in order to be destroyed. Medical tools are disinfected and than sterilized after use. The medical tools are sterilized 1-2x for a day. Common life cycle is 3 years approximately, but it depends on a treatment with such a tool. Unfortunately, it is not exception when paramedical departments have not got money enough to buy new medical tools and the life cycles of these medical tools are longer. The coatings can save basic material and increase the life cycles and the usage of the medical tools.

Steam sterilization has been performed with the help of Halina Doležalová in the Sterilization center of Hospital in Liberec in an autoclave PS 625 CHIRANA (chamber 600l) and GETINGE (chamber 400l) variable. This investigation were performed with coated and uncoated specimens. One sterilization cycle took approximately 1 hour and there has been made 60 cycles in altogether.

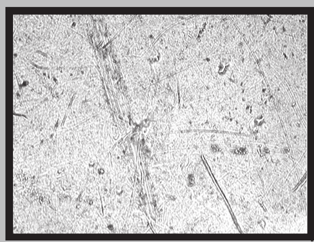

FIG. 6. The surface of the uncoated specimen 1.4301 before sterilization (300x).

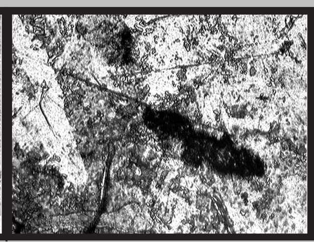

FIG. 7. The surface of the uncoated specimen 1.4301 after sterilization (300x).

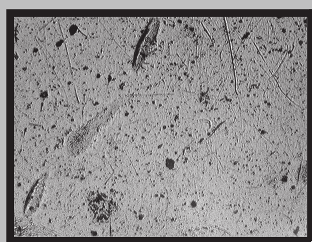

FIG. 8. The surface of TiAlN deposited on 1.4301 before sterilization (300x).

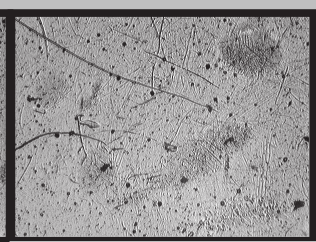

FIG. 9. The surface of TiAlN deposited on 1.4301 after sterilization (300x).

Sterilization's holding time was 20 minutes under the temperature 121°C and the pressure 105kPa. We have done photos of a surface of the coated specimens before and after the sterilization and photos of uncoated specimens before and after sterilization for comparison. FIG.6-9. You can see corossion on uncoated specimen after sterilization cyckles clearly, but there was no corossion on the coated speciment after sterilization.

Conclusion

One of many ways how to enhance useful value of the solid matter is positive influencing its surface properties. We may speak about the coating of substrat by protective surface that leads to compact system with size of cluster of a few nanometrs. The coatings for medical tools schouldn't have just good mechanical and optikal properties (dark layers TiAlN, TiCN - because bright light from the surface of surgical tools mustn't dirsturb the visual operating field of the surgeon). But this coating must be non-toxically for human body and must protect human body against nickel diffusion and save substrates from corrosion. We can say, that all of the considered PVD - coatings have shown a sufficient sterilization resistance and the necessary biotolerance which is required from the legislatosr for short-term applications of tools.

References

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