Research Journal of Material Sciences ISSN 2320–6055
Vol. **7(1),** 11-13, January **(2019)** *Res. J. Material Sci.*

Short Communication

Influence of substrate properties on titanium dental implant coating adhesion

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Available online at: www.isca.in, www.isca.me
Received 5th July 2018, revised 20th December 2018, accepted 10th January 2019

Abstract

In order to study the reactivity of titanium implant in the saliva environment in the presence of food, thin coating films of titanium were prepared. The dental implant was made using tantalum, molybdenum and copper substrate coated with titanium thin coating. The titanium coating films was deposited using radiofrequency magnetron sputtering reactive method. The adhesion characterization was made although the scratch test and observed using microscope. In the case of tantalum substrate, the periodic patterns appear inside the failure as a circle form.

Keywords: Adhesion, coatings, dental implant molybdenum, tantalum, copper, scratch test, radio frequency magnetron sputtering reactive.

Introduction

The dental implant formed by metallic substrate coated with titanium thin film was prepared using radio frequency magnetron sputtering reactive method. In order to prevent adhesion failure in saliva environment, the titanium coating was deposited on bulk molybdenum, tantalum and copper substrates. The objective of this work was to identity the best substrate with high titanium adhesion values. The scratch method was used for adhesion determination[1-5]. Scratch method was first developed by HEAVENS for adhesion of metal coatings[6]. He considered the critical regime reached when the film was completely eliminated in the scratch.

Benjamin and Weaver to characterize the adhesion of metallic films deposited on glass, have defined the critical load as the load for which the coating leaves on the sample a translucent trace to the light[7]. However, this argument cannot be applied in the case of metallic substrates that are not transparent. The scratch experiment is used to deform the interface of the coating-substrate system with a spherical indenter. The adhesion measurement of the coating is obtained by a critical charge determination. The scratch test results have demonstrated that the critical charge can be used as an adhesion measurement[6-9].

Methodology

Bulk copper, molybdenum and tantalum were used as substrate in this work. The size of rectangular substrates was 2cm x 1cm x 2mm.

Radiofrequency magnetron reactive sputtering devices: The experimental device (Figure-1) is mainly composed of: i. a pumping unit, ii. a vacuum enclosure, iii. a radiofrequency generator.

The pumping unit consists of primary pump with pressure ranged between 1.10^5 Pa and 0.1 Pa and an oil diffusion secondary pump which allows a limiting vacuum to be reached about $1.5.10^{-5}$ Pa. This is surmounted by one trap cooled with liquid nitrogen.

The vacuum chamber is cylindrical in shape with a diameter of about 0.80m and a height of 0.50m. Inside, a substrate-carrying anode may be optionally polarized. This is located at a distance of about 0.15m from the target which constitutes the cathode. The target used is titanium of purity 99.99%. It is rectangular, its length is 0.30m and its width of 0.15m. It is coupled to the radio frequency generator by means of a capacitance enabling a direct and negative voltage to be sent to the target. The target is placed on a plate which acts as an electrode.

The cathode is cooled by a water between 15°C and 20°C. The limiting pressure inside the enclosure does not exceed $1.5.10^{-5}$ Pa. Two PIRANI and Albert BAYER gauges are used to control the pressure in the vaccum chamber.

The argon gas is introduced using a micro valve. Argon are of high purity 99.997%. A thermocouple in Allumel-chromium is placed on the back of the substrate for the temperature control.

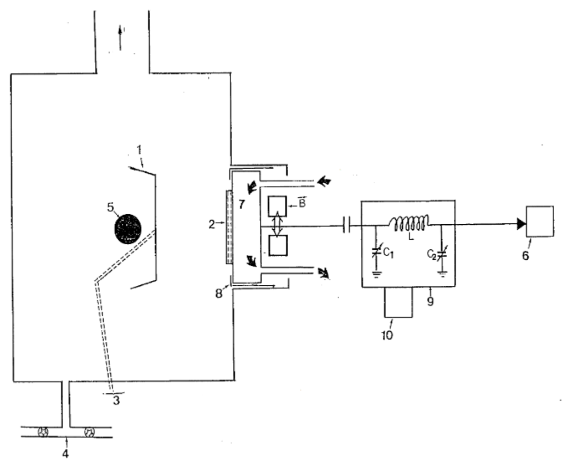

Figure-1: Radiofrequency reactive magnetron sputtering device.
1. Substrate plate, 2. Titanium target, 3. Thermocouple, 4. Micro vanne, 5. System of substrate plate, 6. Radiofrequency generator, 7. Target plate, 8. Blindage, 9. Impedence adaptator, 10. Voltmeter.

The radio frequency generator delivers a maximum power of 1000 W at 13.5 MHz. It has an output impedance of 50 Ω. The generator has two watt meters to measure incident and reflected power.

An impedance adapter interposed between the generator and the target is used to provide the maximum power to the target by reducing the reflected power. This adapter is equipped with a voltmeter which indicates the bias voltage of the target.

Sample preparation: The substrate used are copper, molybdenum and tantalum. The choice of these substrates was defined by the need to have different hardness in order to be able to characterize the adhesion of the titanium coatings as function of both coating and substrate mechanical properties. The copper was cleaned in 1% concentrated hydrochloric acid to remove the oxide layer which covers it. Molybdenum is cleaned in ortho-phosphoric acid. Tantalum is cleaned with acetone. Once cleaned, these supports are rinsed several times with distilled water and then ultrasonically cleaned in an alcohol bath. A mask is used during pre-sputtering to prevent the atoms torn from the target from polluting the substrates.

Operating procedure for titanium deposition: The prepared supports are fixed on a substrate holder in the vacuum enclosure. When the pressure in the enclosure reaches 1.5×10-5Pa, argon is introduced to the pressure of 6.5×10-3Pa. By throttling the communication valve enclosure - diffusion pump, the pumping rate once the gases are introduced into the chamber so as to achieve a stable pressure equilibrium. The plasma is then primed. Before any deposition, it is necessary to carry out a pre-sputtering for about ten minutes. After the cleaning of the target, a pumping is carried out until a residual pressure of 1.5×10^{-5} Pa is obtained.

Argon is again introduced under a pressure such that the plasma can start by adjusting the incident power to 1000w under the conditions of our experiments. In each experiment, the thermocouple is placed behind one of the metal substrates in order to determine the substrate temperature during deposition.

Adhesion scratch test: Experimental device consists of a balance of which one arm is connected to the point on which the load is applied. The end of the other arm is fixed to a counterweight as to maintain the balance in the absence of the load. The sample to be analyzed is placed on a small mobile plate which can move in the X direction or in the Y direction.

By means of two micrometric screws. A micrometric screw fixed on the sample carrier plate, is coupled to a motor rotating at a constant speed in order to maintain uniform displacement in the X direction during each experiment.

A is done by operating the second micrometer screw to allow the plate to move in the Y direction. Scratches are visualized during the experiment using an optical microscope or by illuminating the sample to be analyzed. Scratches are then observed on a scanning microscope and the difference in contrast on the micrographs allows us to distinguish the support from the coating. The point used in this scratch test is made of diamond with a radius of curvature of 18.10^{-6}m.

Results and discussion

Scratch scanning electron microscopic observations of coating failure mode: Titanium dental implant coatings on copper substrate: Figure-2 shows a micrography of adhesion failure of titanium film coating deposited on copper substrate at the critical load .when the coating loss is limited. Micrograph shows cohesive damage in the coating and some plastic deformation of the substrate. The observed damage inside the substrate were found to be in agreement with other authors[9].

Titanium dental implant coatings on molybdelum substrate: Figure-3 shows a micrograph of scratch test at the critical charge. Typical failure mode of titanium coatings was observed with periodical patterns. In this case, the elastic energy in the front of indenter is enough to give the necessary adhesion work to detach the film. The result has shown that the critical regime can be manifested by the formation of a stripe with straight edges. It may happen that the trace is not rectilinear but lined with periodic patterns.

Titanium dental implant coatings on tantalum substrate: The Figure-4 presents the results of scratch test for titanium coating deposited on tantalum substrate. At the critical charge, the film can detach in a ring around the point application and in front of the tip. The increase in the load can then cause the formation of successive annular folds.

These results are in agreement with BUTLER in case of a soft and ductile deposit[9]. According to Laugier, the coating in front of the tip undergoes a compression during which a certain energy is stored in front[6]. When this energy is sufficient to provide both the work necessary to elastically deform the film and the adhesion work, the tip moving gradually, the raised portion of the coating in front of the tip is pushed back, the film on the edge of the scratch being under stress state can deform plastically. At this point, the energy stored in front of the tip becomes minimal and the process begins again. This phenomenon shows a succession of periodic patterns on the edges of the trace. The energy required to elastically deform the film can be neglected in front of the adhesion energy. Scanning electron scratch of titanium thin films prepared with tantalum are illustrated in Figure-4.

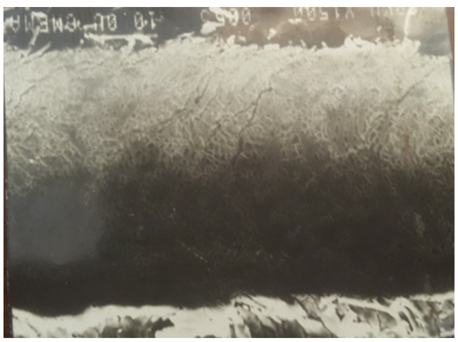

Figure-2: Electron microscopic micrographs of titanium scratch on copper at 5g.

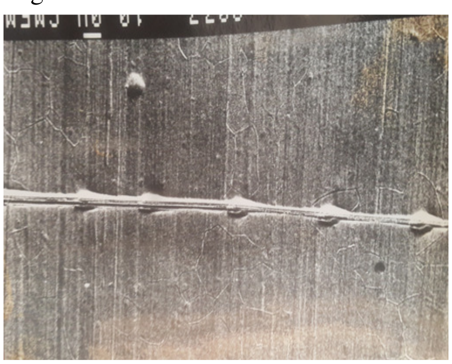

Figure-3: Electron microscopic micrographs of titanium scratch on molybdenum at 5g.

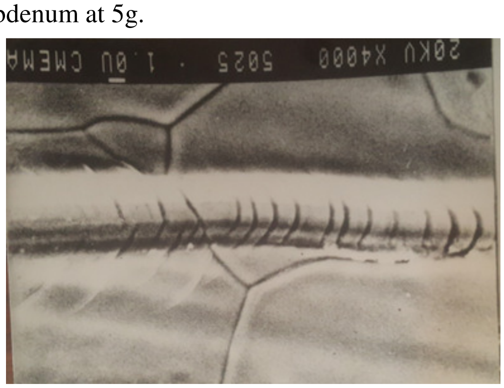

Figure-4: Optical micrographs of titanium scratch on tantalum at 5g.

Conclusion

Titanium thin coating films were prepared by radio frequency magnetron sputtering techniques in different metallic substrate in order to make dental implant with a little titanium content. The scratch test were used to characterize the adhesion applied on dental implant coated with titanium, a very typical period patterns were obtained related to the nature of substrate. The mode was can be related to the physical properties of both substrate and titanium films. The result was found to be in agreement with those obtained by other authors.

References

1. Timothée Nsongo (1995). Adhesion characterization of titanium and titanium nitride thin coatings on metals using the scratch test. *International journal of adhesion and adhesives*, 15(3), 191-196.
2. Nsongo Timothée (1987). Study of the structure and adhesion of titanium nitride thin films prepared by reactive sputtering. Doctoral thesis, Aix Marseille University France.
3. Oh See Ghee (1984). Le vide, Les couches minces, 220.
4. Hintermann H.E. (1981). Tribological and Protective Coatings by Chemical Vapor Deposition. *Thin Solid Film*, 84(3), 215-243.
5. Lewis B. and Anderson J.C. (1978). Nucleation and Growth of Thin films. Academic Press, New York.
6. Heavens O.S. (1950). Some factors influencing the adhesion of films produced by vacuum evaporation. *Journal de physique et le radium*, 11(7), 355-360.
7. Benjamin P. and Weaver C. (1960). Measurement of adhesion of thin films. *Proceedings of Royal Society*, *series A.*, 254, 163-176.
8. Goodman L.E. (1962). Contact stress analysis of normally loaded Rough Spheres. *Journal of Applied Mechanics*, 29(3), 515-522.
9. Butler D.W., Stoddart C.T.H. and Stuart P.R. (1970). The stylus or scratch method for thin film adhesion measurement: some observations and comments. *Journal of Physics D: Applied Physics*, 3(6), 877-883.
10. Timothée Nsongo, Hilaire Elenga, Bernard Mabiala, David Bilembi and Ferland Ngoro Elenga (2016). Adhesion of Clay Based Paint Coatings on Wood and Glass Substrates. *International Journal of Research in Environmental Science (IJRES)*, 2(6).
11. Laugier M.T. (1984). An energy approach to the adhesion of coatings using the scratch test. *Thin Solid Films*, 117(4), 243-249.