# Biomedical properties of tantalum coatings prepared by multi arc

W. Cai, Y. Cheng, Y.F. Zheng, H.T. Li, L.C. Zhao

School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China Email: weicai@hit.edu.cn

Keywords: TiNi alloy, ion-plating, tantalum, coatings, hemocompatibility

Abstract: Ti-50.6 at.% Ni shape memory alloy was coated with tantalum using multi arc ion-plating technique with the aim to increase its radiopacity and biocompatibility. The surface characteristics were investigated by X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM). The results of XPS survey spectra show that a thin oxide film are formed inside tantalum coating as a result of natural passivation of Ta in atmosphere. The hemocompatibility was evaluated in vitro by clotting time and platelet adhesion measurement. The results of our study showed that the clotting time of tantalum was higher than that of the TiNi alloys and no sign of accumulation and only slight pseudopodium was observed on the tantalum coatings, suggesting that the tantalum coatings can improve the biocompatibility of TiNi alloy.

# Introduction

TiNi alloys are known for their remarkable mechanical and chemical properties such as shape memory effect (SMA), superelasticity and anti-corrosion property. They have been found widespread applications in the medical field[1,2]. However, when small stents, guildwire and catheter are quite thin and space farther apart, the detection of the implantable devices or tools become very difficult [3]. Moreover, there is often concern regarding Ni allergies and the 50% nickel content of TiNi alloy[4,5]. So it is often more desirable to improve the implanting materials radiopacity and biocompatibility.

It is a simple and feasible method to coat TiNi alloy with the aim to improve its radiopacity and biocompatibility. The arc ion-plating technique can ensure a good banding strength for the implant life service in the implanting environment[6]. Tantalum possesses excellent corrosion resistance, chemical stability, high radiopacity and histocompatibility, it has been used to improve the biomaterials radiopacity and biocompatibility[7-9]. The lowest corrosion currents were observed when coupling nitinol with tantalum, which indicates that coating TiNi with tantalum can be considerably safe[10].

In this study, Ti-50.6 at% Ni alloy samples were coated with tantalum by multi-arc ion plating method in order to improve their radiopacity and biocompatibility. The aim of the present study is to investigate the surface characteristics and biocompatibility of TiNi alloy coated with Ta.

# Experimental

The experimental alloy have a composition of Ti-50.6 at.% Ni, and all samples were polished and then cleaned ultrasonically in acetone.

Surface of the air-exposed coating samples were characterized using a ESCA PHI500 spectrometer with a  $MgK_{\alpha}X$ -ray source. A Nano-ScopeIII Atomic Force Microscopy (AFM),

2350 PRICM-5

Digital Instruments, Inc. was used for surface observations of samples. The thromboresistant property of the materials was evaluated using an adult fresh blood by the kinetic clotting method. In vitro platelet adhesion test was subsequently performed to identify the blood compatibility of the tantalum coatings according to the method presented in the paper<sup>[11]</sup>.

### Results and discussion

# 1. Surface characteristics of the Ta coatings

It is well known that the interaction between blood and biomaterials occurs at their interface, the blood compatibility of the biomaterials depends strongly on the surface characteristics. So in our study we investigated the surface characteristics of the materials.

Fig. 1(a) is the XPS survey spectrum of the surface of the TiNi sample coated with tantalum after 1 min argon sputtering. Four major tantalum peaks observed in the spectra at 24.1, 230.6, 240.3 and 459.1eV positions are corresponding to Ta4f, Ta4d3, Ta4d5 and Ta4p<sub>1/2</sub>, respectively[12]. O signal is also detected on the surface. The survey spectrum of Ta coating after 5 min sputtering is showed in Fig.1 (b). It can be seen that only Ta peaks appear at different binding energies which correspond to Ta4f, Ta 5p, Ta4d<sub>5/2</sub>, Ta4d<sub>3/2</sub>, Ta4p<sub>1/2</sub>, etc. this results show that pure tantalum layer is reached on the surface of TiNi alloys. This result indicates that a thin tantalum oxide film is formed on the outmost surface of the Ta coating as a result of natural passivation of Ta in atmosphere, as expected.

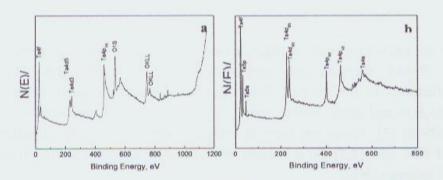


Fig.1 Typical XPS survey spectra of of TiNi alloys coated with Ta after argon sputtering for (a) 1 min; (b) 5 min

Fig.2 shows the AFM image and section analysis of the uncoated and coated TiNi alloys. The surface of the sample after coating is relatively smooth compared to that of the uncoated sample. The roughness parameters were determined from AFM Nano-Scope III software. The root mean square (RMS) values of the coated and uncoated samples are 12.533 nm and 31.043 nm, respectively.

# 2. Hmocompatibility

The optical density vs. time curves of the coated and uncoated TiNi alloys are shown in the fig.3. The optical density represents the relatively concentration of the hemolyzed hemoglobin when the materials contact with the blood for a predetermined time. The higher the absorbance the better the thromboresistance is. It can be seen from the figure that the clotting tendency of the coated TiNi alloy is lower than the uncoated one. The time at which the absorbance equals 0.1 is generally defined as clotting time. The longer the clotting time, the better the hemocompatibility. Obviously, the clotting time of the TiNi alloy coated with tantalum is much higher than that of the uncoated sample, which indicate that the tantalum coatings can improve the hemocompatibility of TiNi alloy.

As platelet activation can elicit a variety of physiologic cellular responses including shape

change, release of granule contents, and initiation of aggregation, platelets have been extensively

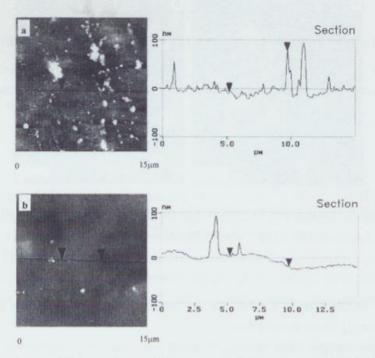


Fig.2 Surface section analysis of the uncoated(a) and coated(b) TiNi alloys

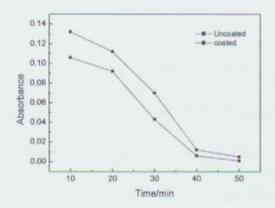
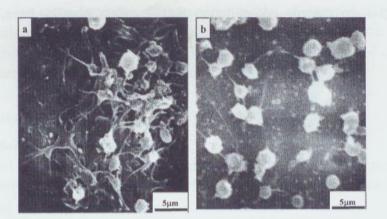


Fig.3 Optical density vs. time curves of the coated and uncoated TiNi alloys

studied due to their role in thrombogenesis when the blood makes contact with the materials surfaces. In this study, we use in vitro platelet adhesion experiments to evaluate the preliminary blood compatibility of the materials. Fig.4. shows the morphology of the platelet adhered on the coated and uncoated samples. There are many fully spread and partially activated platelets (pseudopodia) exist on the surface of the coated samples, but no aggregation to form thrombi is observed. While pseudopodia extension, platelets aggregation and interlocking is found on the surface of the uncoated samples, indicating that the platelets are completely activated. The observation is consistent with the results of the clotting time measurement, verifying the fact that the hemocompatibility of the TiNi alloy coated with tantalum is better than that of the uncoated one.



PRICM-5

Fig.4 The platelet morphologies on the surface of the uncoated (a) and coated (b) TiNi alloys

# Conclusions

- A thin tantalum oxide film is formed on the outmost surface of the Ta coating as a result of natural passivation of Ta in atmosphere.
- The surface of the sample after coating is relatively smooth compared to that of the uncoated sample.
- The hemocompatibility of the TiNi alloy coated with tantalum is better than that of the uncoated one.

# References

- [1] T. Duerig, A. Pelton and D. Stockel: Mater. Sci. Eng Vol. A273-275 (1999), p. 149
- [2] S. Shabalovskaya: Bio-Med. Mater. Eng. Vol. 6 (1996), p. 267
- [3] T. W. Duerig, D. E. Tolomea and M. Wholey: Min Invas Ther & Allied Technol Vol. 9 (2000) p.235
- [4] J. Ryhänen, E. Niemi, S. Serlo, E. Niemela, P. Sandvik, H. Pernu and T. Salo: J. Biomed. Mater. Res Vol. 6 (1997), p. 451
- [5] D. J. Wever, A. G. Veldhuizen, J. Vries, H. J. Busscher, D. R. A. Uges and J. R. van Horn: Biomaterials Vol. 19 (1998), p. 761
- [6] H. Randawa and P. C. Johnson: Res & Devel Vol. 2 (1987), p. 173
- [7] R. Venugopalan and C. Trepanier: Min Invas Ther & Allied Technol Vol. 9 (2000), p. 67
- [8] D. E. Hodgson and S. A. Smith: Proceedings of the Second International Conference on Shape Memory and Superelastic Technologies. 1997, p. 461
- [9] A. Warren: Radiol Vol. (1970), p. 327
- [10] H. Zitter and H. Plenk: J. Biomed. Mater. Res Vol. 21 (1987) p. 881
- [11] N. Huang, P. Yang, X. Cheng: Biomaterials Vol. 19 (1998), p. 771
- [12] F. Moulder, W. F. Stickle, P. E. Soblo, K. D. Bomben: Perkin-Elmer Corporation, Eden Prairie, MN, 1992, p. 171