Surface Characterization of Co-Cr Alloy L605 Electropolishing in 15 vol % Phosphoric acid

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Statement of Purpose: Electropolishing has been used to form a surface oxide passivation layer, and smoothen the surface, to improve biocompatibility. For implants such as stents or vena cava filters, the material needs to be resistant to corrosion; the presence of a passive oxide layer is necessary for long term biocompatibility. The current study was undertaken to investigate the surface characteristics and the biocompatibility of Co-Cr Alloy L605 as a function of the electropolishing parameters.

Methods: 15 x 15 mm square specimens were first mechanically polished with 600 grit SiC sand paper and cleaned. The current plateau was determined by profiling the characteristic I-V curve for the specific temperature (45°C, 35°C, 25°C and 0°C) and the concentration of phosphoric acid (85 vol %, 50 vol %, and 15 vol %). After the current plateau was established, the test specimens were electropolished by varying the polishing duration and temperature. Surface roughness was quantified by Atomic Force Microscopy (AFM), contact angle was determined using the sessile drop method, and the surface chemistry was determined by X-ray Photoelectron Spectroscopy (XPS). The cytotoxicity tests of the electropolished Co-Cr Alloy L605 is currently under investigation, in accordance with ISO 10993-5 standards.

Results: A distinct current plateau was obtained only for 15 vol % acid concentration, at temperatures of 35°C, 25°C and 0°C. The current at the plateau was determined to be 0.39A and the voltage range was between 2.3-4 V. The electropolishing rate (EP rate) was found to be heavily influenced by the temperature of the acid bath. The EP rate decreased proportionally as the bath temperature decreased. The Root Mean Square (RMS) surface roughness of the specimens electropolished at the different temperatures was compared. The RMS value decreased as the internal bath temperature decreased. The AFM survey revealed a wavy morphology and the presence of small spherical etch pits which were detected under the stereomicroscope, as seen on Figure 1. This finding is in accordance with Placko et al. [1]. As can be seen on Figure 2, the contact angle decreased as the EP temperature decreased. This finding can be correlated with the AFM finding. From the AFM analysis, the decrease in the RMS was inversely proportional to the increase in the contact angle. This finding indicated that the specimen surface became more hydrophobic after electropolishing. For stents, it is essential for the material surface to exhibit resistance to cell adhesion. According to Ponsonnet et al. [2], cell adhesion and spread were significantly greater on a hydrophilic than a hydrophobic surface. Therefore, electropolishing prepares the surface to resist cell adhesion by producing a smooth surface. The XPS analysis revealed that three distinctively different oxides were found on the surface: Cr-oxide, W-oxide, and Co- oxide. Further investigation by AES analysis revealed that the specimen surfaces were primarily Cr-rich.

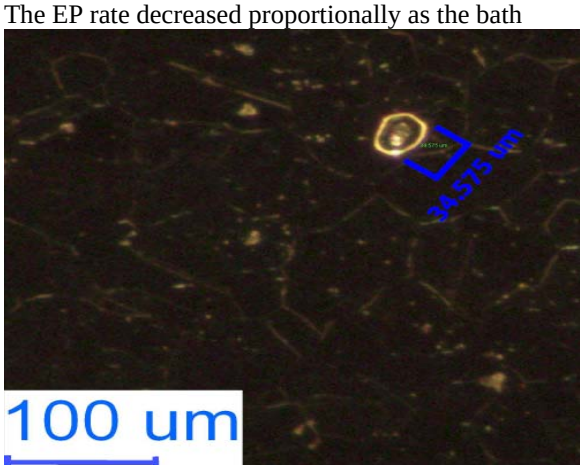

Figure 1 300X magnification survey of a spherical etch pit found on L605 sample electropolished at 0°C for 3min.

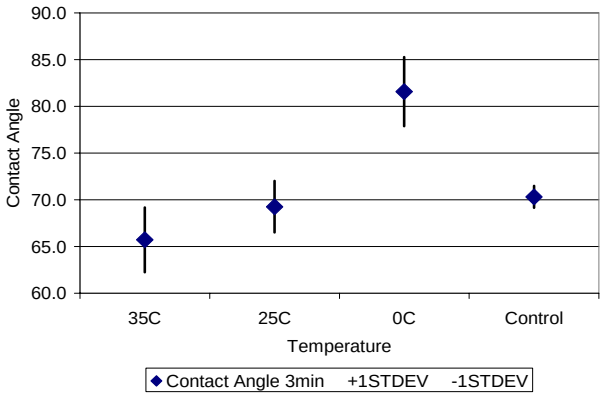

Figure 2 Temperature dependent contact angle comparison between test specimens.

Conclusions: Electropolishing was found to alter the surface characteristics. The presence of the current plateau on the I-V curve was influenced heavily by temperature and acid concentration. Electropolishing can potentially produce smooth hydrophobic and passive surfaces. However, etch pits and small regions of the surface that do not react to the electropolshing process may occur. Therefore, a close monitoring of the process is required in order to generate a smooth passive surface, free from pits and un-reactive regions. Biocompatibility testing is currently being conducted on the electropolished L605 specimens.

References:

[1] Placko EH. Int J Oral Maxillofac Implants. 2000;15:355-363.
[2] Ponsonnet L. Mater. Sci. Eng., C. 2003;23:551-560.