Effects of temperature and cleaning on the surface characteristics of cardiovascular stainless steel stents

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Statement of Purpose: One issue facing the development of polymer coated cardiovascular stents is the adherence of hydrophobic polymers to the stainless steel surface. Particularly with drug/polymer coated stents delamination may cause an increase in the release rate of the drug relative to the intended stent design. It is thought that changes in the chemical species with time on the stent surface are ultimately responsible for the delamination. Experience shows that stents that are held for a period of time before coating application perform different than newly cleaned stents. The purpose of this study was to determine what changes are happening on the surface of new clean stents.

Methods: Stainless steel stents were cut from stainless steel tubes, passivated, and cleaned. For part one of the study, one group was exposed to elevated temperature, one to elevated humidity, and two groups were controls held in separate ambient environments. Stents from part one were analyzed once a week for six weeks and then at week nine for a total of eight time points. For part two of the study, during the cleaning process steps were taken to maximize and minimize both the carbon compound exposure and nitrogen compound exposure to create four experimental groups for comparison with one control group with no change to the cleaning process. Stents from part two were analyzed once every three weeks for six weeks for a total of three time points. All XPS spectra were taken on a Kratos Axis-Ultra DLD spectrometer. This instrument has a monochromatized Al Kα X-ray and a low energy electron flood gun for charge neutralization. ToF-SIMS spectra were acquired on a IonTof ToF-SIMS 5 spectrometer using an 25 keV Bi_3^+ ion source in the pulsed mode. Along with XPS and ToF-SIMS the sessile drop contact angle was measured.

Results: XPS results from part one of the study show an increase in oxygen and oxidized carbon species over time for all conditions. The elevated temperature showed a wide separation between the nitride and organic nitrogen species with a significantly lower level of nitride nitrogen (Figure 1). ToF-SIMS data shows an increase in the CN and CNO containing fragments. This suggests nitrogen from the steel is reacting with the advantageous hydrocarbon species from the environment. The source of the nitrogen is hypothesized as coming from the steel because atmospheric nitrogen is inert and the surface nitrogen is statistically constant over the course of the experiment. For both parts one and two, oxidized carbon increased with time for all groups. Figure 2 shows the high resolution C 1s peak fit results for OC=O for all groups from part two of the project. Since both control groups from part one behaved the same it was hypothesized that the cleaning procedure was an important variable in the stent manufacturing process. Part two of the study was designed to modulate the exposure of the stents to carbon and nitrogen. However, results from part two of the study show that there is no statistical difference between the different cleaning procedures. For both parts of the study the contact angle decreases with time.

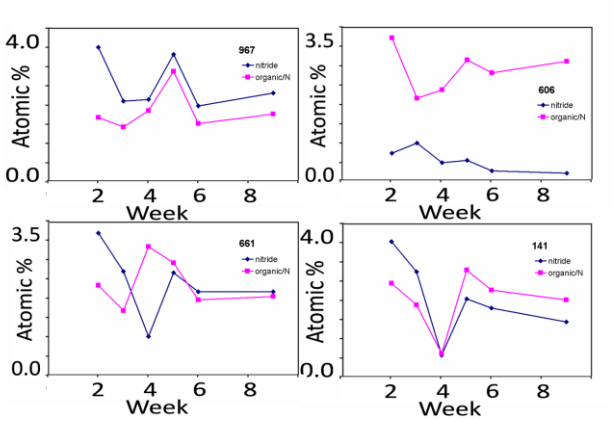

Figure 1. XPS high resolution N 1s peak fit trend. The ping line is organic nitrogen and the blue line is nitride from part 1 of the study.

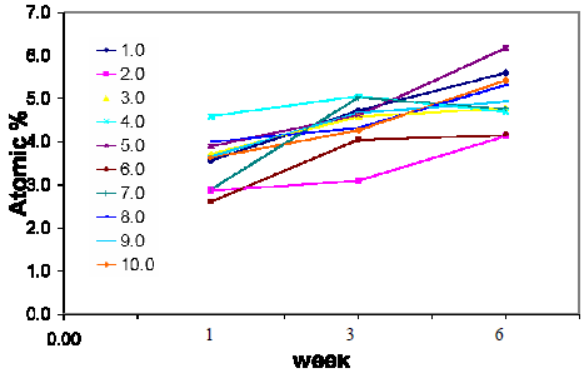

Figure2. XPS high resolution C 1s peak fit trend for the carbonyl functional group from part 2 of the study. The only significant change across all groups is an increase with time.

Conclusions: Part one of the study suggests that temperature is the most significant variable affecting surface characteristics and that possibly the metal surface is catalyzing the oxidation reaction with advantageous hydrocarbons and nitrogen from the bulk steel. Part two of the study shows that the variations in the cleaning procedure have no effect on the surface characteristics of the stent. These data suggest that the surface of the stent is evolving weeks after the manufacturing process.