Robbins Professor of Sustainable Manufacturing

Annual Report – July 2020

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The Robbins Professor of Sustainable Manufacturing supports high risk and potentially high payoff research by Dr. Friedrich and his students in sustainable manufacturing. During the past year, work has focused on further developing inherently antibacterial orthopedic implant surfaces and the breakthroughs to sustainably manufacture these new implants faster and less expensively than currently used processes. The work is now nearing commercialization requiring a systematic evaluation for FDA application.

The work reported here was funded by the Robbins Professorship during 2019-2020. We were granted US Patent 9,376,759 in June 2016 “Compositions, Methods and Devices for Generating Nanotubes on a Surface” to make the implant surfaces inherently antibacterial by integrating nano-silver particles on and inside the nanotubes in the same process that generates the nanotube surfaces thereby reducing cost, complexity, and hazardous materials. This process for fabricating nanotubes into the surface of titanium alloy implants is considered by industry to be the favorable process over one based on highly toxic hydrofluoric acid.

Although on-campus lab work was shut down for approximately four months during the spring 2020 semester, the work continued.

- A ME-EM senior capstone design group was sponsored during 2019-20 whereby the students developed a software package that simulates an implant production line for different implants, different production rates, and the changing chemistry of the process to better ensure quality control of the resulting implant surfaces.
- We have demonstrated nanotube surfaces on 3D printed implants solidified by either electron-beam sintering or by laser sintering of titanium alloy powders suitable for orthopedic implants. Both are mainstream 3D printing processes.
- We have demonstrated our electrochemical anodizing process on three types of spinal implants and machined implants used to stabilize long bones, such as the femur.
- We have demonstrated a process with an acceptable range of processing parameters that is necessary for eventual regulatory approval.

Background

Our orthopedic implant surface technology is based on a safe, low-cost 3-D electrochemical fabrication platform for etching nanotubes into the surfaces of titanium orthopedic implants, including 3D powder laser and electron beam sintered implants, as shown in Figure 1.

The 3-D electrochemical etching process uses low cost and non-hazardous materials, requires minimal process equipment and maintenance, is environmentally safe, and requires less energy than current surface coating technologies that deposit materials at high temperatures and therefore have thermal mismatch resulting in a brittle surface.

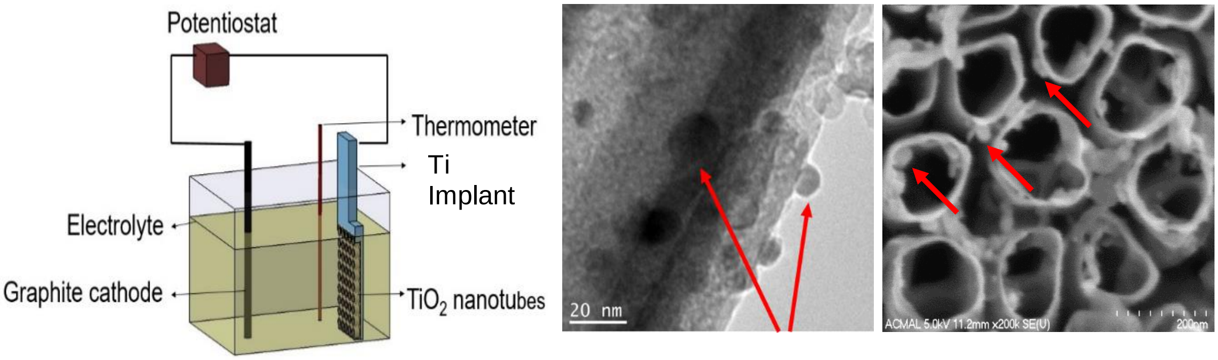


Fig. 1. Simple DC anodization with non-hazardous electrolyte etches surface leaving nanotubes, in as little as 10 minutes. Very small addition of silver compound (patented) produces nano-silver spheres decorating nanotubes in the same concurrent process.

Toward Commercialization

For the nanotube fabrication process to be approved, it must be shown that the process is understood and under control. For small numbers of very specialized implants (fewer than 100 per year), the process can take place in a one-off or small batch mode. However, for large scale production, such as hip and knee components on the order of hundreds-of-thousands per year, the process must be continuous and automated.

The Robbins funding has allowed continuous work and the fabrication process is understood and documented. We can even adjust the nanotube diameter by 10 nanometers by adjusting the anodizing voltage. To put this into perspective, 10 nanometers is approximately one-thousandth the diameter of a hair, or less than one-half of one-millionth of an inch. The chemistry of the electrolyte changes as parts are anodized and that change is a function of total area anodized. If different types of parts with different surface areas are processed together, a control system must be able to predict when the electrolyte chemistry needs to be adjusted. This is satisfied by adding the proper amount of fluorine to the electrolyte at the appropriate times, prolonging its life and reducing the waste stream.

Senior Capstone Design

Erika A Carne, Kristine E Fink, Drew D. Marion, Kassity K Swanson, Ben T. Wood
Dr. Jaclyn Johnson - Advisor

A Senior Capstone Design team was sponsored in 2019-2020 to develop a virtual processing system that can monitor the various parameters of the process and alarm or warn when action must be taken to assure consistent parts. The implants chosen for the simulation are a hip stems, a hip cups, and bone screws as shown at right. While these parts are not shown at the same scale, the surface area of each is vastly different.

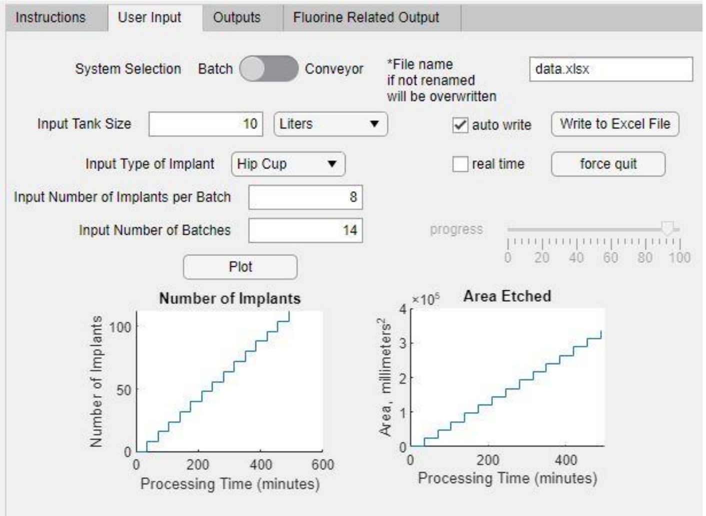

Hip Implant


Hip Cup Implant


Bone Screw Implant

Because of the real-time complexity of the process, it was necessary for the students to develop a control flow chart with calculations at every step and decision point. The simulation software must output variables including number of implants and surface area already processed, the number of implants and surface area in process, the removal of electrolyte from the process due to clinging to the finished parts as they leave the anodizing tank, and the fluorine that is chemically bound to the nanotubes themselves.

The students wrote the simulation software to predict the process, provide real-time in-process information to an automated control system, and to demonstrate to orthopedic companies that this process can be controlled in high-volume manufacturing. An example of one of the input-output screens of the simulation is at right.



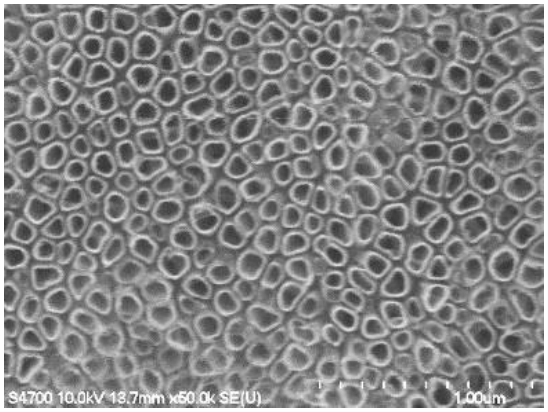
System Selection: Batch
*File name if not renamed will be overwritten: data.xlsx
Input Tank Size: 10 Liters
☑ auto write
Input Type of Implant: Hip Cup
☐ real time
Input Number of Implants per Batch: 8
Input Number of Batches: 14

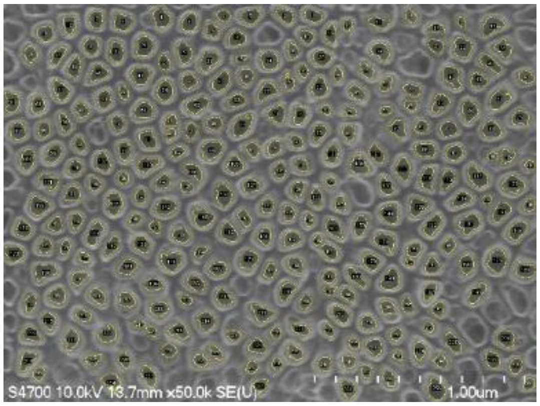
Process Qualification
Jabez James – MS student

A requirement for commercialization and regulatory compliance is that the resulting nanotube characteristics can be quantified so that acceptable nanotubes are not “in the eye of the beholder”. Further, the process limits that create acceptable nanotubes must be established so that effective tolerances may be placed on the processing parameters.

For this aspect of the work, the processing parameters that control nanotube fabrication were varied over several ranges and the results examined in SEM. The critical processing parameters are anodizing voltage, anodizing time, and the fluorine concentration in the electrolyte. Voltage and time were varied around accepted values to determine processing limits. While such an analysis provides qualitative results, the acceptable and not-acceptable nanotubes must have a numerical metric attached to them.

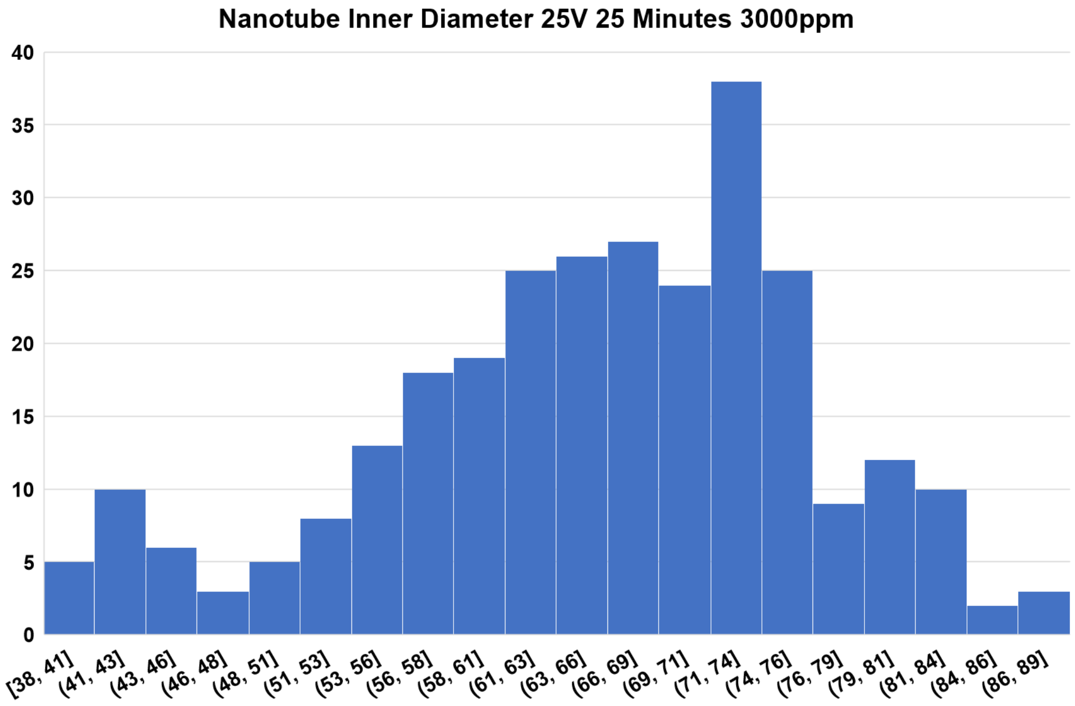
Image processing is being used to quantify nanotube images generated by scanning electron microscopy. The image to the right was analyzed using the free software ImageJ originally developed by the National Institutes of Health for analyzing biological images. The software identifies the region of the inside diameter of each nanotube. A processed image is shown at lower right. Each full nanotube is identified by a number with an automatically calculated area in square-nanometers. Using this information, an equivalent inside diameter of a round nanotube is calculated. From this analysis, the density of nanotubes (number of nanotubes per square micrometer of image) is quantified, as is the average nanotube inner diameter (nanotube diameter is linked to how strongly bone bonds to an implant surface and how effective is its anti-bacterial characteristic). Other metrics can be generated including the lateral aspect ratio of each nanotube (the ratio of the major to minor axes of a best-fit ellipse of the inner diameter), and a metric termed circularity (how close to a perfect circle is each nanotube).





Because the nanotubes are of different sizes and shapes, this information is best presented as histograms. The nanotube inner diameter histogram is shown on the next page as an example.

From this, the most common nanotubes are 71 to 74 nanometers in inside diameter, and many are smaller which is preferable to larger diameters for antibacterial effectiveness.



Conference Presentation

Craig Friedrich, Shuo Wang, Adam Francis, Erin Baker, "Mechanical Integrity of MRSA Antibacterial Nanotube Surfaces", podium presentation at International Society for Technology in Arthroplasty, October 2019, Toronto, Canada.

Published Abstracts

Craig Friedrich, Shuo Wang, Adam Francis, Erin Baker, "Mechanical Integrity of MRSA Antibacterial Nanotube Surfaces", Orthopaedic Proceedings Volume 102-B, Issue SUPP_2, 01 Feb 2020.