Laser Processed Condensing Heat Exchanger Technology Development

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The reliance on non-permanent coatings in Condensing Heat Exchanger (CHX) designs is a significant technical issue to be solved before long-duration spaceflight can occur. Therefore, high reliability CHXs have been identified by the Evolvable Mars Campaign (EMC) as critical technologies needed to move beyond low earth orbit. The Laser Processed Condensing Heat Exchanger project aims to solve these problems through the use of femtosecond laser processed surfaces, which have unique wetting properties and potentially exhibit anti-microbial growth properties. These surfaces were investigated to identify if they would be suitable candidates for a replacement CHX surface. Among the areas researched in this project include microbial growth testing, siloxane flow testing in which laser processed surfaces were exposed to siloxanes in an air stream, and manufacturability.

Nomenclature

AQM = air quality monitor
CHX = condensing heat exchanger
DMSD = dimethylsilanediol

D4 = octamethylcyclotetrasiloxane D5 = decamethylcyclopentasiloxane

ECLSS = environmental control and life support system

FLPS = femtosecond laser surface processing

HX = heat exchanger

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LP = laser processed PDMS = polydimethylsiloxane TMS = trimethylsilanol

I. Introduction

Aspacecraft's environmental control and life support system (ECLSS) because they provide air cooling to critical spacecraft components and occupants. Additionally, CHXs recycle up to 50% of the reclaimed water on a spacecraft, an essential function of long duration space exploration and a critical function of closed-loop life support. Current CHX's rely on a hydrophilic and microbial growth resistant coating to gather condensate and ensure condensing surfaces are limited in microbial growth. Three problems with this coating have been continually observed throughout the Space Shuttle and International Space Station (ISS) Programs. First, although measures are taken to preserve a coatings' lifetime, the coatings still slough off over time. This causes the CHX to lose its hydrophilicity, decreasing its efficiency and increasing its susceptibility to microbial growth. As a result, the CHX must be recoated. This recoating is an expensive and time consuming process requiring the unit to be uninstalled and returned to Earth for recoating. While this procedure is manageable for the ISS, recoating of CHX cores will not be feasible on the long duration, deep-space missions. Second, the sloughed off coatings frequently become trapped in the air/water separator causing the separator to become clogged and ineffective at removing condensate from the heat exchanger.

The third major problem with current CHX technology is thought to be an adverse reaction caused by the hydrophilic coating and siloxanes in the air. It is thought that these siloxanes catalyze with the hydrophilic coating and form dimethylsilanediol (DMSD). DMSDs are water soluble and dissolve into the condensate water from the CHX. Once in the condensate, these contaminates are removed by the multi-filtrations beds on the ISS and breakthrough occurs at an accelerated rate. Their lifespan was designed for two years, however, because of the accelerated degradation from DMSD removal, their lifespan is reduced to about one year.

Ultimately,a new coating is needed to enable longer duration spaceflight and reduce upmass/downmass. The Laser Processed CHX (LP-CHX) project aims to solve these problems through creation of a high reliability CHX and laser processed CHXs. It was originally desired that a hydrophilic LP-CHX be designed to replace current CHXs.

II. Overview of Laser Processing and Materials Laser Processed

Femtosecond lasers are a type of ultrashort pulse laser and are commonly used for LASIK eye surgery or laser tattoo removal. When femtosecond laser surface processing (FLSP) is applied to a metallic surface, micron/nanoscale features are created, which can be hydrophilic in nature. Over the past decade, FLSP has rapidly grown into a viable surface functionalization technology with a wide range of applications, including, enhancing heat transfer^{1,2}, reducing drag ³, and producing antibacterial surfaces ⁴. Multiscale surface structures fabricated by FLSP are typically characterized by a micron-scale, mound-like structure covered by a layer of nanoparticles^{5–7}. It is the specific interplay between the micron- and nano-scale roughnesses's that accounts for the surface properties. Figure 1 is a picture of self-organized structures typically created through laser processing of stainless steel.

FLSP surfaces are unique as they are physically created by removal/reorganization of substrate material. Similar

surfaces can be created through combinations of lithography, atomic layer deposition, chemical vapor deposition, physical vapor deposition, etc., but are created through the addition of material. Therefore, laser processing potentially provides more robust and stable micron/nanoscale features because they are created in the substrate material versus through the addition of material. FLSP also results in the creation of micron and nano-scale surface features in a single processing step, while multiscale features produced using other techniques require multiple, often complex, processing steps. Additionally, hydrophobic surfaces can be created by introducing low surface energy materials such as hydrocarbons, PDMS, or siloxanes to the laser processed surfaces. The original intention of this research was to create a hydrophilic LP-CHX that could

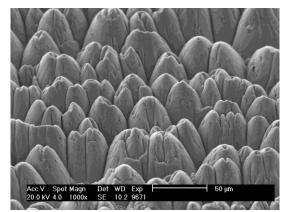


Figure 1. Stainless steel laser processed surface.

potentially serve as a 1:1 dimensional replacement for the current ISS CHXs. However, upon further research, it was discovered that certain surfaces, particularly hydrophobic surfaces, have antimicrobial properties. Additionally, experiments performed at Johnson Space Center demonstrated that these hydrophilic laser-processed surface did not remain hydrophilic after exposure to siloxanes, a class of common ISS air trace contaminants. This research is described further in section III.

Two materials were of primary focus for initial studies, stainless steel and silver. Instrumental to the laser

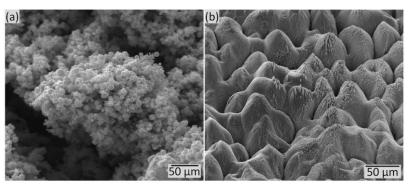


Figure 2. Self-organized structures on silver using single pulse femtosecond laser surface processing at (a) high laser fluence (peak fluence – 3.40 J/cm²; 1063 pulses/area), and (b) low laser fluence (peak fluence – 0.34 J/cm²; 1600 pulses/area). Note nanoparticle cluster formation at high laser fluence.

processing of these materials was the collaboration between University of Nebraska-Lincoln's (UNL) Laser Science Engineering Research (LaSER) lab and Johnson Space Center. Stainless steel has commonly been processed by the LaSER lab and was well understood before work on the LP-CHX started.⁵⁻⁹

Prior to this project, the LaSER lab had not applied FLSP to silver and initial processing of silver using traditional FLSP techniques had limited success. Figure 2 includes SEM images of two unique types of surfaces created on silver using traditional FLSP techniques. The structures in Figure 2a are silver micron/nanoscale particle clusters attached to the surface of silver and were developed using high laser fluence. These structures are composed completely of micro/nanoparticles, with no bulk microstructure and are therefore expected to not be as durable as other FLSP structure types. The micro/nanoparticles are created during the femtosecond ablation process and are

sintered onto the sample surface during processing. structures in Figure 2b are mound-like structures with less nanostructure than most common FLSP structure types. A major downside of using traditional FLSP on silver is the time required for processing. Due to the unique FLSP parameter requirements to create structures on silver, the process was less efficient than is typical for processing other materials such as nickel or stainless steel. For example, using the current research grade lasers (6 mJ pulses at a 1 kHz repetition rate) available in the LaSER lab a 60 square inch area (6"x10"), would take approximately seven days (24 hours per day) of processing for the structures in Figure 2a and 17 days (24 hours per day) for the structures in Figure 2b. This method of processing would be time prohibitive for manufacturing of a full scale CHX or even carrying out some of the research needed to prove the effectiveness of the technology for the CHXs.

In order to functionalized silver using FLSP, two new methods of processing were investigated: dual pulse FLSP and FLSP direct writing. Each of these techniques has a number of advantages and disadvantages and result in unique structure types further described in this section. Results on anti-microbial properties, processing speeds and other manufacturing concerns will be considered when choosing a technique for future research on application of FLSP to the CHXs.

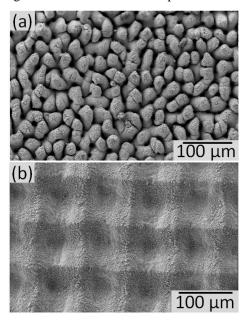


Figure 3. Self-organized structures on silver using (a) dual pulse FLSP and (b) femtosecond direct write processing of silver.

The dual pulse technique consists of splitting the femtosecond pulses into two pulses that are separated in time by < 1 ns. A variable delay arm is used to control the temporal spacing between the two pulses, and the pulses are collinear when focused onto the sample surface. The dual pulse FLSP technique takes advantage of the time dependent processes associated with femtosecond pulse interaction with materials in order to maximize the amount of energy deposited in the material.

Due to the material properties of silver, more energy is needed to drive self-organization processes than is need for other materials such as nickel or SS. The LaSER lab is still investigating the critical material properties that determine the amount of input energy needed to drive self-organization in different materials. When femtosecond pulses are focused onto a sample in open atmosphere

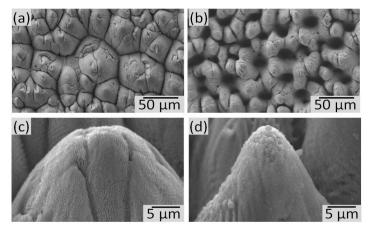


Figure 4. SEM images of (a) and (c) nanoparticle-coverd pyramids (b) and (d) above surface growth mounds created on stainless steel substrate. Images taken viewing (top row) normal to the surface and (bottom row) at a 45 degree angle.

a plasma is formed in the air above the sample, once a threshold intensity is achieved. This plasma can serve to shield the surface, limiting the amount of energy deposited in the material. The dual pulse technique allows more energy to be deposited in the material by reducing the peak intensity (same total energy split between two pulses) and limiting plasma shielding. Figure 3a is an SEM image of silver which has been processed using dual pulse FLSP with the energy split evenly between the two pulses and a 135 ps delay between pulses. The dual pulse FLSP structures presented in Figure 3a are similar to the types of structures created on nickel or stainless steels using traditional FLSP.^{7,10,6,8}

The second method for laser processing silver that is being investigated is called FLSP direct writing. With this technique, femtosecond pulses are focused to a very small spot (a few microns to tens of microns; much smaller than the structures being produced) and the sample is raster scanned in a grid pattern to preferentially remove material creating peaks and valleys. Figure 3b is an SEM image of a direct writing pattern. This process is commonly used in industry to accelerate laser processing of materials and is more compatible with current industrial lasers than the self-organized FLSP techniques.

Two major types of patterns on stainless steel and two types of patterns on silver were created for the work presented in this paper. On stainless steel, pyramid-like structures (Figure 4a and c) and mound-like structures (Figure 4b and d) were created. For this set of samples the mounds were created using a peak fluence of $7.09 \, \text{J/cm}^2$ and $409 \, \text{pulses/area}$, and have an average structure height of $80 \, \mu \text{m}$. The pyramids were created using a peak fluence of $0.26 \, \text{J/cm}^2$ and $14,925 \, \text{pulses/area}$, and have an average structure height of $47 \, \mu \text{m}$. The silver samples created for this study, were produced prior to the development of the dual pulse and direct writing techniques. The two types of structures on silver produced for this sample set are presented in Figure 2a and b.

III. Siloxane Flow Loop Testing of Laser Processed Surfaces

Because the intended future use of this research is to create a longer-lasting, contaminant resistant CHX surface, it is essential to test the surface attributes. A siloxane flow loop was built at Johnson Space Center (JSC) to test laser processed coupons in a controlled environment to determine the effect of siloxanes on the wettability of these surfaces. This testing monitors the change in contact angle, a method of measuring surface wettability. The changes in the surface properties over time when exposed to contaminants offer insight into how these surfaces might change in a space vehicle environment. This testing focused on using only stainless steel samples for this study.

A. Test Set-Up

The siloxane flow loop is a simple open flow loop system. Charcoal filters are located at both the inlet and outlet, with a fan driving the system between 30 and 200 CFM. The siloxanes used are a liquid mixture of three siloxane species, injected into the airstream using a syringe pump at ISS-like concentrations (Table 1). Target test concetrations were adjusted from measured ISS concentrations to account for loss of siloxanes in test environment (air grab samples).

Table 1. Concentrations of TMS, D4, and D5 in ISS and test environments.

	TMS (mg/m ³)	D4 (mg/m ³)	D5 (mg/m ³)	Total Mixture (mg/m³)
ISS Concentrations	0.30	0.11	0.76	1.17
Target Concentrations	0.28	0.10	1.71	2.09



Figure 5. a) Laser processed louvers b) non-processed lovers in louver holder.

The siloxane species used are decamethylcyclopentasiloxane (D5), octamethylcyclotetrasiloxane (D4), and trimethylsilanol (TMS) and were chosen because they are the most common ISS atmosphere siloxane constituents. The siloxanes were injected by a syringe pump then distributed in the airflow by the fan and flowed across the test article cassette. Figure 5 shows the processed louver samples and the louver cassette.

UNL provided two types of stainless steel louver samples: mounds (Figures 4b and d) and pyramids (Figures 4a and c). The samples were stacked alternatingly in the cassette to ensure equitable testing environments. Air quality measurements were not taken during this testing, and so test points were run with and without the injection of siloxanes as a control.

B. Measurement Methodology

Contact Angle (CA) measurements were made using photography and an open source scientific image processing software called ImageJ. Photos were taken on a camera mounted to a stationary tripod with controlled lighting to ensure consistency among the photographs. In ImageJ, each droplet (~5ml) was processed manually using the Low-Bond Axisymmetric Drop Shape Analysis for Surface Tension and Contact Angle Measurements of Sessile Drops (LBADSA). This plug-in allows the user to fit a guide droplet around the droplet to be measured and create a best-fit CA measurement.

C. Results

All data was compiled to compare the results across flow rates, surface pattern, and if siloxane injection occured. In general, the mound patterned samples started and ended the test more hydrophobic than the pyramid patterned samples. In tests ranging from 51-58 hours for both the 60 and 120 CFM air flow cases, the mound patterned samples began with average CA measurements of 32° (±13) and ended with average CA measurements of 117°

 (± 10) when siloxane injection was present. In the same tests, the pyramid patterned samples began with average CA measurements of 24° (±18) and with concluded average CA measurements of 79° (±15). For each change in variable (location in the louver cassette, laser pattern, siloxane injection, air flow rate) and each test run, a plot similar to Figure 6 was created to determine the rate of change in contact angle over time for each case. The rates of change determined by the trendlines for each of the cases previously mentioned were compiled, averaged with their like types, and compared across two flow rates. Figure 7 summarizes the change in contact angle for all test cases.

The ultimate conclusion drawn

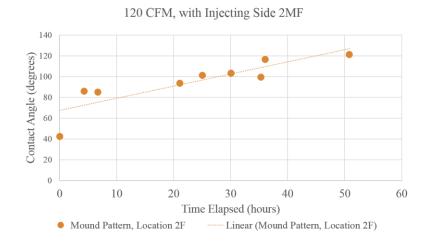


Figure 6. Siloxane injecting at 120 CFM, for single louver surface. Surface is front side of a mound pattern, second louver from the top.

from these reults is that all samples turn hydrophobic when exposed to siloxanes. This change is independent of pattern and fan speed. Silver tests have not been thoroughly completed, but preliminary results indicate they too turn hydrophobic when exposed to siloxanes. Because of the results of this study, a hydrophilic LP CHX is not feasible for spaceflight as the order of time in which this change occurs is in hours. It appears a hydrophobic LP CHX would be a preferred route, as the environment may help maintain its hydrophobicity. One of the other attractive features of a hydrophobic LP CHX is the improved heat transfer due to dropwise condensation. This may present alternatives to reduce mass and volume of a CHX. There are still a great deal of questions that need answering to determine the full feasibility of a hydrophobic LP CHX, most principally whether the substances used to make it hydrophobic will then be transferred to the condensate. Testing is currently underway to determine what, if any, contaminates are introduced to the condensate via these surfaces.

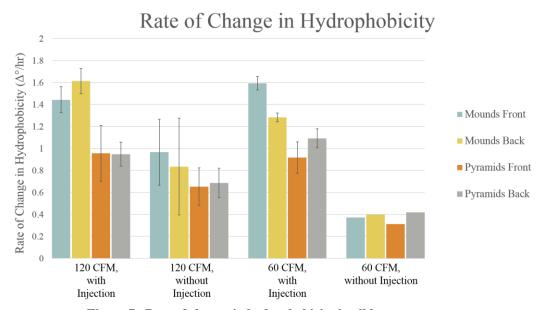


Figure 7. Rate of change in hydrophobicity in all louvers.

IV. Microbial Adhesion of Laser Processed Surfaces Study

A. Materials and Methods

Bacterial Strains and Growth Conditions

In order to capture diversity among bacterial strains, a Gram-positive human commensal, *Staphylococcus epidermidis*, a common *Bacillus* found in extensively diverse environments, *Bacillus megaterium*, and a Gramnegative frequently associated with water, *Sphingomonas paucimobilis* were used in all studies. All bacteria were isolated from air or surface samples collected on board the International Space Station (ISS). Bacterial strains were initially identified via biochemical reactions using the VITEK 2 Compact instrument (Biomerieux) and verified via

16S rDNA sequencing on the ABI 3500 Genetic Analyzer (ThermoFisher Scientific). Prior to each individual test run, a 10 µl loop of *S. epidermis*, *B. megaterium*, and *S. paucimobilis* were taken from a frozen stock, separately inoculated into 3 mL of Tryptic Soy Broth (TSB), and grown statically at 35 °C overnight. Initial bacterial concentrations were determined using standard serial dilution and plating methods followed by overnight culture at 35 °C.

Table 2. Microbial adhesion testing coupon samples.

<u>Wettability</u>	Substrate Material	Laser Processed Pattern	
Hydrophillic	Stainless Steel	Pyramid (Similar to Figure 4a,c)	
	Stalliess Steel	Mounds (Similar to Figure 4b,d)	
	Silver	Pyramid (Similar to Figure 2a)	
	Silvei	Mounds (Similar to Figure 2b)	
Hydrophobic	Stainless Steel	Pyramid	
	Stanness Steet	Mounds	
	Silver	Pyramid	
	Silvei	Mounds	
-	Silver	-	

Coupon Inoculation and Testing

A total of nine coupons were tested and are highlighted in Table 2. Each coupon was individually sealed and autoclaved prior to testing. To generate hydrophobic coupons, surfaces were treated with 250 μ l of filter-sterilized liquid siloxanes (D4, D5, TMS) following the autoclave process to create a hydrophobic surface. Based on growth profile estimates, bacterial concentrations were adjusted and a consortium of the three organisms was generated at a final concentration of approximately 1×10^5 colony forming units (cfu)/mL. Each coupon was inoculated with 100 μ l of the bacterial consortium and dried for two hours within a biological safety cabinet (BSC). Following the two hour exposure time, each coupon was aseptically placed into a sterile 50 mL conical tube containing 10 mL of phosphate buffer. Individual conical tubes were placed into a plastic tube rack, loaded into an Ultrasonic Cleaner FS30 (Fisher Scientific) filled with water, and sonicated for 10 min. The conical tube rack was weighted down to ensure that all coupons within their buffer were submerged and evenly distributed during sonication. Following

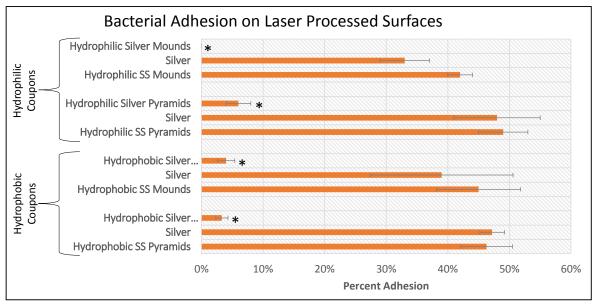


Figure 8. Bacterial Adhesion to Surfaces of Various Materials and Patterns. Bacterial adhesion was determined as the number of surviving cells relative to those recovered from corresponding unprocessed stainless steel control coupons included with every test. There was a significant reduction in adherence to laser processed silver surfaces as compared to all other coupons tested (*, P < 0.05).

sonication, the conical tubes were vortexed for 30 seconds. Prior assessments demonstrated this level of sonication and vortexing did not impact the viability of the microorganisms evaluated. The bacterial concentrations recovered from the coupons were determined by direct plating as described above. All assays were performed in technical triplicate, and adhesion percentages were determined from three independent biological replicates.

B. Results

Bacterial adhesion was determined as the number of surviving cells relative to those recovered from corresponding unprocessed stainless steel control coupons included with every test. Additionally, unprocessed silver coupons were included in every test with the variable laser processed surfaces. Across the four tests, the average bacterial adhesion level to stainless steel, despite the hydrophobicity or hydrophilicity of the surface, and unprocessed silver was $46\% \pm 3\%$ and $42\% \pm 6\%$, respectively (Fig. 8). Interestingly, little-to-no bacteria could be recovered from the functionalized silver coupons. Adhesion was $3\% \pm 1\%$ for hydrophobic silver pyramid coupons, $4\% \pm 1\%$ for hydrophobic silver mound coupons, and $6\% \pm 0\%$ for hydrophilic silver pyramid coupons. Bacteria could not be recovered from any replicate of the hydrophilic silver mound coupons. To determine if viable bacteria were strongly affixed to the surface of the hydrophilic silver mound coupons, coupons were submerged into TSB growth medium post the two hour exposure time. The media remained sterile, while bacterial growth was noted in the media of other coupons strongly suggesting that interaction with the surface resulted in bacterial killing. Throughout the duration of testing, visible growth of each individual bacterium was characterized based on colony

morphology. Not surprisingly, *S. epidermidis* and *B. megaterium* were recovered in the highest abundance, as *S. paucimobilis* is prone to killing as a result of desiccation.

Discussion

Based on the microbial testing presented, further analysis of the silver-processed surfaces is warranted over other laser processed patterns and materials. As there is not a significant difference between the two pattern types, a randomized design or non-conforming pattern on a silver coupon should be included in further assessments to determine if pattern plays a role in deterring bacterial adherence or if the suggested bacterial killing is resulting from the biocidal nature of silver nanoparticles created during laser processing.

V. Manufacturability

Clear line of sight with respect to manufacturability of a LP-CHX was also of concern during initial idea formulation. One typical processes occurring during CHX manufacturing is fin bending. Ideally, laser processing of

CHX surfaces would take place on a flat sheet, before fin bending occurs. To determine if this was feasible a flat 1" wide strip of stainless steel was processed and bent into fins. This process destroyed nanofeatures created on the surface hydrophilicity of the surface is no longer maintained. Therefore, to develop a CHX, fins must be created before laser processing and laser processing must occur on the fins. The LaSER lab was able to successfully process stainless steel fins. Figure 9 is a picture of a laser processed fin. The fin included in Figure 10 has eight fins per inch, which is about half of typical CHX fins at sixteen per inch. Future investigation is needed to determine if laser processing can occur with fins of this spacing.

Another typical manufacturing processing used in constructing a CHX is brazing. Because laser processing creates a unique and initially hydrophilic surface braze material may wick into the laser processed areas or the surfaces may prevent brazing from occurring. Laser processed finned samples were sent to United Technologies Corporation Aerospace Systems (UTAS) for brazing investigation. UTAS conducted a semi-quantitative chemical analysis of processed areas and unprocessed areas and discovered elevated amounts of oxygen in the processed areas, which indicates the presence of an oxide layer. Brazing was conducted using a nickel braze alloy. The fins were unable to braze properly due to poor wetting of the braze alloy to the base metal. It is assumed the oxide layer created during the laser process was the cause of the poor braze.

Laser welding was also investigated as a means of assembly of a CHX and a possible braze alternative. Stainless steel finned samples and parting sheets (both laser processed), 0.010" thick were sent to Edare to be laser welded. Edare, Lebanon, NH, offers manual and CNC laser welding services for a growing variety of precision applications. Their current focus is on hermetic heat exchanger assemblies for aerospace applications and miniature vacuum insulated tubing assemblies for advanced manufacturing and medical applications. The ultra-small heat affected zone and programmable interface of the laser permits extremely repeatable results on a large variety of metal structures. Overall, results of laser welding are

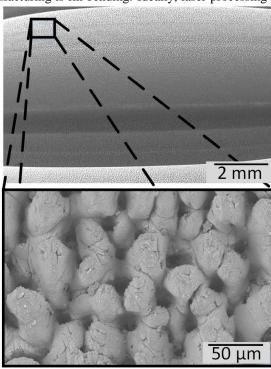


Figure 9. SEM imagies of processed fins.

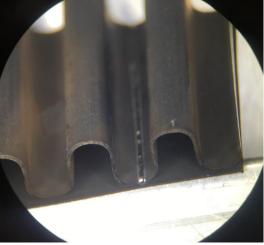


Figure 10. Laser Welding of Fins.

promising as initial laser welding trials of fins to parting sheets was successful (Figure 10). While further, more detailed laser welding studies need to be completed, including use of silver substrate materials and different surface geometries, laser welding may be an appropriate method to construct a LP-CHX test article.

In addition to identifying appropriate manufacturing methods, research is being carried out by the LaSER lab on Atomic Layer Deposition (ALD) of silver on the laser processed surfaces. Since silver is a strong candidate for construction of a LP-CHX, substantial weight may be added to a CHX. One method to solve this problem is using ALD to deposit several atomic layers of silver on a laser processed stainless steel substrate. Therefore, the CHX would primarily be made from stainless steel and would weigh very similar to current CHX concepts. The LaSER lab has had some successes in developing the ALD process, with initial work carried out on silver ALD onto silicon.

VI. Conclusion

While many questions still must be addressed in the development of a LP-CHX, initial studies have proven that the concept may be viable. With the research described in this paper, a silver, hydrophobic, laser processed CHX will be pursued for further studies. Individual studies to be completed include a second microbial adhesion study of only FLSP silver samples, condensing study in which hydrophobic FLSP surfaces are condensed upon and the condensate quality analyzed, sixteen fin per in laser processing, and further manufacturing studies.

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