EFFECTIVENESS OF THERMAL OXIDATION IN RELATION TO ANTERIOR CERVICAL
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ABSTRACT

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Ti-6Al-4V anterior cervical plates (ACP) are used in spinal fusion surgeries to fixate cervical vertebrae during graft adhesion. However, documented cases of implant failure and the potential for ACP corrosion raise concerns regarding any degradation of material resulting from extended implantation. In addition, abrasion during implantation may damage a section of the protective oxide layer, potentially exposing surrounding tissues to the harmful effects of bare titanium, aluminum, and vanadium. Thermal oxidation has been shown to improve corrosion-resistance and wear-resistance, depending on temperature and time. To quantify the attributes of the thermally grown oxide layer, Ti-6Al-4V coupons underwent thermal oxidation treatments in an atmosphere environment at 600 and 675 °C for 1, 4, 8, and 16 hours. Two sample types were produced: non-abraded and abraded.

Non-abraded samples underwent potentiodynamic polarization according to ASTM F2129, which included open circuit potential tests. Open circuit potentials (E_{OC}) increased with increasing treatment time, indicating that longer treatment time resulted in thicker oxides. All samples treated at 675°C displayed higher E_{OC} than samples treated at 600°C, indicating an increase in oxide thickness with higher temperature. During the first hour of treatment at 675°C, the rate of oxide growth was greater than the rate of oxide growth of all samples treated at 600°C. Samples treated at 600°C for 4 and 8 hours displayed pitting during potentiodynamic polarization, but all other samples withstood the applied potentials and surfaces were further passivated.

To simulate damage during surgery, a single abrasion was made across samples in the abraded group with a diamond-tip indenter under a load of 471g at 4.4 mm/s. Abraded samples were

subjected to potential-step tests to assess repassivation ability after abrasion. All samples displayed repassivation ability, except for the sample treated at 600°C for 4 hours.

Surface roughness was measured with atomic force microscopy before and after thermal oxidation treatments. Lower surface roughness was desired to discourage osseointegration, or the growth of bone cells. No isothermal surface roughness trends were observed, as high surface roughness outliers were seen in samples treated at 675°C for 8 hours and 600°C for 4 hours. Rockwell hardness and Vickers microhardness were also measured to assess bulk changes in mechanical properties and hardness of the oxidized surfaces. No statistical change was seen in Rockwell hardness. Vickers hardness increased with increasing temperature and time, with the exception of the sample treated at 600°C for 4 hours. Metallography of the thermally oxidized samples was analyzed to determine if a change in microstructure had occurred due to thermal processing. No major change in grain size or the amount of alpha and beta grains was seen in samples treated at 600°C, but samples treated for extended times at 675°C showed equiaxed enlarged alpha grains and a reduction in beta grains.

The breakdown of samples treated at 600°C exemplified possible differences in the alpha-beta oxide behavior during thermal oxidation and corrosion. Outlying surface roughness and microhardness values related to the thermal oxidation treatments and resulting oxide structure. Due to delamination of oxides grown at 675°C for 4, 8, and 16 hours, the treatment parameters would not be effective in the ACP application. Therefore, through corrosion resistance, repassivation ability, low surface roughness, increased microhardness, and no microstructural change, thermal oxidation treatments at 600°C for more than 16 hours, and 675°C for 1 hour or less would be suitable treatments for anterior cervical plates.

Key words: Ti-6Al-4V, anterior cervical plates, thermal oxidation, corrosion, repassivation

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1 INTRODUCTION

Anterior cervical plates (ACP) promote fusion of the cervical vertebrae by providing adequate spinal placement and pressure during the healing process. ACPs are often installed with a disc graft, fixating the graft and the involved vertebrae in place while the bones fuse over time. As seen in Figure 1.1, ACPs generally consist of a metal plate with at least two holes to be screwed and secured into the spinal column. Various plate designs suited for different patient needs are pictured in Figure 1.2 a-d.

The placement and angle of the screws is at the surgeon's discretion, and some

Figure 1.1. X-ray of patient's cervical vertebrae, with screws of ACP inserted directly into individual vertebrae (**Maroon**).

plates provide more freedom for screw movement during arthrodesis, or spinal fusion.

Figure 1.2. The different ACP designs are held in place with bone screws, the placement of which depends on the patient's anatomy and the surgeon's judgment. Constrained "Orion" design by Medtronic Sofamor Danek (a.), "Slim-LOC" ACP by DePuy Spine (b), Semi-constrained "ABC" design by Medtronic Sofamor Danek (c), and segmented pieces of the "Atlantis" design by Medtronic Sofamor Danek (d), all provided by Beaumont Hospitals.

1.1 Anterior cervical plate failure due to damage and corrosion

Although the plate is designed to remain within the body for the life of the patient, an average of 15-25% of plates fail (Shapiro & Snyder, 1997) and have to be removed. Forms of failure include plate fracture, screw fracture, and screw backout. Many failures are attributed to “surgical damage.” Damage may include improper ACP screw positioning during surgery, placing a load on the ACP that the system was not designed to carry, or abrasion prior to implantation, which damages the surface of the plate as it is implanted into the patient’s body. Figure 1.3 depicts an explanted ACP with surface damage, providing insight as to the amount and type of damage that may be caused during surgery and in-situ.

Figure 1.3. Scanning electron microscope image of abrasions across the surface of explanted ACP (a), found on other section of the ACP (b) (K. Miyashiro).

In addition, 5-10% of cases result in infection of surrounding tissues (Shapiro & Snyder, 1997). The metallic composition of ACPs creates a risk for *in vivo* corrosion, which may be related to the infection rate of periprosthetic tissues (Baker, 2007). The release of titanium and alloying elements of aluminum and vanadium are cause for concern, as the alloying elements have toxic effects, such as damage to the central nervous system (Aluminum). In addition, the

local and systemic discoveries of metallic elements in cadavers from implants that undergo repeated wear indicate that a degradation of the surface may permit the migration of ions to nearby tissues (Jacobs, Gilbert, & Urban, 1998). Although ACPs do not undergo repeated wear on a macro scale, the proximity of the ACP to vital tissues such as the spinal cord and brain make it a device of concern.

1.2 Base metal corrosion resistance

Ti-6Al-4V is a common alloy for ACPs, and is also used in other orthopedic devices such as hip implants, bone plates, and dental implants (Komotori, Lee, Dong, & Dearnley, 2001; Vadiraj & Kamaraj, 2007; Deanrley, Dahm, & Cimenoglu, 2004). Its high strength-to-weight ratio and biocompatibility are effective in the ACP application, where loading and repeated wear are minimal and osseointegration, or the growth of bone cells, is not desirable.

Ti-6Al-4V is selected for ACPs for its strength and formability, but is not completely immune to corrosion and wear, both of which are affected by the surface conditions of the alloy (Deanrley *et al.*, 2004). Therefore, the question remains as to whether surface modification may aid in corrosion resistance and reaction to wear, without altering the advantageous properties of the material that promoted Ti-6Al-4V selection for ACPs in the first place.

As a highly reactive metal, titanium readily oxidizes with its surrounding environment. The exposed substrate can form a thin natural oxide layer almost instantaneously, at 10^{-8} s (Gibbs, 2008), but the rate of formation of the natural oxide decreases greatly after establishment. The protective layer may be altered with various treatments to improve certain facets of biocompatibility, such as osseointegration, wear resistance, or corrosion resistance. Surface modifications depend on the application, the purpose of the implant, and the state of the Ti-6Al-4V surface upon which they form.

Titanium is often chosen for biomaterials not based on its cost, but for its strength, stiffness, and natural corrosion resistance. In addition, there has been no evidence of Ti causing inflammatory cells, granulation tissue, or necrotic debris at a metal-tissue interface. Formable to fit a variety of applications, titanium is alloyed with aluminum and vanadium to improve bulk material properties, and treated to improve surface properties. The natural oxide thickness can be enhanced by various treatments, but thermal oxidation shows superiority and validity by comparison in tests specific to conditions surrounding an ACP.

1.2.1 Surface modification comparison for improved wear and corrosion resistance

Various surface treatments serve as options to improve the corrosion and wear resistance of Ti-6Al-4V, in addition to altering the surface-cell interaction. The treatments include nitriding, aging in deionized water, passivating in nitric acid, anodizing, and thermal oxidation. Plasma nitriding involves bombarding the surface of a sample with nitrogen ions to create a TiN passive layer. Aging in deionized water requires the immersion of a sample into boiling water for a specified time to create a passive hydrated oxide layer on the surface. Immersion into nitric acid solutions at specific temperatures and times has been researched as an alternate oxidation treatment, creating a passive surface with stoichiometry similar to plasma nitriding. Thermal oxidation uses elevated temperatures and specific environments to create a crystallographic oxide layer. Each surface treatment holds potential for the ACP application, which requires a surface treatment that is corrosion resistant, provides wear protection, does not detrimentally alter the bulk properties of the material, and discourages osseointegration. As discussed in the following section, a comparison of the corrosive behavior, wear behavior, alteration of mechanical properties, and surface roughness after treatment presents thermal oxidation as an improved treatment for the ACP application.

1.2.2 Comparison of different surface treatments using corrosion tests

Thermal oxidation, aging in deionized water, and nitric acid immersion were compared by Lee, Chang, and Yang (1999) through immersion tests and measurement of constituent release. Sample groups underwent a single passivation treatment: 400°C thermal oxidation treatment in air for 45 minutes, immersion in 34% nitric acid for 1 hour, or aging in boiling deionized water for 24 hours. After immersion tests in an ethylenediaminetetraacetic acid (EDTA) Hank's solution, the thermal oxidation treatment released the least titanium, aluminum, and vanadium trace elements compared to the passivated and aged treatments. Comparison of the three treatments indicated thermal oxidation is most suited for the highly sensitive location of ACPs.

Nitrided samples were compared to thermally oxidized Ti-6Al-4V samples by Komotori *et al.* (2001). The open circuit potential (E_{OC}), or potential of an immersed sample in equilibrium, of thermally oxidized samples was more consistent and reproducible, unlike the scattered E_{OC} values obtained for TiN treatments. The reproducibility indicated higher stability of the oxide layer fabricated by thermal oxidation compared to nitriding. During cyclic polarization tests, breakdown of either surface modification occurred at potentials too high to be physiologically relevant (Rosenbloom & Corbett, 2007).

The corrosive properties of thermally oxidized Ti-6Al-4V were also superior to nitric acid immersed Ti-6Al-4V in cyclic polarization tests (Petersen, Venugopalan, Lemons, & Lucas). Samples were either passivated in 40% nitric acid for 0.5 hours or thermally oxidized at 350°C for 1 hour. The 14.8 ± 0.3 nm thickness of oxide layers produced by thermal oxidation surpassed the 5.4 ± 0.3 nm thickness produced by nitric acid immersion. Compared to untreated and passivated samples, the corrosion potential (E_{CORR}) of thermally oxidized samples was more noble, and the rate of corrosion slower, displaying greater corrosion resistance than nitric acid passivation.

Compared to other surface modification methods, thermal oxidation appears to be a viable surface treatment for corrosion resistance, but the potential wear of the ACP also requires a surface treatment that is wear-resistant and able to repassivate *in-vivo*.

1.2.3 Comparison of surface treatments using repassivation and prior abrasion tests

Wear testing of surface-treated Ti-6Al-4V resulted in delamination of the protective surface layer in some cases. According to Borgioli, Galvanetto, Iozzelli, and Pradelli (2005), the amount of oxide in wear debris of samples that were thermally oxidized was greater than samples that were not surface treated. The increase in oxide in the debris indicated a decreased adhesion as a result of thermal oxidation treatments. However, the thermally oxidized samples resulted in more oxide in wear debris, as their treatment created a thicker oxide layer. The oxide debris was minimal in untreated samples, as the thickness of the oxide was significantly less than thermally oxidized samples. The thermal oxidation samples wore *via* the delamination of the hardened oxide layer, as opposed to wearing down the oxide of the sample and creating substrate wear. Samples that underwent thermal oxidation treatments also increased the hardness of the surface layers.

Wear of nitrided and thermally oxidized samples was also investigated by Borgioli, Galvanetto, Fossati, and Pradelli (2004), determining that glow-discharge nitride samples did not show an improved wear resistance of Ti-6Al-4V compared to furnace-fabricated oxide treatments. Oxide treatments were conducted in air at 700°C and 900°C for 0.5, 2, and 4 hours, forming a rough outer oxide in samples treated at 900°C. The adherence of the layer fabricated at 700°C was greater than the adherence of the layer fabricated at 900°C, but the wear volume of the oxidized samples was between 6 and 8 times lower than untreated and nitride samples. The nitride surface layer microfragmented during wear tests, creating more abrasive wear with the resulting nitride fragments.

Komotori *et al.* (2001) studied the wear behavior of nitrided Ti-6Al-4V compared to thermally oxidized Ti-6Al-4V. After abrading with a diamond tip indenter under a 500g load at 0.25mm/s, thermally oxidized samples showed greater oxide layer adhesion and greater resistance to scratch depth than TiN layers. In addition, the thermally oxidized samples showed a gradual change in hardness between the oxide and substrate, while TiN coatings showed a great change in hardness between coating and substrate. The TiN coatings showed more scratch damage, displaying larger scratch areas and corresponding scratch depths, compared to the thermal oxidation counterpart.

1.2.4 Comparison of surface treatments based on material properties after treatment

Komotori *et al.* (2001) also compared the hardness of nitrided Ti-6Al-4V samples to thermally oxidized Ti-6Al-4V samples. The surface roughness was greater for the TiN samples due to the large defects created by the nitriding process, but the two treatments had similar hardness values. Because bone cells better cultivate on a rough surface, the smoother surface of thermal oxidation is more applicable in the case of ACPs, where osseointegration is not desirable.

In addition, MacDonald, Rapuano, Deo, Stranick, Somasundaran, and Boskey (2004), and Ku, Pioletti, Browne, and Gregson (2002), determined that the presence of aluminum on the surface of a material affects the attachment of osteoblasts. Particularly in the case of Ti-6Al-4V, where surface modification alters the surface content of aluminum, Al content can regulate the biological activity of a cell-adhesive protein, but cell growth reduces when the surface content of Al is reduced. Ku (2002) found less surface content of Al in samples that underwent thermal oxidation treatments than standard passivation treatments or no surface treatment at all.

In addition to surface roughness alterations, many researchers (Borgioli *et al.*, 2004; Gülyerüz *et al.*, 2004; Dearnley *et al.*, 2004, Frangini *et al.*, 1994) found that thermal oxidation increased the surface microhardness and microhardness gradients, according to temperature. Higher

temperatures achieved higher hardness and roughness, but more delamination and scarring in wear studies. Therefore, the parameters of the thermal oxidation treatment can create oxidation layers with drastically different properties and must be selected with respect to the application, which in this study, is anterior cervical plates.

1.2.5 Parameters of effective thermal oxidation for ACP application

For anterior cervical plates, where corrosion resistance is required, fretting is minimal, and bone growth is not desired, thermal oxidation becomes an option for surface modification, with the proper treatment parameters. Important parameters of thermal oxidation treatments include environment, time, and treatment temperature.

Treatment parameters influence bone cell growth. The surface content of Al increases cell growth, and heating between 650 and 850°C encourages the diffusion of Ti and Al from the substrate, forming a multilayer of Al_2O_3 and TiO_2 (MacDonald *et al.* 2004). For thermal oxidation of Ti-6Al-4V anterior cervical plates, the diffusion of Al from the substrate should be minimal and the treatment temperature selected accordingly. Osteoblast cell adhesion to Ti-6Al-4V that had been thermally oxidized at 500°C and 700°C was compared by Saldaña, Vilaboa, Valles, Gonzales-Cabrero, and Munuera (2005), who determined that cell attachment was greater for the higher temperature. A comparison of treatment times determined that the maximum number of cell attachments was found on samples that had been oxidized for 3 hours, and remained constant for samples with longer oxidation times. Therefore, to discourage osseointegration, the oxidation temperature should be below 700°C, and ideally, below 650°C.

The importance of temperature in the thermal oxidation process is also brought to light in a study by S.J. Li, Yang, and S. Li (2004). The group stated that thermal oxidation treatments used to change mechanical properties of Ti-6Al-4V and to create an oxide layer of TiO_2 are not effective in improving wear resistance. Three different experimental conditions were tested:

750°C for 1 hour, water quench to cool; 1050°C 0.5 hours, water quench to cool; 750°C for 1 hour, water quenched, and treated at 500°C for 24 hours, air cooled. The last of the three represented the thermally oxidized sample. The sample treated at 1050°C showed the greatest change in mechanical properties, the 750°C sample showed the least, and the thermally oxidized sample in between.

The susceptibility of Ti-6Al-4V's mechanical properties to treatment temperature was exercised, both with the treatment at 1050°C for 0.5 hours and 500°C for 24 hours. Therefore, to state that thermal oxidation treatments are not effective in improving wear resistance was not properly investigated by Li *et al.* (2004), as only one treatment compared to the “substrate” 750°C and 1050°C treatments was analyzed. Treatment time and temperature greatly affect the adhesion of the oxide layer to the substrate, an important treatment parameter not taken in to account by Li *et al.*(2004).

Dearnley *et al.* (2004) further emphasized the importance of thermal oxidation temperature. The group researched the corrosion-wear resistance of thermally oxidized Ti-6Al-4V compared to commercially pure Ti, stating that a thermal oxidation temperature of “at least 600°C is required to produce an external TiO₂ layer that has sufficient functionality for tribological situations.” In addition, the group determined that higher temperatures create oxide layers that poorly adhere to their Ti substrate. Commercially pure and Ti-6Al-4V samples were thermally oxidized in air at 625°C for 36 hours. The microhardness (100g load) of the Ti-6Al-4V sample increased in both the commercially pure and Ti-6Al-4V samples. The thickness of the TiO₂ layer on the Ti-6Al-4V samples was three times the thickness of the TiO₂ layer on the CP samples, but correspondingly, the residual stress in the ceramic coating was greater for the Ti-6Al-4V samples than the CP samples. The wear resistance of both materials increased with thermal oxidation, and the coating failed *via* fracture along the TiO₂-oxygen diffusion zone interface. Although the CP samples showed slightly more wear resistance than the Ti-6Al-4V samples, both maintained oxygen

diffusion zones that wore at much lower rates than their untreated counterparts. Therefore, according to Dearnley *et al.* (2004), thermal oxidation was effective in wear resistance and increased microhardness at temperatures above 600°C.

According to kinetics, temperature plays a vital role in oxide structure, but the structure and corresponding behavior of the oxide is also dependent upon treatment time. The effect of time and temperature on the Ti-6Al-4V oxide was investigated by Güleriyüz *et al.* (2004). Treatments at 600 and 650°C in atmosphere for between 12 and 60 hours were analyzed using x-ray diffraction (XRD), surface roughness measurements, hardness measurements, microstructural examination, and immersion in 5M HCl for 60 hours.

XRD analysis showed that the surface oxide of the sample treated at 650°C mostly consisted of rutile TiO₂, which differs in density from the small quantity of anatase and alpha Ti also present in the oxide formed at 600°C. Surface roughness measurements depicted an increasing trend in roughness for both oxidation temperatures, with a greater rate in samples treated at 600°C. Surface hardness also increased with an increase in oxidation temperature and time.

The weight loss for samples oxidized at 600°C increased with immersion time. Samples treated at 650° did not show any weight loss until 36 hours of immersion, and the oxides of samples treated for longer times delaminated after immersion longer than 36 hours. Therefore, the thick oxide grown at higher temperatures was initially protective, and later rapidly detrimental to the substrate. The thermal oxide treatment at 600°C for 60 hours grew the most stable oxide layer, with the least weight loss in the aggressive environment.

Failure of oxide layers formed at 650°C was attributed to microcrack and micropore defects in the structure, resulting from thermal stresses placed on the oxide during its rapid formation. Longer treatment times at 600°C resulted in more rutile with fewer defects at the surface, creating a more immune layer over the reactive substrate.

In summary, the various research groups provided data supporting temperature's primary influence on the performance of a thermal oxidation treatment. According to MacDonald *et al.*

(2004), Dearnley *et al.* (2004), and Güleriyüz *et al.* (2004), treatments between 600 and 700°C would provide comparatively low surface roughness, increased surface microhardness and wear resistance, and a more stable, corrosion-resistant oxide layer. Much of the thermal oxidation reaction is dependent upon the selected temperatures, which affect the oxide thickness hardness, surface roughness and aluminum content, adhesion, and depth of the oxygen diffusion zone. Each attribute is related to the overall performance of the oxide layer in a particular application, and in this study, the oxide layer must withstand conditions specific to ACPs. Therefore, the parameters of the study must test the susceptibility of fabricated oxide layers to corrosion and abrasion.

1.3 Project goals

Based on the location and surrounding environment of anterior cervical plates, three goals emerge in investigating surface modification *via* thermal oxidation of the Ti-6Al-4V alloy:

1. Determine if thermal oxidation is an effective treatment for corrosion resistance in this biomedical application
2. Determine whether a thermally oxidized sample can repassivate in a physiological media despite prior abrasion
3. Determine whether the process of thermal oxidation alters material characteristics, reducing the alloy's performance in an ACP application.

1.4 Overview of method

Temperatures of 600°C and 675°C were selected for thermal oxidation treatments in order to create reasonably thick, wear-resistant, corrosion resistant surfaces with appropriate adhesion, roughness, and ductility. Times of 1, 4, 8, and 16 hours were chosen for each treatment.

The first goal was tested using open circuit (OC) and cyclic polarization (CP) tests according to ASTM F2129. Repassivation ability of Ti-6Al-4V, the second goal, was analyzed using

potential-step tests. Material modification as a result of thermal oxidation was characterized using scanning electron microscopy (SEM) and atomic force microscopy (AFM) and quantified using hardness and microhardness measurements.

2 MATERIALS AND PROCEDURE

2.1 Test plan overview

Corrosion samples were separated into two groups: non-abraded and abraded. Non-abraded samples were designated with “N” and abraded samples were designated with “A.” Samples were also designated with the treatment temperature and time to reference the thermal oxidation treatment during testing. The non-abraded and abraded groups underwent the same thermal oxidation treatments and surface analysis, but differed slightly in surface preparation, corrosion tests, and hardness tests. The non-abraded samples underwent open circuit (OC) and cyclic polarization (CP) tests to evaluate corrosion resistance, and Rockwell hardness measurements to determine any change in bulk properties. Abraded samples underwent abrasion and potential step tests to assess repassivation ability, and microhardness measurements to investigate the oxide hardness. The non-abraded group was analyzed first to determine if thermal oxidation fabricated oxide layers with effective corrosion resistance. Data from the abraded samples determined whether the thermally oxidized samples were able to repassivate in the physiological media despite prior abrasion and related surface damage. Atomic force microscopy (AFM) data and scanning electron microscope (SEM) were used to examine the alteration of the surface due to oxidation and testing.

2.2 Materials

Ti-6Al-4V rod stock was provided by Beaumont Hospitals, in Michigan. The chemical composition of the Ti-6Al-4V stock is included in Table I.

Table I. Composition (at. %) of Ti-6Al-4V rod stock, tested by Allvac.

	Ti	Al	V	Fe	O	C	N	Cu
Top	Bal	6.50	4.04	.17	.19	.040	.03	.02
Bottom	Bal	6.30	4.11	.19	.16	.048	.03	<.01

Aluminum and vanadium act as α -phase and β -phase stabilizers, respectively. The HCP α -phase is rich in Al, but depleted in V, and precipitates out of the titanium matrix in the form of plates or needles, oriented within the BCC β grains. Generally, the alloy is heated above 1000 °C to stabilize the β -phase and oil-quenched into the two-phase Widmanstatten structure. It is later annealed and cold worked at temperatures near, but not above the β transus for fine-grained α and β particles isolated at the grain boundaries.

In accordance with ASTM F1472, “Standard Specification for Wrought Titanium - 6Aluminum -4Vanadium Alloy for Surgical Implant Applications,” the as-rolled rod stock received from Beaumont was heat treated at 704°C for 2 hours and air cooled. The rod stock was then heated at 954°C for 1 hour, cooled in water, aged at 530°C for 4 hours, and air cooled. The β transus was reported to be 1004°C at the top of the rod, and 1007°C at the bottom. As seen in Figure 2.1, the microstructure of Ti-6Al-4V is a compilation of fine α grains (light) and β particles (dark).

Figure 2.1 Microstructure of Ti-6Al-4V received from Beaumont Hospital, with light alpha grains bordered by dark beta regions.

2.3 Sample preparation

The rods were sectioned into discs with a water-cooled abrasive saw and a diamond saw. All samples had a consistent diameter of 1.375 inches. Sample surfaces were ground from 35 grit to

600 grit with silicon carbide sandpaper. Non-abraded samples were polished with 6 μ m diamond paste, and abraded samples were further polished with 3 μ m diamond paste.

Because each sample was cut, sanded, and polished by hand, samples were individually analyzed with an easyScan AFM before thermal oxidation. The samples were treated under atmospheric conditions in a Lindberg tube furnace at either 600 or 675°C for 1, 4, 8, and 16 hours. One sample was treated for each temperature and time combination, and surface analysis was conducted after thermal oxidation treatments to assess individual changes.

Abraded samples underwent an abrasion process using a milling machine and the abrader pictured and detailed in Figure 2.2. The apparatus consisted of a hollow tube, a rod, an indenter holder, and a diamond-tip indenter. One end of the rod was inserted into the hollow tube and the other was inserted into an indenter holder that held the diamond-tip indenter as it abraded the surface. The hollow tube was held by the mill chuck. Before fitting into the hollow tube, the rod was heavily lubricated with a highly viscous synthetic lubricant to allow for free vertical motion and minimal horizontal motion of the fixture.

Figure 2.2. Abrasion apparatus for repassivation tests paused over recycled test sample (left). A SolidWorks representation shows the separate detailed pieces of the apparatus (right).

A 395.2g weight was placed onto the fixture before the rod was inserted into the hollow bit. Added to the weight of the fixture and the diamond tip indenter, the final weight placed onto a sample was 471.5g, slightly less than the 500g weight used by other researchers (Komotori *et al.* (2001), Komotori *et al.* (2007), Kahn *et al.* (1996). The load was applied to the surface at a rate of 4.4mm/min, following literature and ASTM standard G171, “Scratch hardness of materials using a diamond stylus.” A single abrasion was made across the diameter of each sample.

According to the literature, the depth of the scratch is dependent upon the oxide characteristics, such as hardness, roughness, and the depth of oxygen penetration. Therefore, the test required an abrasion system that repeatedly created the same abrasion, although the width and depth of scratches would vary from treatment to treatment. Following abrasion, samples were examined with an optical microscope, measuring sections of the scratch and comparing the scratch widths to ensure a consistent scratch across the sample. A typical abrasion section is pictured in Figure 2.3.

Figure 2.3. Section of abrasion across test sample. Width was measured for process consistency.

2.4 Corrosion test methods

In order to investigate a material's behavior *in vitro*, electrochemical tests apply potentials that assess the material's capabilities to withstand degradation. An applied potential simulates the potential difference between a sample and the human body, accelerating an *in vivo* corrosion reaction, which is monitored and controlled in the corrosion cell.

A corrosion cell, with anode, cathode, and electrolyte, creates the simulated physiological environment used in electrochemical tests of biomedical alloys. Seen in Figure 2.4, the corrosion cell is maintained at 37°C. The phosphate buffered saline (PBS) solution is at a pH similar to the human body, 7.35 in the case of blood. The corrosion cell was composed of a three-electrode

system, including a KCl saturated calomel electrode (SCE), platinum counter electrodes, and working electrode, or sample under investigation. A potentiostat applied the potential that drives the experiment and measured the resulting current of the sample.

For both non-abraded and abraded samples, a small hole was drilled into each sample and a stainless steel wire was looped through to create an electrical connection for testing. Silicone epoxy masked the samples after a specific surface area had been stamped and marked off. The same stamp created a uniform test surface area on the center of each sample. The epoxy covered the rest of the sample and the stainless steel wire, exposing only the stamped area for testing.

Heat shrink tube covered the wire that was not in direct contact with the hole drilled into the sample to ensure that only the specific marked off area of the sample would be tested. The diameter of the exposed area was measured twice and averaged to compute the surface area for the PowerCORR software.

Figure 2.4. Corrosion cell with platinum counter electrodes, SCE, working electrode, and gas purge tube.

2.4.1 Open circuit tests

Potentiodynamic polarization, or cyclic polarization (CP), began with a nitrogen purge, ridding the solution of excess oxygen that could affect the redox reaction. The deaerated PBS served as the electrolyte for the rest of the test, as the nitrogen purge continued throughout. After the nitrogen purge, non-abraded samples were placed into the corrosion cell with no applied potential and open circuit (OC) measurements were taken. The resulting potential difference

between the sample and solution was measured over 1 hour as the sample reached equilibrium, as seen in Figure 2.5. The final potential of the OC test is regarded as the open circuit potential, E_{OC} , and provided the starting point for the cyclic polarization tests.

Figure 2.5. Open circuit test ends with open circuit potential (E_{OC}), the potential difference between the sample and the environment as the sample reaches equilibrium in the PBS. The E_{OC} is the starting potential for cyclic polarization test.

2.4.2 Cyclic polarization tests to assess corrosion resistance

Potentiodynamic polarization cycles a sample through a set of applied potentials at a constant rate, with a maximum applied potential that is pertinent to the application conditions. The potentiodynamic polarization tests in this study followed ASTM standard F2129, “Conducting cyclic potentiodynamic polarization measurements to determine the corrosion susceptibility of small implant devices.” Following the ASTM standard, a phosphate buffered solution (PBS) of pH 7.4 was deaerated using 99.99% pure nitrogen for at least 0.5 hours before each test, and continued to purge throughout the test. The corrosion cells were in a water bath, keeping the temperature of the solution at $37 \pm 1^\circ\text{C}$ throughout the entire test. The temperature and pH were measured before and after each test.

As described previously, the E_{OC} serves as the starting potential for the CP test. Previously, CP tests were initiated at potentials 100mV below the E_{OC} , negating the information determined by the OC test. However, current convention according to ASTM F2129 begins the test at E_{OC} , only applying potentials above the rest potential at 1mV/s. During the forward scan, applied potential was increased to a maximum potential, previously established in the experimental set up. In initial tests, applied potentials reached 800mV, but to fully test the material above parameters that apply to the human body, later CP tests applied up to 1.2mV before cycling back down to the starting potential. *In vivo* rest potentials are usually below +400mV, therefore an 800mV potential limit included a factor of safety and was set by ASTM (Rosenbloom & Corbett, 2007).

The reverse scan, or decrease of applied potential at the same rate as the forward scan, began directly after the maximum potential was reached.

As seen in the potentiodynamic polarization graph in Figure 2.6, the corrosion potential, or E_{CORR} , can also be measured using a CP test. As the equilibrium point between anodic and cathodic reactions, E_{CORR} is helpful for determining the corrosion rate of a sample. However, this study focuses on the oxide layer’s resistance to accelerated corrosion, and assesses whether or not

the protective oxide will be a valid candidate in the repassivation study. Therefore, open circuit, breakdown, and repassivation potentials are more pertinent to the application of ACPs in this study.

Breakdown potential (E_b), or the potential at which a pit is initiated, is defined as a sudden increase in current, greater than a decade, as seen in Figure 2.7 and Figure 2.8. A slow increase is not considered breakdown, by ASTM standards. A pit is initiated when the applied potential is large enough to break down the protective oxide layer and allow current to flow.

The decrease in applied current allows the sample to begin repairing its passive layer.

While the hysteresis curve remains open, pits will not be able to initiate, but will continue to propagate at sites that have already initiated, as the passive layer has not been repaired and bare metal is still exposed to free electrons into the solution.

Repassivation graphically occurs as the sample cycles down through lower applied potential and the current line of the decreasing potential crosses over the current measurement of the increasing potential, as seen in Figure 2.7. Repassivation potentials are designated as E_p .

As the potential continues to decrease into a region where the metal was originally thermodynamically stable, the material is sometimes able to repair the damage, recreating a

Figure 2.6. Example of potentiodynamic polarization curve, with green arrows highlighting forward scan, and red arrows depicting surface passivation during reverse scan.

Figure 2.7. ASTM example of CP curve displaying breakdown during forward scan and repassivation during reverse scan.

passive layer where the potential has been removed. However, as seen in Figure 2.8, not all samples complete their hysteresis curve, indicating that the sample cannot repair the damage done by the test. Pits continue to grow, as the sample is not able to re-oxidize and form a passive protective layer.

In this study, potentiodynamic polarization tests were conducted by the author at Confirmd, a biomedical device testing facility in San Carlos, CA. All equipment for potentiodynamic tests, including the corrosion cell,

electrodes (SCE, platinum mesh), potentiostat (Princeton Applied Research 273A), and software (PowerCORR), was property of Confirmd. Following the standard, the CP tests were started at the rest potential (E_R , E_{OC}) determined by the OC tests, and were conducted at a rate of 1mV/min. Any breakdown was noted and included in the final report, along with other findings.

Figure 2.8. ASTM example of CP curve depicting breakdown, with no hysteresis curve or repassivation.

2.4.3 Potential step tests to assess repassivation ability

A potential step test is similar to the CP test, but the applied potential is controlled by the investigator, not the computer. Instead of changing the potential at a set rate, as in potentiodynamic testing, potential steps change the potential by a set amount at the discretion of the investigator. The potential is held until that section of the test is concluded and the potential is changed, and the resulting current is measured throughout.

The potential hold test, unlike a CP test, monitors the resulting current at a single applied potential. The potential hold can provide insight as to whether or not the material exhibits breakdown and repassivation when a specific potential is applied, and is useful in determining the behavior of an abraded or damaged sample.

At the surface of the sample, the oxide provides a protective barrier between the substrate and the liquid. An increased potential difference between the two relies upon the passive oxide layer to prevent electrons and ions from leaving the metal. However, a large increase in potential may cause a breakdown in the protective oxide layer, leaking electrons out into the surrounding solution, and increasing the current monitored by the computer. If a material is able to repassivate by reconstructing the passive layer through a series of chemical reactions, the current of electrons flowing out into the solution will decrease.

During potential step tests, the corrosion cell was held in a water bath at 37 ± 1 °C, and the pH and temperature of the PBS test solution were measured before and after each test. However, no open circuit potential was measured after the cell was deaerated for at least 30 minutes.

When the sample was submerged into the solution, no potential was applied and the resulting current was measured. As the current approached zero and a steady state was reached, the first 200mV step in potential was applied. Once a steady state was again achieved, another 200mV was applied, adding to a total 400mV. The process was repeated until at least 800mV had been reached.

2.4.4 Surface characterization and microscopy

Each sample was measured for surface roughness using a Nanosurf easyScan atomic force microscope in AC mode, with a vibrational amplitude of 700 and set point of 44-48%. Set point and scan rate varied depending on the roughness of the sample surface, and all scans were over a $25\mu\text{m} \times 25\mu\text{m}$ area.

Average roughness measurements (R_a) were taken three times for each sample and averaged. Measurements were taken before and after thermal oxidation, and after cyclic polarization tests in the case of non-abraded samples. AFM images obtained before and after thermal oxidation are presented in Figure 2.8, and the resulting change in surface roughness can be seen in Figure 2.9.

Average roughness values changed 20-40 R_a for samples treated at 600°C and 46-260 R_a for samples treated at 675°C.

Figure 2.9. AFM scan of sample surface before thermal oxidation treatment (a) and after treatment at 600°C for 16 hours (b).

Figure 2.10. Surface roughness after thermal oxidation of abraded samples at 600°C and 675°C. Measurements taken before abrasion and potential step tests.

Scanning electron microscopy with an FEI Quanta 200, 15-20keV and spot size of 4 or 5, provided qualitative analysis of oxide growth after each treatment, and observation of delamination as a result of abrasion.

To assess the effect of thermal oxidation on bulk microstructure, the hardness (HRc, 150kg load) of non-abraded samples was measured using a Rockwell C indenter. Hardness values were compared before and after heat treatments to determine whether a change in microstructure had taken place as a result of aging. In addition, the microstructure of samples was analyzed for any change in grain size or reduction in volume fraction of grain species.

Microhardness of abraded samples was measured with a Buehler MICROMET® tester (HV, 100g load) after thermal oxidation and before abrasion to analyze the hardness of the coating in relation to the oxidation treatment.

After corrosion tests, cross-sections of heat-treated samples were mounted in Bakelite, ground, polished, and etched with Kroll's solution (94% H₂O, 4% HNO₃, 2% HF) . The microstructures of the heat treated samples were compared through optical microscopy, contrasting the microstructure with temperature and time of treatment.

3 RESULTS

3.1 Research objectives

The procedures in the previous section were used to satisfy the following objectives regarding the corrosion and repassivation study of thermally oxidized Ti-6Al-4V.

1. Determine if thermal oxidation is an effective treatment for corrosion resistance in this biomedical application
2. Determine whether a thermally oxidized sample can repassivate in a physiological media despite prior abrasion
3. Determine whether the process of thermal oxidation alters material characteristics, reducing the alloy's performance in an ACP application.

3.2 Evaluation of corrosion resistance of thermal oxidation

3.2.1 Open circuit results

Oxidation treatments at 600 and 675°C resulted in increased open circuit potentials compared to the untreated sample as seen in Figure 3.1. Samples treated at 675°C had more noble E_{OC} values than samples treated at 600°C. Samples treated at 600°C show a steady increase in E_{OC}

Figure 3.1. Open circuit values for untreated sample and samples treated at 600°C and 675°C.

values with time. Samples treated at 675°C experienced a dramatic increase in E_{OC} after 1 hour of thermal oxidation, but a slight increase in samples treated for longer times.

3.2.2 Cyclic polarization results

The majority of cyclic polarization tests did not result in breakdown, as shown in Table II. These samples all showed CP curves similar to that in Figure 3.2. In each case, no evidence of breakdown was observed, and the reverse scan displayed lower current values than the forward scan.

Table II. Breakdown values for CP tests of thermally oxidized Ti-6Al-4V

Temperature (° C)	Time (hr)	E_b (mV)
25	0	none
600	1	none
	4	364
	8	none
	16	none
675	1	none
	4	none
	8	1170
	16	none

Figure 3.2. Cyclic polarization scan of sample treated at 600°C for 16 hours, with green arrows indicating forward scan and red arrows tracking reverse scan, displaying no breakdown and reduced reverse scan current.

However, N-600-4 and N-675-8 exhibited breakdown during cyclic polarization tests. N-600-4 broke down at 364mV, causing an increase in the current flow (Figure 3.3).

According to the ASTM standard used in the cyclic polarization tests, the protection potential, E_p , is indicated when the reverse scan intersects the forward scan at a value lower than the breakdown potential, E_b . N-600-4 was able to repassivate, but far below the potential at which it broke

Figure 3.3 Cyclic polarization test of sample treated at 600°C for 4 hours, with breakdown at 364mV and repassivation at -180mV.

down. Images of possible pits on the surface of N-600-4 after the polarization tests are presented in Figure 3.4.

Figure 3.4. Pits from cyclic polarization scan, located on the surface of Ti-6Al-4V sample thermally oxidized at 600°C for 4 hours.

N-675-8 broke down at 1.17V, a potential above physiological relevance to the human body. As seen in Figure 3.5, the reverse scan did not cross over the forward scan, indicating that the pit propagated for the duration of the reverse scan. However, no pits were detected on the surface of N-675-8 with the SEM.

Figure 3.5. Cyclic polarization of Ti-6Al-4V sample thermally oxidized at 675°C for 8 hours, with breakdown at 1.17V and no hysteresis.

The CP scan of N-600-8 was also unlike Figure 3.6, in its shape and display of current increases. Although there was an increase in current, beginning at 400mV, the increase was not large enough to declare breakdown by ASTM standards. However, the gradual current increase in the forward scan was overlapped during the reverse scan, and the reverse scan current remained at a higher value throughout the entire reverse scan. No pits were detected on the surface of N-600-8 with the SEM.

Figure 3.6. Cyclic polarization scan of Ti-6Al-4V sample thermally oxidized at 600°C for 8 hours with gradual increase in current and no hysteresis.

3.3 Evaluation of repassivation ability

The samples were abraded once under a 471.5 g weight at 4.4mm/s after TO treatments, masked with silicone epoxy to expose a uniform test area, and tested the next day after the epoxy cured. The prior abrasion simulated damage during surgery, in which a sample may be abraded and later immersed into human body fluids.

The abraded samples underwent a step polarization sequence, in which potentials of 0, 200, 400, 600, 800 mV, and 1V were applied. After the application of a potential step, a decreasing current represented repassivation of the sample.

The potential step graph for A-0 can be seen in Figure 3.7. The sample displays the ability to repassivate until 400mV are applied, and the decreasing current trend is interrupted by sudden small increases in current. However, the sample continues to repassivate, and the current continues to decrease. When 600mV is applied, the sample has more sudden increases in current along its decreasing current path, but the recovery places the current measurements in line with the shape of the curve. The application of 800mV causes many sudden leaks of current and repassivation.

Figure 3.7. Potential step test of untreated sample, displaying repassivation ability at every potential step.

As seen in Figure 3.8, all current trends decreased after each potential application to the sample treated at 600°C for 1 hour. Unlike A-O, there were very few potential leaks after polarizing at each potential, although a slight increase in current occurred as no potential was applied and the sample was reaching equilibrium. The rate of current decrease was higher for applied potentials 600mV, 800mV, and 1V, but the sample was able to repassivate after each polarization step.

Figure 3.8. Potential step graph of sample treated at 600°C for 1 hour, repassivating at every applied potential.

The sample treated at 600°C for 4 hours showed inconsistent attempts to repassivate after 200mV was applied, as seen in Figure 3.9. After the current at the 200mV application began to decrease and A-600-4 was polarized to 400mV and 600mV, the recovery was not smooth or consistent, with great current fluctuations.

Figure 3.9. Potential step graph of sample thermally oxidized at 600°C for 4 hours, with difficulty repassivating.

Breaks in the oxide layer found on the surface of sample A-600-4 after repassivation tests are shown in Figure 3.10. Figure 3.10a shows two possible pits, with the larger right pit about a $20\mu\text{m}$ diameter. All pits and breaks in oxide layer were not in the abrasion, but $200\mu\text{m}$ away, as seen in Figure 3.10b.

Figure 3.10. Pits on the surface of abraded sample treated for 4 hours at 600°C (a), view of pits, formed away from scar of abrasion in upper left corner (b).

The response of the sample treated at 600°C for 8 hours (Figure 3.11) was similar to the sample treated for 4 hours, but the current fluctuations were seen mostly in the 400mV application. After a constant applied potential of 400mV, the sample was able to repassivate, and the higher potentials were applied. Each higher potential displayed a slight decreasing current, indicating that the sample was able to repassivate, although trends were not as smooth as the current resulting from the 200mV polarization. Unlike samples treated for shorter times, the current began at a positive value and decreased, where the current began at negative values for A-0, A-600-4, and A-600-8.

Figure 3.11. Potential step test of sample treated at 600°C for 8 hours, with repassivation difficulty at 400mV application.

Repassivation was also seen in the sample treated at 600°C for 16 hours (Figure 3.12) which did not breakdown during the CP scans. The current with no applied potential was initially positive and decreasing, like A-600-8. A small current increase occurred during the 400mV polarization, but the sample repassivated and the current decreased. The current also increased

for 30 seconds during the 600mV polarization, but decreased and indicated repassivation. Both the 800mV and 1V showed decreasing current.

Figure 3.12. Potential step of sample treated at 600°C for 16 hours showed repassivation ability.

The samples treated at 675°C had similar results as the samples treated at 600°C, with most polarization resulting in a decreasing current.

Seen in Figure 3.13, the current scan of the sample treated at 675°C for 1 hour is initially positive when no potential is applied, and approaches zero before being polarized to 200mV. Decreasing current trends from 200, 400, 600, 800mV, and 1V are all smooth.

Figure 3.13. Potential step graph of sample treated at 675°C for 1 hour, with excellent repassivation ability.

The sample treated at 675°C for 4 hours also began at a positive current (Figure 3.14), which decreased as no potential was applied. The applied current of 200mV led to a decrease in current, with a small increase and decrease during the approach towards 0A. Another slight increase and decrease was seen when A-675-4 was polarized to 400mV, and again when 1V was applied. Similar to CP tests, 1.2V was applied to investigate the capability of the material. Despite current fluctuations throughout the recovery process, the overall current trend decreased and approached zero, indicating repassivation at 1.2V.

Figure 3.14. Potential step test of sample treated at 675°C for 4 hours, with difficulty in repassivation at 200mV and 1.2V.

The test of the sample treated at 675°C for 8 hours began with a positive current (Figure 3.15), but did not approach zero. Instead, equilibrium was reached at 1 μA before the sample was polarized to 200mV. The current fluctuated when 200mV was applied, but eventually the current decreased. Decreasing trends were much smoother for 400, 600, 800mV, and 1V. The sample was polarized to 1.2V and was able to repassivate.

Figure 3.15. Potential step graph of sample treated at 675°C for 8 hours, with only initial difficulty in repassivation.

The potential-step test of the sample treated at 675°C for 16 hours also began at a positive current and decreased when no potential was applied, as seen in Figure 3.16. Throughout each polarization step, the current decreased smoothly, with few current fluctuations. Even polarizing to 1.2V resulted in only a slight current fluctuation, but the sample repassivated and the current continued to decrease.

Figure 3.16. Potential step graph of sample treated at 675°C for 16 hours shows excellent repassivation ability.

3.4 Effects of thermal oxidation on material properties

3.4.1 Surface roughness

AFM measurements of the average surface roughness (Ra) showed an increase in surface roughness for each heat treatment, but no trend according to treatment time, as shown in Figure 3.17. Samples treated at 675°C had higher surface roughness values than samples treated at 600°C. As seen in Figure 3.17, the greatest increase in average surface roughness for non-abraded samples treated at 675°C was the 4 hour treatment, followed by the 8 hour, and 16 hour treatment. Unfortunately, post-heat-treatment roughness measurements for the 600°C 4 hour and 8 hour treatment were lost.

Figure 3.17. Surface roughness of non-abraded samples after thermal oxidation treatment.

Roughness measurements for abraded samples were taken after the thermal oxidation treatments. Before thermal oxidation, the samples were polished with a 3 μ m diamond paste. As seen in Figure 3.18, the highest surface roughness occurred in samples treated at 600°C for 4

hours and 675°C for 8 hours. There was no trend in surface roughness for samples treated at 600°C. The surface roughness of samples treated at 675 °C reached a maximum after 8 hours of treatment, decreasing after 16 hours.

Figure 3.18. Surface roughness of abraded samples after thermal oxidation treatment and before abrasion.

Images of non-abraded samples taken after thermal oxidation treatments showed an increase in surface roughness of 600°C (Figure 3.19) and 675°C (Figure 3.20) treatments, the magnitude of which depended on the time and temperature.

The change in topography of the oxide layer as a result of the thermal oxidation treatments is seen in the SEM images of the non-abraded samples treated at 600°C, presented in Figure 3.19. The non-uniform growth is seen initially emerging in the shorter treatments, and is greatly defined by the white regions of the N-600-16 SEM image.

Figure 3.19. SEM images of samples treated at 600°C for 1 (a), 4 (b), 8 (c), and 16 (d) hours.

Non-uniform oxide growth is also seen in samples treated at 675°C, as the light areas increase in size after longer treatments (Figure 3.20). Images a and c display the dominant growth of one species, as the size of the white components increases after 8 hours of thermal treatments. The decrease in magnification of N-675-16 to 657x causes the peaks to falsely appear smaller compared to the 1024x image of N-675-8.

Figure 3.20. SEM images of samples treated at 675 °C for 1 (a), 4 (b), 8 (c), and 16 (d) hours.

Additional surface roughness measurements of non-abraded samples were taken after potentiodynamic polarization tests to characterize any change in surface properties as a result of the electrochemical treatment. As seen in Figure 3.21, surface roughness changed after corrosion tests, indicating that the surfaces were modified by the applied potentials.

Figure 3.21. Surface roughness of non-abraded samples after potentiodynamic polarization tests

3.4.2 Microstructural evolution

The microstructure after varying heat treatments was examined to analyze any reduction or growth of grains and particles. As seen in Figure 3.22, the microstructures for the samples treated at 600°C show a slight reduction in the presence of β phase, which appears as the darker artifacts. In addition, compared to the untreated sample (Figure 3.22a), N-600-16 shows an increase in the size of the α grains (Figure 3.22e).

Figure 3.22. Microstructure of untreated sample (a), and samples thermally oxidized at 600°C for 1 (b), 4(c), 8(d), and 16(e) hours.

Figure 3.23 presents the microstructural evolution of the samples treated at 675°C, where the reduction in β grains and growth of α grains is more evident, as compared to the samples treated at 600°C.

Figure 3.23. Microstructure of Ti-6Al-4V thermally oxidized at 675 C for 1 (a), 4(b), 8(c), and 16(d) hours.

More grains became equiaxed in the samples treated at 675°C, although the microstructures of both temperatures displayed decreasing contents of beta particles with longer treatment times.

3.4.3 Hardness

Analysis of variance (ANOVA) of HRc measurements taken after heat treatments showed no statistical difference in hardness values as a result of thermal oxidation temperatures. Figure 3.24 presents the range of HRc values. However, one-way ANOVA also indicated that there was a statistical difference in samples treated at 675°C for different times, but no statistical difference between samples treated at 600°C for different times. There was no statistical difference between untreated samples and samples treated at 600°C, and the untreated sample compared to samples treated at 675°C.

Figure 3.24. Range of HRc values of samples after thermal oxidation treatments

3.4.4 Microhardness

The microhardness of the separate heat treatments is displayed in Figure 3.25. The oxide layer of the samples treated at 675°C had higher microhardness values, particularly at extended

oxidation times. Values from the samples treated at 600°C remained relatively low, showing little increase at larger thermal oxidation times.

Figure 3.25. Microhardness data for abraded samples after thermal oxidation.

3.4.5 Adhesion characteristics

During HRc measurements after heat treatment, delamination of the oxide layer was observed in the sample treated at 675°C for 16 hours, but not at shorter times or in samples treated at 600°C. As seen in Figure 3.26, the bare substrate was exposed around the Rockwell indentation, causing the substrate to crack along the indentation circumference.

Abrasion testing also resulted in delamination of oxides fabricated at higher temperatures and times. Samples exhibiting

Figure 3.26. Delamination of oxide around Rockwell hardness indentation (150kg load) of Ti-6Al-4V sample treated at 675°C for 16 hours.

delamination included A-675-16, A-675-8, and A-675-4, as seen in Figure 3.27. The magnitude of delamination decreased with decreasing time and temperature, as most of the length of the abrasion in A-675-16 was lined by delaminated scale, and some regions of A-675-8 and A-675-4 showed no delamination. Abrasion width also varied for each sample, with the width of A-675-16 at 100 μ m, A-675-8 at 75 μ m, and A-675-4 at 50 μ m. The oxide of samples treated at 600°C did not delaminate.

Figure 3.27. SEM image of abrasion and delamination of samples treated at 675°C for 16(a), 8(b) and 4(c) hours.

4 DISCUSSION

4.1 Overview of objectives

A series of tests and examinations were conducted to satisfy objectives concerning the effects of thermal oxidation on Ti-6Al-4V with pertinence to conditions undergone by an anterior cervical plate:

1. Determine if thermal oxidation is an effective treatment for corrosion resistance in this biomedical application
2. Determine whether a thermally oxidized sample can repassivate in a physiological media despite prior abrasion
3. Determine whether the process of thermal oxidation alters material characteristics, reducing the alloy's performance in an ACP application.

4.2 Evaluation of corrosion resistance of thermal oxidation

Open circuit and cyclic polarization tests were conducted to evaluate thermal oxidation of Ti-6Al-4V discs at 600°C and 675°C as a method to increase corrosion resistance.

4.2.1 Open circuit results in relation to oxide characteristics

Open circuit tests measure the potential difference between the sample and the surrounding solution, as no current is applied. Theoretically, the potential difference is proportional to the resistance of the oxide layer separating the reactive substrate from the solution. The open circuit potential of samples thermally oxidized at 600°C and 675°C increased with longer treatment time.

The dependence upon temperature for thickness of the oxide layer is supported in findings by Lee, Yoon, and Yi (2007), who calculated the oxide penetration using an equation derived from weight gain after TO treatments. Using the kinetic values from Lee *et al.* (2007), the oxygen penetration is deeper for the 675°C 1 hour treatment than the depth attained at 600°C for 16 hours,

as seen in Figure 4.1. Therefore, the thermal oxidation process is more dependent upon temperature than time in terms of oxygen penetration and oxide thickness.

Figure 4.1. Calculated values of oxygen penetration into Ti-6Al-4V substrate at 600°C and 675°C (Lee, Yoon, & Yi, 2007)

Although the activation energy for oxygen diffusion differs from the activation energy of oxide formation, the findings of Lee *et al.* (2007) provide insight as to the difference temperature plays in the kinetics of the oxidation. Based on the open circuit trends seen in Figure 4.1, the oxide growth rate of samples treated at 600°C and 675°C are not equal. These trends indicate the oxygen absorption kinetics change with oxidation temperature, supported by the findings of Frangini *et al.* (1994). This group studied the kinetics of thermally oxidized Ti-6Al-4V at 600, 650, and 700°C and observed the linear oxidation rate for the 600°C treatments. However, the 700°C treatments did not possess the same uniform linearity in short and long oxidations. The non-uniform oxidation rate during a short time period at high temperature may explain how the oxide thickness of the samples treated at 675°C dwarfed thicknesses of samples treated at 600°C in the same amount of time. Figure 4.2 graphs the calculated oxide penetration (Lee, Yoon, & Yi,

2007) against the open circuit potential. The oxidation rate for the 675°C 1 hour treatment leads to a greater E_{OC} than the 600°C treatment rate. The small increase in open circuit potential with longer treatments at 675°C illustrates how longer treatment times provide little additional corrosion resistance.

Figure 4.2. Theoretical oxygen penetration vs. open circuit potential shows the different rates of the two thermal processes during the first hour.

Samples treated at 675°C had more noble E_{OC} values than samples treated at 600°C, as their oxides posed greater resistance between the sample substrate and the electrolyte. Based on open circuit potentials, and not on direct measurement, the oxide thickness of the sample treated at 675°C for 1 hour appears thicker than the oxide of the sample treated at 600°C for 16 hours (Figure 3.1), agreeing with theory presented by Lee *et al* (2007). However, the theory of infinitely increasing oxygen penetration with increasing treatment time is disproved in Figure 4.2. As treatment time increased, oxygen penetration increased, but the open circuit potential did not greatly increase. As this group utilized the weight of a sample to assess oxygen penetration, the

increase in weight corresponded with an increase in the density of the oxide, not necessarily the oxide thickness.

According to the findings of Güleriyüz *et al.* (2004), the oxide becomes denser at higher temperatures and longer treatment times. Using XRD analysis, the group determined the presence of more rutile on samples treated at high temperatures and longer times, and anatase on samples treated at lower temperatures and shorter times. As rutile has higher density than anatase, the increase in density with treatment corresponded to the increase in open circuit potentials seen in this study, as higher temperature samples resulted in less porous oxides with higher resistance.

Resistance can be attributed to the thickness or density of the oxide formed on the sample surface, according to Aziz-Kerrzo, Conroy, Fenelon, Farrell, and Breslin (2001). The group measured the resistance of oxide layers on Ti-6Al-4V according to the equivalent circuit seen in Figure 4.3, which indicates the dependence of the potential difference on the oxide layer characteristics. R_s represents the resistance of the solution, R_{pr} and Q_{pr} the resistance and capacitance of the porous oxide layer, and Q_1 , R_1 , and Q_2 represent capacitance and resistance of the oxygen diffusion zone, or barrier layer, respectively.

Figure 4.3. Equivalent circuit used to generate impedance data for Ti-6Al-4V in PBS at 37°C by Azziz-Kerrzo *et al.* (2001).

The open circuit values are a reflection of the resistance formed by the oxygen diffusion zone on the substrate (R_1) and the resistance of the porous outer layer (R_{pr}). The open circuit potentials of both thermal oxidation temperatures increased compared to the untreated sample, as the

increased resistance of the thermal oxide layer created a larger barrier between the substrate and the solution. However, the structure of the resulting oxide was dependent upon the treatment temperature, affecting the cyclic polarization and abrasion resistance of the sample.

4.2.2 Cyclic Polarization evaluation of corrosion resistance

Six of the eight thermal oxidation treatments were successful, serving as effective barriers to corrosion resistance. Samples treated at 600°C for 1 and 16 hours and 675°C for 1, 4, and 16 hours did not breakdown, despite relatively wide the difference in their open circuit potentials. In addition, the current values of the reverse scans were lower than the forward scans, indicating that the surfaces were further passivated by the polarization process. Corrosion potential (E_{CORR}) values above the E_{OC} values indicated that samples had become nobler, and reactions on the sample surfaces would become anodic at higher voltages. N-675-8 experienced breakdown at 1.17mV, a value too high to be considered physiologically relevant, therefore N-675-8 can be considered corrosion resistant in this case. In addition, the evolution of oxygen can occur at 800mV, which may lead to false readings of current increase and breakdown (Rosenbloom & Corbett, 2007).

Unlike the successful treatments, the sample treated at 600°C for 4 hours broke down at 364mV, repassivating far below the potential at which it broke down. The sample followed the open circuit potential trend, but displayed increased current values during the reverse scan. The sample treated at 600°C for 8 hours also displayed increased current values during the reverse scan. Unlike samples treated at 600°C for 1 and 16 hours, higher current values on the reverse scans of samples treated for 4 and 8 hours indicated that the surface was detrimentally modified, not further passivated. Damage from cyclic polarization indicates that the oxide grown at 600°C may be structurally vulnerable to corrosion.

According to Göbel, Haanappel, and Stroosnijder (2001), the diffusion coefficients of oxygen in β titanium are higher than α titanium, at $1.8 \times 10^{-8} \text{ cm}^2/\text{s}$ and $1.8 \pm .3 \times 10^{-10} \text{ cm}^2/\text{s}$, respectively. A higher oxygen diffusion coefficient for β titanium compared to α titanium may have caused structural differences in the thermally-grown oxide (Anandan & Rajam, 2008), as the oxide structures on the β grains differ in thickness and texture from the oxide on the α grains. Anadan and Rajam (2008) also determined that during the oxidation reaction, α oxide dissolves faster than β oxide.

The difference in the oxide structures may be related to the breakdown of samples treated at 600°C for 4 and 8 hours, as one oxide species may have been preferentially dissolved in the CP scan. At these temperatures, the β oxide may have grown in greater proportion than the α oxide, due to its higher diffusion coefficient. A large topographical range with high β “peaks” and low α “valleys” could lead to a slightly more volatile environment in the valleys, causing the deterioration of alpha oxide. The smoother topography of samples treated at 600°C for 1 and 16 hours would not have the variation in environments along the sample surface that cause localized breakdown, as the 1 hour treatment would not create high enough peaks, and the 16 hour treatment would allow the valleys to grow to heights comparable to the peaks. However, without the surface roughness data for N-600-4 and N-600-8, these conclusions are only speculation.

The breakdown of N-675-8 is also contrary to theory, as the open circuit value of N-675-8 fit the trend of samples treated at 675°C . In addition, no pits were found on the surface of the tested area with the SEM, nor was corrosion macroscopically observed on any other portion of the sample. The lack of hysteresis in the CP scan indicates that a pit would have propagated for the duration of the reverse scan, but the pit may not have been observed in the SEM or may have formed between the mask and the surface. In any case, the breakdown occurred at 1.17mV, generally greater than *in vivo* rest potentials.

Although three CP scans showed atypical behavior compared to the other five scans, six of the eight treatments were deemed successful, verifying thermal oxidation as a valid treatment for corrosion resistance.

4.3 Repassivation

Six of the eight thermal oxidation treatments were successful in resisting corrosion during cyclic polarization tests, and to satisfy the second project objective, all treatments were abraded before step polarization to assess their repassivation ability.

The most consistent and smooth repassivation was seen in A-600-1, A-600-16, A-675-1 and A-675-16, with minimal leaks of current during the repassivation process. Each treatment showed more consistent and smooth recovery than A-0, possibly indicating an improvement in repassivation with thermal oxidation. Some samples did exhibit transient effects, such as A-600-8, which repassivated after every potential step, with slight current fluctuations at 200mV polarization. A-675-4 and A-675-8 also displayed the ability to repassivate, although A-675-8 was less stable for 0mV and 200mV applied potentials. The current fluctuations of A-675-4 were at 1.2V, above physiological relevance.

During polarization, the current of A-600-4 fluctuated more than any other treatment, even A-0. An increase in current after polarization would indicate the material's inability to repassivate, releasing electrons to the surrounding solution. Instability during repassivation was depicted by the current fluctuations, which indicated an oxidation reaction occurring on the surface of the sample. As a hypothesis, the fluctuations may have been indicative of multiple oxidation sites, causing increases in current while other sites were reducing and repassivating.

In addition to breakdown in the CP scan, the Potential step scan illustrates how a 4 hour thermal oxidation treatment at 600°C creates a vulnerable oxide regardless of abrasion, as seen by the location of the pits developed in this study. Komotori *et al.* (2001) also found corrosion near,

but not within the abrasion scar in a thermally oxidized Ti-6Al-4V sample that underwent cyclic polarization in a NaCl solution. This provides evidence that the corrosive behavior of thermally oxidized Ti-6Al-4V is more dependent upon the oxide fabricated during treatment than the effects of a single abrasion.

Seven of the eight abraded thermally oxidized samples showed less current fluctuation than the untreated sample, indicating that the oxide layer and oxide diffusion zone beneath were more effective in repassivating than the untreated material. Komotori *et al.* (2001) also found that thermal oxidation of Ti-6Al-4V alloys made the alloys less prone to corrosion after prior abrasion.

4.4 Effects of thermal oxidation

4.4.1 Surface roughness

As seen in this experiment and observed by other researchers, the surface roughness of the Ti-6Al-4V samples increased after thermal oxidation, and the magnitude of surface roughness was dependent upon treatment temperature and time.

Gülyerüz *et al.* (2004) found that the growth of the oxide layer is accompanied by increasing surface roughness, and the increase accelerated with treatment at 650°C, compared to 600°C. The trend seen by Gülyerüz *et al.* (2004) was also seen in this experiment, as the samples treated at 675°C had greater surface roughness values than the samples treated at 600°C.

Great surface roughness encourages osseointegration, which is not desired in ACPs. Therefore, greater roughness values seen in the samples treated at 675°C for 8 and 16 hours would be less advantageous candidates for the ACP application. The sample treated at 600°C for 4 hours also had an outlying surface roughness compared to the lower roughness of samples treated for 1, 8, and 16 hours, but all samples treated at 600°C had lower roughness values than samples treated at 675 °C.

Outliers within the isothermal groups indicate the difference in oxide structure at different thermal oxidation times. A difference in oxide structure was observed by Anadan and Rajam (2008), who determined that the texture and thickness of the oxide was affected by the presence of alpha grains and beta grains on the substrate. The texture and thickness differences were a result of the higher diffusion coefficients of nitrogen and oxygen in beta titanium, compared to alpha titanium (Anadan & Rajam, 2008). The different diffusion coefficients would cause the oxides to grow at uneven rates, contributing to their topographical differences and a high surface roughness at particular treatment times. In the present study, uneven oxide growth was displayed in the SEM images of the surfaces after heat treatment, where the β oxide appears as high peaks.

The abraded sample treated at 600°C for 4 hours had a higher surface roughness than other samples treated at 600°C. The high surface roughness may indicate that after 4 hours more oxygen was absorbed by one phase than the other. According to Anadan and Rajam (2008), alpha oxide is also preferentially dissolved in a polarization experiment, which may be why the sample treated at 600°C for 4 hours broke down in cyclic polarization tests and was unable to repassivate in potential step tests.

The surface roughness for the sample treated at 675°C for 8 hours was also highest among all samples treated at 675°C, but the sample treated for 16 hours was similar to the roughness for the 4 hour treatment. The changing oxidation rates are verified by the findings of Frangini *et al.* (1994), who found that the oxidation rates of different temperatures change with the treatment time, as does the surface morphology. Alpha and beta phases have different maximum dissolution levels for oxygen (Gobel *et al.*, 2001), which may affect the rate at which they are absorbed. This theory was supported by the decreasing surface roughness of the longer treatments, as one species may have started to reach its maximum dissolution level, changing its rate of absorption, while the other species continued to absorb oxygen at its same rate.

Researchers (Güteryüz *et al.*, 2004; Frangini *et al.*, 1994) determined that at oxidation temperatures above 650°C, the surface oxide becomes a dual-layer system; with the surface scale

mostly composed of rutile, but the surface of samples treated at 600°C contain mostly rutile and some anatase. The rate of oxygen absorption and oxide scale growth increased with treatment temperature, but changed according to treatment time (Frangini *et al.*, 1994). The variation in oxide growth was displayed in this study by the change in surface roughness of the samples treated at 675°C.

4.4.2 Microstructure

Microstructural analysis was indicative of any microstructural changes as a result of thermal oxidation, and any bulk changes may be insinuate of surface changes. The analysis provided evidence to help determine whether any microstructural changes occurred, possibly altering material characteristics such as hardness.

The grain size and amount of β phase, was similar across the samples treated at 600°C. Both observations indicate little microstructural change throughout the thermal oxidation treatment, with the microstructure of samples treated for 16 hours appearing similar to the microstructure of samples treated for 4 and 8 hours. Therefore, thermal oxidation treatments at 600°C did not result in microstructural changes.

Changes in β content and the α grain size were seen in the samples treated at 675°C. The size of the α grains grew with increasing treatment time, as the amount of β phase decreased. The change in microstructure is consistent with the findings of Semiatin *et al.* (2004), as the heat treatment caused primary α to grow, and the β to reduce to form secondary platelet α .

The content of α and β in the microstructure may directly relate to the proportion of their respective oxides on the sample surface. Of the sample treated at 600°C for 16 hours, the microstructure and an inverted SEM image of the surface oxide are pictured in Figure 4.4. An inverted SEM surface image and the microstructure of the sample treated at 675°C for 16 hours are presented in Figure 4.5. In both images, the dark regions represent the β content, and the light represent the α . Although the slightly scales differ, the amount of α and β content is similar

between the images. Comparing the SEM image and micrograph does not confirm the possibility that the slight reduction of β particles in the microstructure is related to the accumulation of β oxide on the surface. However, the amount of dark β content in the images is similar, and the possibility of direct proportion cannot be eliminated.

Figure 4.5. Inverted SEM image (a) and microstructure (b) of sample treated at 600°C for 16 hours. Light content represents the α phase and dark content represents the β phase. The β content is similar between the two images, but does not confirm whether the microstructure directly predicts the surface content.

Figure 4.4. Inverted SEM image (a) and microstructure (b) of sample treated at 675°C for 16 hours. Light content represents the α phase and dark content represents the β phase. Amount of β content is not similar between two images, neither confirming nor denying the direct relationship between β content in microstructure and on sample surface.

4.4.3 Hardness

The hardness values (HR_C) of 600°C and 675°C were not significantly different from the hardness of the untreated sample, indicating that the hardness of the material was not altered by the thermal oxidation process. Although hardness values of the two temperatures were statistically different from one another, their values were within the range of the untreated sample hardness values, indicating that the oxidation treatment had little, if any, effect on hardness. Even the 16 hour treatment at 675°C maintained the same range of hardness values. Although major microstructural changes resulting in grain enlargement and decreased hardness may occur at higher temperatures and longer treatment times, but they were not observed in this study.

4.4.4 Microhardness

The increasing trend in microhardness with temperature and time is supported by Gülyerüz *et al.* (2004), who reported that surface hardness increases with thermal oxidation as the oxygen diffuses into the matrix and places strain into the lattice. The microhardness values are in accordance with findings by Dearnley *et al.* (2004). After thermal oxidation, the microhardness (100g) increased from 369 HV in the untreated sample to 755 HV in the thermally oxidized sample (Dearnley *et al.*, 2004). An increasing trend in microhardness with treatment time seen in this study indicates that the 600°C 16 hour treatment microhardness value of 560HV is on the same scale as Dearnley *et al.* (2004), who thermally oxidized at 625°C for 36 hours.

The microhardness of A-600-4 was lower than the other samples treated at 600°C, but its surface roughness was the highest of the group. Therefore, in accordance with the findings of other researchers (Gülyerüz *et al.* 2004), the high surface roughness may have affected the indentations by altering the indentation width.

4.4.5 Abrasion characteristics

Dearnley *et al.* (2004) also determined that the increased thickness and microhardness of the oxide layer contained greater residual stresses than untreated samples, which was seen by the

delamination behavior of the samples with higher microhardness values. The increased strain on the lattice as a result of thermal oxidation treatments at the higher temperature (Gülyerüz *et al.*, 2004) resulted in a more brittle surface oxide scale, which delaminated during both hardness tests and abrasion tests.

The delamination in N-675-16, A-675-16, A-675-8, and A-675-4 appeared to separate the rutile scale of the dual-layer oxide that formed at higher temperatures. The oxide fractured above the oxygen diffusion zone, similar to the findings of Dearnley *et al.* (2004), who determined that higher temperatures create outer oxide layers that poorly adhere to the substrate and fracture along the TiO₂-oxygen diffusion zone interface. However, the inner oxygen diffusion zone is far more adherent (Frangini *et al.* 1994, Borgioli *et al.* 2004), but not as hard or thick as the outer scale. In addition, the accelerated growth rate at the higher temperature may have placed more intrinsic and thermal stress on the oxide (Gülyerüz *et al.*, 2004), causing its brittle behavior during abrasion.

The lack of delamination in samples treated at 600°C is in accordance with the findings of researchers (Gülyerüz *et al.*, 2004, Frangini *et al.*, 1994), as longer treatments resulted in fewer defects, more rutile, and better adhesion of the oxide to the substrate.

The abrasion width varied from sample to sample, indicating that the thermal oxidation treatments affected the abrasion resistance. Larger abrasion widths were seen in samples treated at higher temperatures and times. As the diamond-tip indenter had a tip radius of 120°, a larger width would indicate deeper penetration of the indenter into the sample surface. As all samples were abraded with the same indenter, weight, and rate, less resistance to abrasion was displayed in samples with wider abrasion scars.

Similar to the findings of Li *et al.* (2004), thermal oxidation treatments that altered the mechanical properties of a material were ineffective in improving wear resistance. The Rockwell hardness was not affected by the thermal oxidation treatments, but the wear resistance and

damage to the material were affected, particularly in samples treated at 675°C. A-675-16 showed the widest abrasion length and microstructural grain growth, partially verifying the findings of Li *et al.* (2004), as the mechanical properties were not altered, but the wear resistance was not increased. Samples treated for shorter time periods had smaller abrasion scars, indicating more wear resistance than the sample treated at 675°C for 16 hours, with no statistical difference in hardness.

5 CONCLUSION

Overall, thermal oxidation appears as a viable option for enhancing corrosion and wear performance in Ti-6Al-4V anterior cervical plates. Samples treated at 600°C for 1 and 16 hours showed improved characteristics, including microhardness increase, more noble open circuit potential, resistance to corrosion, repassivation ability, and oxide adhesion. The sample treated for 1 hour displayed the positive attributes to a lesser degree, with a less noble open circuit potential and lower microhardness values. Therefore, treatments at 600°C for 16 hours created a protective surface oxide that was resistant to corrosion and abrasion, and with lower surface roughness than samples treated at 675°C.

Despite success in the cyclic polarization and Potential step tests, the samples treated at 675°C for 4, 8, and 16 hours had the highest microhardness and the most brittle scale due to their dual-layer oxide, causing the most delamination after a single abrasion. In addition to the high surface roughness measured in the treatments, samples treated at 675°C for longer than 1 hour would not create viable surface oxides for an ACP that may undergo surgical damage.

Thermal oxidation treatments altered the corrosion resistance, repassivation ability, surface roughness, microhardness, and adhesion of the protective oxide layer of Ti-6Al-4V. Trends with increasing time and temperature were seen in the open circuit potential values, microhardness, and quantity of delamination. Hardness was not significantly different for thermally oxidized samples compared to untreated samples, indicating that any grain growth from heat treatment did not alter the bulk material properties. Outliers in cyclic polarization tests, Potential step tests, and surface roughness measurements reflected the characteristics of the fabricated oxide structures and the propensity of the oxide to particular temperature and time combinations.

In non-abraded thermal oxidation treatments, the corrosion resistance was improved in some samples and reduced in others. All samples treated at 675°C were successfully resistant to breakdown, although the sample treated for 8 hours broke down at a physiologically irrelevant 1.7V. The samples treated at 600°C for 1 and 16 hours were also successful and did not breakdown during cyclic polarization. The oxide layers produced by 4 and 8 hour treatments broke down and retained surface damage, indicating the susceptibility of specific treatment times and temperatures to corrosion.

Of the abraded samples, all but the sample treated at 600°C for 4 hours were able to repassivate. Although some samples displayed leaks of current during Potential step tests, they were eventually able to repassivate, restoring their passive layer and decreasing the flow of current to the surrounding environment. As speculation, preferential dissolution of one species of oxide may have occurred in the samples treated at 600°C for 4 hours. Pits located on the surface of both the non-abraded and abraded samples, away from the abrasion scar, indicated the surface's lack of corrosion resistance.

Of samples treated at 675°C, the highest surface roughness of non-abraded and abraded samples were the 4 and 8 hour treatments, respectively. The highest surface roughness of abraded samples treated at 600°C was after 4 hours of treatment. The non-linear increases in surface roughness of both temperatures represented heterogeneous oxide structure, with growth rates of alpha and beta oxides dependent on temperature and time. In addition, the higher surface roughness seen in samples treated at 675°C would be advantageous for osseointegration, which is not desired for the ACP application.

6 RECOMMENDATIONS FOR FUTURE WORK

Corrosion resistance and surface effects from thermal oxidation of Ti-6Al-4V may be further explored in many ways, but two advancements are specific to this study: analysis of the oxide produced by 600°C for times within a short range of 4 hours; studies of oxides produced at 600°C for longer than 16 hours.

In this study, the structure of the oxide produced at 600°C for 4 hours was an outlier in surface roughness and breakdown. A study of oxides produced at times between 1 and 8 hours may provide insight to the structural evolution of the oxide during thermal oxidation and the growth rates of the alpha and beta oxides. Additional techniques such as x-ray diffraction (XRD) or an AFM with higher resolution would provide data to compare to resources reviewed in this study.

Oxides produced at 600°C for longer than 16 hours could be investigated to determine whether the microhardness and open circuit potential continue to increase, and whether the surface roughness and adhesion continue to change with treatment time. The oxides could also be corrosion tested under different conditions, such as *in vitro* wear to simulate abrasion from screws during arthrodesis. Such testing could determine whether a 16 hour treatment is the minimum time necessary to achieve desirable properties, or whether a longer treatment is required.

An additional study may further analyze the cyclic polarization behavior of the thermally oxidized samples for corrosion rates, behavior in the transpassive region, and current density monitoring. Much material information apart from breakdown potential can be obtained from cyclic polarization graphs, and a comparison of different thermal oxidation treatments, or even different surface treatments, can be compared to find optimal treatment methods.

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APPENDIX A: AFM Data of Non-Abraded Samples

N-0 before heat treatment

Nanosurf Image Document

File: Image1

Parameter:

-- Area Roughness --	Lines	= 256	Excitation ampl.	= 0.57 V	
Area	= 618 μm^2	X-Slope	= 500m°	Error range	= 20 V
Sa	= 18.154nm	Y-Slope	= 7.5 °	-- Global --	
Sq	= 23.653nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 346.22nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 241.32nm	Y-Pos	= 0 m	Head type	= E22-AFM
Sv	= -104.9nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= -188.22fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --	Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2	
Ra	= 19.519nm	Date	= 16-07-2008	Controller S/N	= 023-06-252
Rq	= 27.869nm	Time	= 14:55:00	-- Module --	
Ry	= 235.94nm	-- Feedback --		Controller Board	= 2
Rp	= 159.92nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -76.028nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 15.651pm	I-Gain	= 1800	AFM Extension Module	= 1
-- Scan --	Tip voltage	= 0 V	Video Module	= 0	
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 183.647kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image2

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.55 V
Area	= 618pm ²	X-Slope	= 3.5 °	Error range	= 20 V
Sa	= 28.97nm	Y-Slope	= 7 °	-- Global --	
Sq	= 35.363nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 339.68nm	X-Pos	= 8.9μm	Cantilever type	= ACLA
Sp	= 230.15nm	Y-Pos	= 18μm	Head type	= EZ2-AFM
Sv	= -109.54nm	Z-Plane	= 2.7μm	Scan head	= 10-07-239.hed
Sm	= 117.72pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 28.992nm	Date	= 16-07-2008	Controller S/N	= 023-06-252
Rq	= 34.312nm	Time	= 15:16:51	-- Module --	
Ry	= 185.55nm	-- Feedback --		Controller Board	= 2
Rp	= 103.68nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -81.867nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 161.44pm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 183.627kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image3

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.56 V
Area	= 618 μm^2	X-Slope	= 3.5 °	Error range	= 20 V
Sa	= 18.551 nm	Y-Slope	= 6 °	-- Global --	
Sq	= 23.692 nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 270.31 nm	X-Pos	= 8.9 μm	Cantilever type	= ACLA
Sp	= 163.31 nm	Y-Pos	= 18 μm	Head type	= EZ2-AFM
Sv	= -107.01 nm	Z-Plane	= 0 μm	Scan head	= 10-07-239.hed
Sm	= 215.43 μm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 21.657 nm	Date	= 16-07-2008	Controller S/N	= 023-06-252
Rq	= 28.23 nm	Time	= 15:28:36	-- Module --	
Ry	= 153.08 nm	-- Feedback --		Controller Board	= 2
Rp	= 88.668 nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -64.411 nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 206.74 μm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 183.617 kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-0 after potentiodynamic polarization Nanosurf Image Document

File: Image4

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.46 V
Area	= 559pm ²	X-Slope	= 6.5 °	Error range	= 20 V
Sa	= 28.424nm	Y-Slope	= 3.5 °	-- Global --	
Sq	= 36.181nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 303.21nm	X-Pos	= -1.1μm	Cantilever type	= ACLA
Sp	= 131.26nm	Y-Pos	= 19μm	Head type	= EZ2-AFM
Sv	= -171.95nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 210.69pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 30.861nm	Date	= 21-07-2008	Controller S/N	= 023-06-252
Rq	= 36.256nm	Time	= 16:11:35	-- Module --	
Ry	= 148.65nm	-- Feedback --		Controller Board	= 2
Rp	= 75.506nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -73.145nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 295.69pm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 24μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 183.577kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image1

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.29 V
Area	= 474.3pm ²	X-Slope	= 6 °	Error range	= 20 V
Sa	= 13.425nm	Y-Slope	= 4 °	-- Global --	
Sq	= 17.56nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 173.32nm	X-Pos	= 15µm	Cantilever type	= ACLA
Sp	= 107.43nm	Y-Pos	= 13µm	Head type	= EZ2-AFM
Sv	= -65.891nm	Z-Plane	= 2.5µm	Scan head	= 10-07-239.hed
Sm	= 464.28pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 13.018nm	Date	= 10-08-2008	Controller S/N	= 023-06-252
Rq	= 16.301nm	Time	= 14:39:56	-- Module --	
Ry	= 82.022nm	-- Feedback --		Controller Board	= 2
Rp	= 46.043nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -35.978nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 238.86pm	I-Gain	= 1400	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 179.207kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image2

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.21 V
Area	= 629.9pm ²	X-Slope	= 5.5 °	Error range	= 20 V
Sa	= 20.656nm	Y-Slope	= 5 °	-- Global --	
Sq	= 25.905nm	Rotation	= -0.888°	Op. mode	= Dynamic Force
Sy	= 211.06nm	X-Pos	= 15 μ m	Cantilever type	= ACLA
Sp	= 118.6nm	Y-Pos	= 13 μ m	Head type	= EZ2-AFM
Sv	= -92.459nm	Z-Plane	= 0 μ m	Scan head	= 10-07-239.hed
Sm	= 8.0434fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 14.549nm	Date	= 10-08-2008	Controller S/N	= 023-06-252
Rq	= 19.349nm	Time	= 14:47:19	-- Module --	
Ry	= 103.32nm	-- Feedback --		Controller Board	= 2
Rp	= 41.222nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -62.096nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 1.6087fm	I-Gain	= 1400	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μ m	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 179.207kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-600-1 after heat treatment

Nanosurf Image Document

File: Image5

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.18 V
Area	= 637.4pm ²	X-Slope	= -500m°	Error range	= 20 V
Sa	= 30.223nm	Y-Slope	= 0m°	-- Global --	
Sq	= 39.409nm	Rotation	= -1.78°	Op. mode	= Dynamic Force
Sy	= 286.55nm	X-Pos	= -15μm	Cantilever type	= ACLA
Sp	= 191.77nm	Y-Pos	= -16μm	Head type	= EZ2-AFM
Sv	= -94.778nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 208.84pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 30.726nm	Date	= 03-06-2008	Controller S/N	= 023-06-252
Rq	= 40.163nm	Time	= 21:43:16	-- Module --	
Ry	= 203.47nm	-- Feedback --		Controller Board	= 2
Rp	= 126.14nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -77.334nm	P-Gain	= 8000	AFM Dynamic Module	= 2
Rm	= 267.69pm	I-Gain	= 2000	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.2 s	Vibration freq.	= 184.532kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image6

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.19 V
Area	= 637.4µm²	X-Slope	= -500m°	Error range	= 20 V
Sa	= 62.714nm	Y-Slope	= 0m°	-- Global --	
Sq	= 76.151nm	Rotation	= -1.78f°	Op. mode	= Dynamic Force
Sy	= 500.77nm	X-Pos	= -15µm	Cantilever type	= ACLA
Sp	= 259.88nm	Y-Pos	= -16µm	Head type	= EZ2-AFM
Sv	= -240.9nm	Z-Plane	= 0µm	Scan head	= 10-07-239.hed
Sm	= 3.071µm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 61.484nm	Date	= 03-06-2008	Controller S/N	= 023-06-252
Rq	= 71.423nm	Time	= 21:52:52	-- Module --	
Ry	= 272.42nm	-- Feedback --		Controller Board	= 2
Rp	= 132.21nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -140.22nm	P-Gain	= 8000	AFM Dynamic Module	= 2
Rm	= 104.6µm	I-Gain	= 2000	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.2 s	Vibration freq.	= 184.532kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-600-1 after CP

Nanosurf Image Document

File: Image11

Parameter:

-- Area Roughness --	Lines	= 256	Excitation ampl.	= 0.22 V	
Area	= 618pm ²	X-Slope	= 1 °	Error range	= 20 V
Sa	= 38.725nm	Y-Slope	= 500m°	-- Global --	
Sq	= 48.252nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 445.74nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 305.42nm	Y-Pos	= 0 m	Head type	= E22-AFM
Sv	= -140.32nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 3157.8fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --	Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2	
Ra	= 31.789nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 37.56nm	Time	= 13:03:09	-- Module --	
Ry	= 160.46nm	-- Feedback --		Controller Board	= 2
Rp	= 85.183nm	Set point	= 44 %	AFM Basic Module	= 2
Rv	= -75.276nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -103.78pm	I-Gain	= 2300	AFM Extension Module	= 1
-- Scan --	Tip voltage	= 0.1 V	Video Module	= 0	
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.347kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image12

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.22 V
Area	= 618pm^2	X-Slope	= 1 °	Error range	= 20 V
Sa	= 35.11nm	Y-Slope	= 500m°	-- Global --	
Sq	= 44.337nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 392.82nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 256.29nm	Y-Pos	= 0 m	Head type	= E22-AFM
Sv	= -136.53nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 210.56pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 28.649nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 35.294nm	Time	= 13:16:22	-- Module --	
Ry	= 182.81nm	-- Feedback --		Controller Board	= 2
Rp	= 79.99nm	Set point	= 44 %	AFM Basic Module	= 2
Rv	= -102.82nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 238.86pm	I-Gain	= 2200	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0.1 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.344kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image13

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.23 V
Area	= 618 μm^2	X-Slope	= 1 °	Error range	= 20 V
Sa	= 37.157nm	Y-Slope	= 1 °	-- Global --	
Sq	= 45.097nm	Rotation	= -0.888°	Op. mode	= Dynamic Force
Sy	= 307.42nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 180.6nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -126.83nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 163.6pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 43.541nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 61.145nm	Time	= 13:24:51	-- Module --	
Ry	= 275.58nm	-- Feedback --		Controller Board	= 2
Rp	= 174.01nm	Set point	= 44 %	AFM Basic Module	= 2
Rv	= -101.58nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 262.74pm	I-Gain	= 2100	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0.1 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.344kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-600-4 after CP

Nanosurf Image Document

File: Image14

Parameter:

-- Tool --		Time/Line	= 0.6 s	Vibration ampl.	= 0.7 V
Width	= 12.86µm	Points	= 256	Excitation ampl.	= 0.23 V
Height	= 12.86µm	Lines	= 256	Error range	= 20 V
-- Area Roughness --		X-Slope	= -1 °	-- Global --	
Area	= 168pm ²	Y-Slope	= 1.5 °	Op. mode	= Dynamic Force
Sa	= 113.82nm	Rotation	= 90 °	Cantilever type	= ACLA
Sq	= 135.79nm	X-Pos	= 0 m	Head type	= EZ2-AFM
Sy	= 774.88nm	Y-Pos	= 0 m	Scan head	= 10-07-239.hed
Sp	= 441nm	Z-Plane	= 0 m	Software ver.	= 1-5-1-0
Sv	= -333.88nm	Overscan	= 5 %	Firmware ver.	= 2-1-1-2
Sm	= -86.08nm	Const.Height-Mode	= Disabled	Controller S/N	= 023-06-252
-- Line Roughness --		Date	= 08-08-2008	-- Module --	
Ra	= 99.685nm	Time	= 13:46:44	Controller Board	= 2
Rq	= 118nm	-- Feedback --		AFM Basic Module	= 2
Ry	= 482.01nm	Set point	= 48 %	AFM Dynamic Module	= 2
Rp	= 284.26nm	P-Gain	= 10000	AFM Extension Module	= 1
Rv	= -197.75nm	I-Gain	= 1900	Video Module	= 0
Rm	= 916.92pm	Tip voltage	= 0.1 V	Signal Module S	= 0
-- Scan --		Feedback mode	= Free	Signal Module A	= 0
Image size	= 25µm	Feedback algo.	= Adaptive PI	Nanosurf Report	= 0
Scan direction	= Down	Vibration freq.	= 181.334kHz	Scripting Interface	= 0

Nanosurf Image Document

File: Image15

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.22 V
Area	= 618 μm^2	X-Slope	= 500m°	Error range	= 20 V
Sa	= 35.054nm	Y-Slope	= 3 °	-- Global --	
Sq	= 43.557nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 336.31nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 178.06nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -158.24nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= -3016.3fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 31.146nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 39.703nm	Time	= 14:15:41	-- Module --	
Ry	= 214.23nm	-- Feedback --		Controller Board	= 2
Rp	= 136.9nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -77.324nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 46.949pm	I-Gain	= 1800	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0.1 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 181.334kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image16

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.22 V
Area	= 618µm ²	X-Slope	= 2 °	Error range	= 20 V
Sa	= 27.188nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 33.671nm	Rotation	= -14.2°	Op. mode	= Dynamic Force
Sy	= 245.22nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 146.44nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -98.784nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 1642.5fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 35.159nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 42.384nm	Time	= 14:26:24	-- Module --	
Ry	= 178.59nm	-- Feedback --		Controller Board	= 2
Rp	= 88.164nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -90.428nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 77.424pm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0.1 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 181.344kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-600-8 after CP

Nanosurf Image Document

File: Image1

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.37 V
Area	= 618pm ²	X-Slope	= 2.5 °	Error range	= 20 V
Sa	= 58.331nm	Y-Slope	= 500m°	-- Global --	
Sq	= 76.196nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 662.92nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 434.25nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -228.67nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 583.95fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 37.287nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 47.477nm	Time	= 11:37:56	-- Module --	
Ry	= 225.4nm	-- Feedback --		Controller Board	= 2
Rp	= 134.14nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -91.26nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 66.717pm	I-Gain	= 2200	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 181.367kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image2

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.27 V
Area	= 618pm^2	X-Slope	= 2 °	Error range	= 20 V
Sa	= 65.672nm	Y-Slope	= 500m°	-- Global --	
Sq	= 82.834nm	Rotation	= -0.888f°	Op. mode	= Dynamic Force
Sy	= 626.02nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 395.88nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -230.15nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 203.95pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 57.071nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 70.466nm	Time	= 11:50:00	-- Module --	
Ry	= 365.83nm	-- Feedback --		Controller Board	= 2
Rp	= 212.6nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -153.23nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 254.51pm	I-Gain	= 2100	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.367kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image4

Parameter:

-- Area Roughness --			Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 618 μm ²	X-Slope	= 2 °	Error range	= 20 V	
Sa	= 50.943 nm	Y-Slope	= 500 m°	-- Global --		
Sq	= 65.991 nm	Rotation	= -0.888 f°	Op. mode	= Dynamic Force	
Sy	= 548.01 nm	X-Pos	= 0 fm	Cantilever type	= ACLA	
Sp	= 370.57 nm	Y-Pos	= 0 fm	Head type	= EZ2-AFM	
Sv	= -177.43 nm	Z-Plane	= 0 fm	Scan head	= 10-07-239.hed	
Sm	= 79.699 pm	Overscan	= 5 %	Software ver.	= 1-5-1-0	
-- Line Roughness --			Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 42.079 nm	Date	= 08-08-2008	Controller S/N	= 023-06-252	
Rq	= 51.877 nm	Time	= 12:04:42	-- Module --		
Ry	= 236.37 nm	-- Feedback --		Controller Board	= 2	
Rp	= 124.31 nm	Set point	= 48 %	AFM Basic Module	= 2	
Rv	= -112.06 nm	P-Gain	= 10000	AFM Dynamic Module	= 2	
Rm	= -9.8821 pm	I-Gain	= 2100	AFM Extension Module	= 1	
-- Scan --			Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0	
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0	
Time/Line	= 0.4 s	Vibration freq.	= 181.357 kHz	Nanosurf Report	= 0	
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0	

N-600-16 after heat treatment

Nanosurf Image Document

File: Image5

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 618pm ²	X-Slope	= -1 °	Error range	= 20 V
Sa	= 77.73nm	Y-Slope	= 1.5 °	-- Global --	
Sq	= 96.645nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 659.97nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 356.02nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -303.94nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 208.15pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 80.528nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 98.613nm	Time	= 14:46:27	-- Module --	
Ry	= 429.72nm	-- Feedback --		Controller Board	= 2
Rp	= 232.69nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -197.03nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 196.85pm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image6

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.24 V
Area	= 618µm²	X-Slope	= -1 °	Error range	= 20 V
Sa	= 67.184nm	Y-Slope	= 1.5 °	-- Global --	
Sq	= 83.178nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 561.08nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 303.94nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -257.13nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= -98.129fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 43.861nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 52.976nm	Time	= 14:57:32	-- Module --	
Ry	= 242.69nm	-- Feedback --		Controller Board	= 2
Rp	= 138.64nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -104.06nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 1.6087fm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image8

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 618pm^2	X-Slope	= -1 °	Error range	= 20 V
Sa	= 73.116nm	Y-Slope	= 1 °	-- Global --	
Sq	= 90.555nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 670.09nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 391.87nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -278.22nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 42.471pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 59.873nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 73.576nm	Time	= 15:13:36	-- Module --	
Ry	= 336.52nm	-- Feedback --		Controller Board	= 2
Rp	= 192.36nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -144.16nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 43.655pm	I-Gain	= 1600	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-600-16 after CP

Nanosurf Image Document

File: Image5

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.17 V
Area	= 629.9µm ²	X-Slope	= -500m°	Error range	= 20 V
Sa	= 81.339nm	Y-Slope	= -500m°	-- Global --	
Sq	= 103.77nm	Rotation	= -1.78f°	Op. mode	= Dynamic Force
Sy	= 1029.4nm	X-Pos	= 6.3µm	Cantilever type	= ACLA
Sp	= 685.59nm	Y-Pos	= 19µm	Head type	= EZ2-AFM
Sv	= -343.79nm	Z-Plane	= 0µm	Scan head	= 10-07-239.hed
Sm	= 2099.3fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 85.601nm	Date	= 10-08-2008	Controller S/N	= 023-06-252
Rq	= 103.66nm	Time	= 15:33:48	-- Module --	
Ry	= 449.12nm	-- Feedback --		Controller Board	= 2
Rp	= 268.73nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -180.39nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 1648.9fm	I-Gain	= 1100	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.237kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image6

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.17 V
Area	= 629.9 μm^2	X-Slope	= -500 m°	Error range	= 20 V
Sa	= 102.43 nm	Y-Slope	= -500 m°	-- Global --	
Sq	= 136.38 nm	Rotation	= -1.78 f°	Op. mode	= Dynamic Force
Sy	= 1202.7 nm	X-Pos	= 6.3 μm	Cantilever type	= ACLA
Sp	= 832.34 nm	Y-Pos	= 19 μm	Head type	= EZ2-AFM
Sv	= -370.36 nm	Z-Plane	= 0 μm	Scan head	= 10-07-239.hed
Sm	= 210.81 pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 81.618 nm	Date	= 10-08-2008	Controller S/N	= 023-06-252
Rq	= 99.092 nm	Time	= 15:41:38	-- Module --	
Ry	= 464.09 nm	-- Feedback --		Controller Board	= 2
Rp	= 279.7 nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -184.39 nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 210.03 pm	I-Gain	= 1100	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.247 kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-675-1 after heat treatment

Nanosurf Image Document

File: Image3

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.27 V
Area	= 618 μm ²	X-Slope	= 2 °	Error range	= 20 V
Sa	= 62.134 nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 78.277 nm	Rotation	= -14.2 f°	Op. mode	= Dynamic Force
Sy	= 613.58 nm	X-Pos	= 0 fm	Cantilever type	= ACLA
Sp	= 366.99 nm	Y-Pos	= 0 fm	Head type	= EZ2-AFM
Sv	= -246.59 nm	Z-Plane	= 0 fm	Scan head	= 10-07-239.hed
Sm	= 93.121 μm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 56.218 nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 70.657 nm	Time	= 14:27:26	-- Module --	
Ry	= 324.29 nm	-- Feedback --		Controller Board	= 2
Rp	= 174.82 nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -149.48 nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 86.484 μm	I-Gain	= 1800	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.347 kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image4

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.26 V
Area	= 180.8pm^2	X-Slope	= 2 °	Error range	= 20 V
Sa	= 112.4nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 133.39nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 620.33nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 452.17nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -168.15nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 101.39nm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 111.3nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 124.19nm	Time	= 14:37:01	-- Module --	
Ry	= 510.68nm	-- Feedback --		Controller Board	= 2
Rp	= 228.65nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -282.03nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 16.474pm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.347kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image3

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.27 V
Area	= 618 μm^2	X-Slope	= 2 °	Error range	= 20 V
Sa	= 62.134 nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 78.277 nm	Rotation	= -14.2 °	Op. mode	= Dynamic Force
Sy	= 613.58 nm	X-Pos	= 0 fm	Cantilever type	= ACLA
Sp	= 366.99 nm	Y-Pos	= 0 fm	Head type	= EZ2-AFM
Sv	= -246.59 nm	Z-Plane	= 0 fm	Scan head	= 10-07-239.hed
Sm	= 93.121 μm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 56.218 nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 70.657 nm	Time	= 14:27:26	-- Module --	
Ry	= 324.29 nm	-- Feedback --		Controller Board	= 2
Rp	= 174.82 nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -149.48 nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 86.484 μm	I-Gain	= 1800	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.347 kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-675-1 after CP

Nanosurf Image Document

File: Image6

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.21 V
Area	= 629.9µm ²	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 93.521nm	Y-Slope	= 4.5 °	-- Global --	
Sq	= 117.37nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 858.8nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 508.05nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -350.75nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 22.967µm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 92.697nm	Date	= 09-08-2008	Controller S/N	= 023-06-252
Rq	= 111.7nm	Time	= 16:12:30	-- Module --	
Ry	= 481.38nm	-- Feedback --		Controller Board	= 2
Rp	= 260.26nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -221.11nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 35.418µm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Idle	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 181.307kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image7

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.22 V
Area	= 629.9 μm^2	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 80.775nm	Y-Slope	= 4.5 °	-- Global --	
Sq	= 100.64nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 663.55nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 374.37nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -289.18nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= -3.3895 μm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 69.139nm	Date	= 09-08-2008	Controller S/N	= 023-06-252
Rq	= 82.023nm	Time	= 16:18:18	-- Module --	
Ry	= 372.79nm	-- Feedback --		Controller Board	= 2
Rp	= 184.67nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -188.12nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -64.242 μm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 181.307kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image8

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.22 V
Area	= 629.9 μ m ²	X-Slope	= 3.5 °	Error range	= 20 V
Sa	= 90.669nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 111.77nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 755.91nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 419.49nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -336.41nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= -3157.8fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 71.963nm	Date	= 09-08-2008	Controller S/N	= 023-06-252
Rq	= 92.506nm	Time	= 16:27:29	-- Module --	
Ry	= 455.44nm	-- Feedback --		Controller Board	= 2
Rp	= 227.5nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -227.94nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -97.188pm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μ m	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.307kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-675-4 after heat treatment

Nanosurf Image Document

File: Image18

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.24 V
Area	= 638.1 μm^2	X-Slope	= -500m°	Error range	= 20 V
Sa	= 291.96nm	Y-Slope	= -1.5 °	-- Global --	
Sq	= 339.79nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 1628.6nm	X-Pos	= 5.5 μm	Cantilever type	= ACLA
Sp	= 808.09nm	Y-Pos	= -7.7 μm	Head type	= EZ2-AFM
Sv	= -820.53nm	Z-Plane	= 0 μm	Scan head	= 10-07-239.hed
Sm	= -9.4478pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 288.48nm	Date	= 04-06-2008	Controller S/N	= 023-06-252
Rq	= 334.02nm	Time	= 21:39:22	-- Module --	
Ry	= 1256.3nm	-- Feedback --		Controller Board	= 2
Rp	= 624.25nm	Set point	= 44 %	AFM Basic Module	= 2
Rv	= -632.01nm	P-Gain	= 12000	AFM Dynamic Module	= 2
Rm	= -22.237pm	I-Gain	= 2100	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.2 s	Vibration freq.	= 184.509kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image21

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.24 V
Area	= 638.1 μ m ²	X-Slope	= -500m°	Error range	= 20 V
Sa	= 245.69nm	Y-Slope	= -1.5 °	-- Global --	
Sq	= 305.19nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 1757.2nm	X-Pos	= 5.5 μ m	Cantilever type	= ACLA
Sp	= 1094.9nm	Y-Pos	= -7.7 μ m	Head type	= EZ2-AFM
Sv	= -662.39nm	Z-Plane	= 0 μ m	Scan head	= 10-07-239.hed
Sm	= 2514.4fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 294.93nm	Date	= 04-06-2008	Controller S/N	= 023-06-252
Rq	= 344.88nm	Time	= 21:51:10	-- Module --	
Ry	= 1280.1nm	-- Feedback --		Controller Board	= 2
Rp	= 695.25nm	Set point	= 44 %	AFM Basic Module	= 2
Rv	= -584.84nm	P-Gain	= 12000	AFM Dynamic Module	= 2
Rm	= 37.066 μ m	I-Gain	= 2200	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μ m	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.2 s	Vibration freq.	= 184.509kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image23

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.23 V
Area	= 638.1pm ²	X-Slope	= -500m°	Error range	= 20 V
Sa	= 243.1nm	Y-Slope	= -1.5 °	-- Global --	
Sq	= 297.45nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 1750.3nm	X-Pos	= 5.5µm	Cantilever type	= ACLA
Sp	= 974.03nm	Y-Pos	= -7.7µm	Head type	= EZ2-AFM
Sv	= -776.25nm	Z-Plane	= 0µm	Scan head	= 10-07-239.hed
Sm	= 204.74pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 197.08nm	Date	= 04-06-2008	Controller S/N	= 023-06-252
Rq	= 255.02nm	Time	= 22:04:32	-- Module --	
Ry	= 1171.3nm	-- Feedback --		Controller Board	= 2
Rp	= 396.14nm	Set point	= 44 %	AFM Basic Module	= 2
Rv	= -775.14nm	P-Gain	= 12000	AFM Dynamic Module	= 2
Rm	= 155.67pm	I-Gain	= 2000	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 184.509kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-675-4 after CP

Nanosurf Image Document

File: Image1

Parameter:

-- Area Roughness --	Lines	= 256	Excitation ampl.	= 0.35 V	
Area	= 629.9pm^2	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 130.04nm	Y-Slope	= 1 °	-- Global --	
Sq	= 161.41nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 1125.3nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 587.75nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -537.57nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 168.16pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --	Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2	
Ra	= 116.97nm	Date	= 09-08-2008	Controller S/N	= 023-06-252
Rq	= 139.83nm	Time	= 15:21:58	-- Module --	
Ry	= 598.61nm	-- Feedback --		Controller Board	= 2
Rp	= 351.77nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -246.84nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 250.39pm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --	Tip voltage	= 0 V	Video Module	= 0	
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.197kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image2

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 629.9pm^2	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 113.8nm	Y-Slope	= 1 °	-- Global --	
Sq	= 140.69nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 961.91nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 576.79nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -385.12nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 85.538pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 97.317nm	Date	= 09-08-2008	Controller S/N	= 023-06-252
Rq	= 117.79nm	Time	= 15:28:05	-- Module --	
Ry	= 550.11nm	-- Feedback --		Controller Board	= 2
Rp	= 283.49nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -266.62nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 825.25fm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.247kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image3

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.24 V
Area	= 629.9pm ²	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 123.35nm	Y-Slope	= 1 °	-- Global --	
Sq	= 155.83nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 1090.5nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 672.09nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -418.44nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 150.01pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 81.927nm	Date	= 09-08-2008	Controller S/N	= 023-06-252
Rq	= 99.046nm	Time	= 15:35:43	-- Module --	
Ry	= 425.29nm	-- Feedback --		Controller Board	= 2
Rp	= 215.27nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -210.02nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 112.02pm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.237kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-675-8 after heat treatment

Nanosurf Image Document

File: Image10

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.24 V
Area	= 618pm ²	X-Slope	= 4.5 °	Error range	= 20 V
Sa	= 104.1nm	Y-Slope	= -0.444f°	-- Global --	
Sq	= 125.12nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 750.85nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 429.61nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -321.23nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 210.7pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 120.51nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 140.44nm	Time	= 15:30:05	-- Module --	
Ry	= 532.19nm	-- Feedback --		Controller Board	= 2
Rp	= 239.69nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -292.51nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 158.96pm	I-Gain	= 1800	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image16

Parameter:

-- Area Roughness --	Lines	= 256	Excitation ampl.	= 0.23 V	
Area	= 618 μm^2	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 229.83nm	Y-Slope	= -2.5 °	-- Global --	
Sq	= 276.71nm	Rotation	= -14.2°	Op. mode	= Dynamic Force
Sy	= 1416.1nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 725.44nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -690.65nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 71.465pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --	Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2	
Ra	= 215.48nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 274.64nm	Time	= 15:59:51	-- Module --	
Ry	= 1007.9nm	-- Feedback --		Controller Board	= 2
Rp	= 652.63nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -355.24nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 272.63pm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --	Tip voltage	= 0 V	Video Module	= 0	
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image19

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.23 V
Area	= 618 μm^2	X-Slope	= 3 °	Error range	= 20 V
Sa	= 156.85nm	Y-Slope	= -1 °	-- Global --	
Sq	= 196.99nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 1235.6nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 766.98nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -468.62nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= -4.8695 μm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 168.47nm	Date	= 01-08-2008	Controller S/N	= 023-06-252
Rq	= 208.6nm	Time	= 16:32:38	-- Module --	
Ry	= 852.05nm	-- Feedback --		Controller Board	= 2
Rp	= 413.26nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -438.79nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -95.541 μm	I-Gain	= 1600	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0.1 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.6 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-675-8 after CP

Nanosurf Image Document

File: Image1

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 618µm²	X-Slope	= 3 °	Error range	= 20 V
Sa	= 198.18nm	Y-Slope	= -1.5 °	-- Global --	
Sq	= 251.11nm	Rotation	= -0.888°	Op. mode	= Dynamic Force
Sy	= 2181.3nm	X-Pos	= 2.2µm	Cantilever type	= ACLA
Sp	= 949.36nm	Y-Pos	= -4.2µm	Head type	= EZ2-AFM
Sv	= -1231.9nm	Z-Plane	= 0µm	Scan head	= 10-07-239.hed
Sm	= 211.36µm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 204.63nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 249.16nm	Time	= 17:21:50	-- Module --	
Ry	= 1012.7nm	-- Feedback --		Controller Board	= 2
Rp	= 540.26nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -472.47nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 262.74µm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.347kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image2

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.23 V
Area	= 618pm^2	X-Slope	= 3.5 °	Error range	= 20 V
Sa	= 159.38nm	Y-Slope	= 500m°	-- Global --	
Sq	= 194.87nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 1171.3nm	X-Pos	= 2.2μm	Cantilever type	= ACLA
Sp	= 762.76nm	Y-Pos	= -4.2μm	Head type	= EZ2-AFM
Sv	= -408.53nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 2704.2fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 173.83nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 201.25nm	Time	= 17:31:33	-- Module --	
Ry	= 717.95nm	-- Feedback --		Controller Board	= 2
Rp	= 391.72nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -326.23nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -59.301pm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 181.327kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image3

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.23 V
Area	= 618pm^2	X-Slope	= 3 °	Error range	= 20 V
Sa	= 141.54nm	Y-Slope	= 3.5 °	-- Global --	
Sq	= 177.74nm	Rotation	= -0.888f°	Op. mode	= Dynamic Force
Sy	= 1261.3nm	X-Pos	= 2.2μm	Cantilever type	= ACLA
Sp	= 760.65nm	Y-Pos	= -4.2μm	Head type	= EZ2-AFM
Sv	= -500.67nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 118.52pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 98.04nm	Date	= 08-08-2008	Controller S/N	= 023-06-252
Rq	= 120.88nm	Time	= 17:43:11	-- Module --	
Ry	= 515.74nm	-- Feedback --		Controller Board	= 2
Rp	= 282.35nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -233.39nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 82.366pm	I-Gain	= 1600	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 181.327kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-675-16 after heat treatment

Nanosurf Image Document

File: Image5

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.27 V
Area	= 618pm ²	X-Slope	= 0 °	Error range	= 20 V
Sa	= 113.27nm	Y-Slope	= -500m°	-- Global --	
Sq	= 141.74nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 1119.2nm	X-Pos	= 18μm	Cantilever type	= ACLA
Sp	= 594.29nm	Y-Pos	= 72nm	Head type	= EZ2-AFM
Sv	= -524.92nm	Z-Plane	= 0nm	Scan head	= 10-07-239.hed
Sm	= 211.65pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 127.53nm	Date	= 31-07-2008	Controller S/N	= 023-06-252
Rq	= 148.92nm	Time	= 17:25:40	-- Module --	
Ry	= 703.4nm	-- Feedback --		Controller Board	= 2
Rp	= 289.62nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -413.78nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 197.68pm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image6

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 618pm^2	X-Slope	= -1 °	Error range	= 20 V
Sa	= 116.65nm	Y-Slope	= -500m°	-- Global --	
Sq	= 146.12nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 918.26nm	X-Pos	= 18µm	Cantilever type	= ACLA
Sp	= 453.65nm	Y-Pos	= 72nm	Head type	= EZ2-AFM
Sv	= -464.61nm	Z-Plane	= 0nm	Scan head	= 10-07-239.hed
Sm	= 212.33pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 119.14nm	Date	= 31-07-2008	Controller S/N	= 023-06-252
Rq	= 151.82nm	Time	= 17:37:07	-- Module --	
Ry	= 682.11nm	-- Feedback --		Controller Board	= 2
Rp	= 340.27nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -341.84nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 154.02pm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image7

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.26 V
Area	= 618pm ²	X-Slope	= 0 °	Error range	= 20 V
Sa	= 143.49nm	Y-Slope	= -1 °	-- Global --	
Sq	= 174.71nm	Rotation	= -16°	Op. mode	= Dynamic Force
Sy	= 1026.9nm	X-Pos	= 16μm	Cantilever type	= ACLA
Sp	= 476.42nm	Y-Pos	= -25nm	Head type	= EZ2-AFM
Sv	= -550.43nm	Z-Plane	= 0nm	Scan head	= 10-07-239.hed
Sm	= 2215.1fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 109.22nm	Date	= 31-07-2008	Controller S/N	= 023-06-252
Rq	= 134.29nm	Time	= 17:46:43	-- Module --	
Ry	= 567.19nm	-- Feedback --		Controller Board	= 2
Rp	= 231.53nm	Set point	= 50 %	AFM Basic Module	= 2
Rv	= -335.66nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 91.426pm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 181.337kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

N-675-16 after CP

Nanosurf Image Document

File: Image7

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.16 V
Area	= 629.9pm ²	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 119.36nm	Y-Slope	= 1 °	-- Global --	
Sq	= 153.39nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 1374.5nm	X-Pos	= 6.3μm	Cantilever type	= ACLA
Sp	= 858.7nm	Y-Pos	= 19μm	Head type	= EZ2-AFM
Sv	= -515.85nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= -4.6796pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 137.69nm	Date	= 10-08-2008	Controller S/N	= 023-06-252
Rq	= 165.93nm	Time	= 15:54:23	-- Module --	
Ry	= 686.96nm	-- Feedback --		Controller Board	= 2
Rp	= 365.93nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -321.03nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -14.541nm	I-Gain	= 1100	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.237kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image8

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.17 V
Area	= 629.9pm^2	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 166.75nm	Y-Slope	= 1 °	-- Global --	
Sq	= 217.84nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 2015.5nm	X-Pos	= 1µm	Cantilever type	= ACLA
Sp	= 1245.4nm	Y-Pos	= 19µm	Head type	= EZ2-AFM
Sv	= -770.14nm	Z-Plane	= 0.62µm	Scan head	= 10-07-239.hed
Sm	= 4.8244pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 140.19nm	Date	= 10-08-2008	Controller S/N	= 023-06-252
Rq	= 179.7nm	Time	= 16:12:32	-- Module --	
Ry	= 907.3nm	-- Feedback --		Controller Board	= 2
Rp	= 411.3nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -496nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -31.297pm	I-Gain	= 1200	AFM Extension Modules	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.257kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image9

Parameter:

-- Area Roughness --	Lines	= 256	Excitation ampl.	= 0.15 V	
Area	= 629.9µm²	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 116.54nm	Y-Slope	= 1 °	-- Global --	
Sq	= 150.77nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 1358.5nm	X-Pos	= 1µm	Cantilever type	= ACLA
Sp	= 794.39nm	Y-Pos	= 19µm	Head type	= EZ2-AFM
Sv	= -564.14nm	Z-Plane	= 0µm	Scan head	= 10-07-239.hed
Sm	= -3138.5fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --	Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2	
Ra	= 134.33nm	Date	= 10-08-2008	Controller S/N	= 023-06-252
Rq	= 186.81nm	Time	= 16:21:25	-- Module --	
Ry	= 847.42nm	-- Feedback --		Controller Board	= 2
Rp	= 394.61nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -452.81nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 2472.5fm	I-Gain	= 1200	AFM Extension Module	= 1
-- Scan --	Tip voltage	= 0 V	Video Module	= 0	
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.257kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

APPENDIX B: AFM Data of Abraded Samples

A-0

Nanosurf Image Document

File: Image3

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.19 V
Area	= 618pm ²	X-Slope	= -250m°	Error range	= 20 V
Sa	= 4.0598nm	Y-Slope	= 500m°	-- Global --	
Sq	= 6.555nm	Rotation	= -1.78°	Op. mode	= Dynamic Force
Sy	= 219.71nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 173.43nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -46.282nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 206.62pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 3156.1pm	Date	= 05-09-2008	Controller S/N	= 023-06-252
Rq	= 3.9659nm	Time	= 12:05:19	-- Module --	
Ry	= 23.405nm	-- Feedback --		Controller Board	= 2
Rp	= 8.4942nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -14.91nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 256.15pm	I-Gain	= 1600	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 179.074kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image7

Parameter:

-- Area Roughness --		Time/Line	= 0.3 s	Vibration ampl.	= 0.7 V
Area	= 153.3 μm^2	Points	= 256	Excitation ampl.	= 0.2 V
Sa	= 10.257nm	Lines	= 256	Error range	= 20 V
Sq	= 12.639nm	X-Slope	= -250m°	-- Global --	
Sy	= 138.53nm	Y-Slope	= 750m°	Op. mode	= Dynamic Force
Sp	= 122.4nm	Rotation	= 90 °	Cantilever type	= ACLA
Sv	= -16.13nm	X-Pos	= 0 m	Head type	= EZ2-AFM
Sm	= 9.5636nm	Y-Pos	= 0 m	Scan head	= 10-07-239.hed
-- Tool --		Z-Plane	= 0 m	Software ver.	= 1-5-1-0
Width	= 12.28 μm	Overscan	= 5 %	Firmware ver.	= 2-1-1-2
Height	= 12.28 μm	Const.Height-Mode	= Disabled	Controller S/N	= 023-06-252
-- Line Roughness --		Date	= 05-09-2008	-- Module --	
Ra	= 5.7415nm	Time	= 12:23:39	Controller Board	= 2
Rq	= 7.351nm	-- Feedback --		AFM Basic Module	= 2
Ry	= 43.225nm	Set point	= 48 %	AFM Dynamic Module	= 2
Rp	= 30.465nm	P-Gain	= 10000	AFM Extension Module	= 1
Rv	= -12.759nm	I-Gain	= 1600	Video Module	= 0
Rm	= 13.497nm	Tip voltage	= 0 V	Signal Module S	= 0
-- Scan --		Feedback mode	= Free	Signal Module A	= 0
Image size	= 25 μm	Feedback algo.	= Adaptive PI	Nanosurf Report	= 0
Scan direction	= Down	Vibration freq.	= 179.044kHz	Scripting Interface	= 0

Nanosurf Image Document

File: Image10

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.19 V
Area	= 155.7pm ²	X-Slope	= -250m°	Error range	= 20 V
Sa	= 5.1719nm	Y-Slope	= 500m°	-- Global --	
Sq	= 7.2843nm	Rotation	= 0m°	Op. mode	= Dynamic Force
Sy	= 149.49nm	X-Pos	= 0mm	Cantilever type	= ACLA
Sp	= 130.62nm	Y-Pos	= 0mm	Head type	= EZ2-AFM
Sv	= -18.871nm	Z-Plane	= 0mm	Scan head	= 10-07-239.hed
Sm	= 4.1017nm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 4.2242nm	Date	= 05-09-2008	Controller S/N	= 023-06-252
Rq	= 5.5436nm	Time	= 12:38:21	-- Module --	
Ry	= 39.64nm	-- Feedback --		Controller Board	= 2
Rp	= 21.89nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -17.75nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 5.4156nm	I-Gain	= 1600	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 179.044kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

A-600-1 after heat treatment

Nanosurf Image Document

File: Image16

Parameter:

-- Tool --		Time/Line	= 0.6 s	Vibration ampl.	= 0.7 V
Length	= 24.81µm	Points	= 256	Excitation ampl.	= 0.23 V
DeltaZ	= 57.35nm	Lines	= 256	Error range	= 20 V
-- Area Roughness --		X-Slope	= -250m°	-- Global --	
Area	= 629.9µm²	Y-Slope	= -2.5°	Op. mode	= Dynamic Force
Sa	= 27.711nm	Rotation	= 90°	Cantilever type	= ACLA
Sq	= 34.497nm	X-Pos	= 11µm	Head type	= EZ2-AFM
Sy	= 297.3nm	Y-Pos	= 0.46µm	Scan head	= 10-07-239.hed
Sp	= 198.94nm	Z-Plane	= 0µm	Software ver.	= 1-5-1-0
Sv	= -98.363nm	Overscan	= 5 %	Firmware ver.	= 2-1-1-2
Sm	= -191.43fm	Const.Height-Mode	= Disabled	Controller S/N	= 023-06-252
-- Line Roughness --		Date	= 09-09-2008	-- Module --	
Ra	= 35.492nm	Time	= 13:39:38	Controller Board	= 2
Rq	= 42.495nm	-- Feedback --		AFM Basic Module	= 2
Ry	= 178.59nm	Set point	= 48 %	AFM Dynamic Module	= 2
Rp	= 111.78nm	P-Gain	= 10000	AFM Extension Module	= 1
Rv	= -66.811nm	I-Gain	= 1900	Video Module	= 0
Rm	= 76.6µm	Tip voltage	= 0 V	Signal Module S	= 0
-- Scan --		Feedback mode	= Free	Signal Module A	= 0
Image size	= 25µm	Feedback algo.	= Adaptive PI	Nanosurf Report	= 0

Nanosurf Image Document

File: Image17

Parameter:

-- Area Roughness --	Lines	= 256	Excitation ampl.	= 0.23 V	
Area	= 629.9pm^2	X-Slope	= -1.25 °	Error range	= 20 V
Sa	= 25.009nm	Y-Slope	= -1.75 °	-- Global --	
Sq	= 34.785nm	Rotation	= 0 °	Op. mode	= Dynamic Force
Sy	= 377nm	X-Pos	= 11μm	Cantilever type	= ACLA
Sp	= 213.28nm	Y-Pos	= 0.46μm	Head type	= EZ2-AFM
Sv	= -163.73nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 215.35pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --	Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2	
Ra	= 20.37nm	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	= 26.298nm	Time	= 13:49:13	-- Module --	
Ry	= 154.34nm	-- Feedback --		Controller Board	= 2
Rp	= 101.7nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -52.64nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 32.344pm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --	Tip voltage	= 0 V	Video Module	= 0	
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.6 s	Vibration freq.	= 179.054kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image18

Parameter:

-- Area Roughness --	Lines	= 256	Excitation ampl.	= 0.23 V
Area	X-Slope	= 250m°	Error range	= 20 V
Sa	Y-Slope	= -2.25 °	-- Global --	
Sq	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	X-Pos	= 11μm	Cantilever type	= ACLA
Sp	Y-Pos	= 0.46μm	Head type	= EZ2-AFM
Sv	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --	Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	Time	= 13:59:45	-- Module --	
Ry	-- Feedback --		Controller Board	= 2
Rp	Set point	= 48 %	AFM Basic Module	= 2
Rv	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	I-Gain	= 1800	AFM Extension Module	= 1
-- Scan --	Tip voltage	= 0 V	Video Module	= 0
Image size	Feedback mode	= Free	Signal Module S	= 0
Scan direction	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	Vibration freq.	= 179.064kHz	Nanosurf Report	= 0
Points	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

A-600-4 after heat treatment

Nanosurf Image Document

File: Image2

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 618pm ²	X-Slope	= 500m°	Error range	= 20 V
Sa	= 89.336nm	Y-Slope	= 5.25 °	-- Global --	
Sq	= 109.84nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 725.96nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 393.56nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -332.41nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 6.7227pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 83.344nm	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	= 105.53nm	Time	= 16:15:08	-- Module --	
Ry	= 500.35nm	-- Feedback --		Controller Board	= 2
Rp	= 278.22nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -222.13nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 1.6087fm	I-Gain	= 1300	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.094kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image3

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 618µm²	X-Slope	= 500m°	Error range	= 20 V
Sa	= 73.841nm	Y-Slope	= 4.75 °	-- Global --	
Sq	= 91.994nm	Rotation	= 0 °	Op. mode	= Dynamic Force
Sy	= 714.58nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 444.79nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -269.79nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 210.94µm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 75.381nm	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	= 99.454nm	Time	= 16:25:07	-- Module --	
Ry	= 485.8nm	-- Feedback --		Controller Board	= 2
Rp	= 311.96nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -173.85nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 210.03µm	I-Gain	= 1300	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.094kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image4

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.23 V
Area	= 618 μm^2	X-Slope	= -1.5 °	Error range	= 20 V
Sa	= 82.47 nm	Y-Slope	= 4.25 °	-- Global --	
Sq	= 102.38 nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 706.14 nm	X-Pos	= 0 m	Cantilever type	= ACLA
Sp	= 383.44 nm	Y-Pos	= 0 m	Head type	= EZ2-AFM
Sv	= -322.71 nm	Z-Plane	= 0 m	Scan head	= 10-07-239.hed
Sm	= 4.2582 μm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 110.37 nm	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	= 127.99 nm	Time	= 16:35:34	-- Module --	
Ry	= 491.08 nm	-- Feedback --		Controller Board	= 2
Rp	= 310.27 nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -180.8 nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -2469.3 fm	I-Gain	= 1300	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.074 kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

A-600-8 after heat treatment

Nanosurf Image Document

File: Image12

Parameter:

-- Area Roughness --	Lines	= 256	Excitation ampl.	= 0.19 V	
Area	= 618pm^2	X-Slope	= 500m°	Error range	= 20 V
Sa	= 42.105nm	Y-Slope	= -1 °	-- Global --	
Sq	= 52.867nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 399.78nm	X-Pos	= -0.19µm	Cantilever type	= ACLA
Sp	= 217.49nm	Y-Pos	= 13µm	Head type	= EZ2-AFM
Sv	= -182.28nm	Z-Plane	= -0.23µm	Scan head	= 10-07-239.hed
Sm	= -6.6937pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --	Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2	
Ra	= 46.235nm	Date	= 05-09-2008	Controller S/N	= 023-06-252
Rq	= 57.025nm	Time	= 12:52:11	-- Module --	
Ry	= 286.34nm	-- Feedback --		Controller Board	= 2
Rp	= 140.52nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -145.82nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 10.709pm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --	Tip voltage	= 0 V	Video Module	= 0	
Image size	= 25µm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 179.044kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image13

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.23 V
Area	= 618pm ²	X-Slope	= 500m°	Error range	= 20 V
Sa	= 28.166nm	Y-Slope	= -1 °	-- Global --	
Sq	= 38.112nm	Rotation	= -0.888°	Op. mode	= Dynamic Force
Sy	= 358.45nm	X-Pos	= -0.19μm	Cantilever type	= ACLA
Sp	= 253.34nm	Y-Pos	= 13μm	Head type	= EZ2-AFM
Sv	= -105.11nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 148.82pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 34.104nm	Date	= 05-09-2008	Controller S/N	= 023-06-252
Rq	= 44.996nm	Time	= 13:04:20	-- Module --	
Ry	= 251.55nm	-- Feedback --		Controller Board	= 2
Rp	= 163.82nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -87.723nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 113.66pm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 179.094kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image14

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.19 V
Area	= 618pm ²	X-Slope	= 0 °	Error range	= 20 V
Sa	= 37.571nm	Y-Slope	= 0.389°	-- Global --	
Sq	= 47.713nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 493.82nm	X-Pos	= -0.19μm	Cantilever type	= ACLA
Sp	= 352.86nm	Y-Pos	= 13μm	Head type	= EZ2-AFM
Sv	= -140.95nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 215.66pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 23.075nm	Date	= 05-09-2008	Controller S/N	= 023-06-252
Rq	= 36.593nm	Time	= 13:11:24	-- Module --	
Ry	= 257.03nm	-- Feedback --		Controller Board	= 2
Rp	= 201.1nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -55.928nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 42.96nm	I-Gain	= 1800	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.3 s	Vibration freq.	= 179.054kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

A-600-16 after heat treatment

Nanosurf Image Document

File: Image15

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.19 V
Area	= 618pm ²	X-Slope	= 3 °	Error range	= 20 V
Sa	= 74.267nm	Y-Slope	= 6 °	-- Global --	
Sq	= 94.2nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 565.93nm	X-Pos	= -0.19μm	Cantilever type	= ACLA
Sp	= 371.84nm	Y-Pos	= 13μm	Head type	= EZ2-AFM
Sv	= -194.09nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 4.892pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 46.303nm	Date	= 05-09-2008	Controller S/N	= 023-06-252
Rq	= 59.784nm	Time	= 13:21:09	-- Module --	
Ry	= 281.7nm	-- Feedback --		Controller Board	= 2
Rp	= 162.17nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -119.53nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 85.66pm	I-Gain	= 1700	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.034kHz	Nanosurf Report	= 0

Nanosurf Image Document

File: Image16

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.19 V
Area	= 618pm ²	X-Slope	= 6 °	Error range	= 20 V
Sa	= 68.638nm	Y-Slope	= 4.25 °	-- Global --	
Sq	= 87.758nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 636.56nm	X-Pos	= -0.19 μm	Cantilever type	= ACLA
Sp	= 417.59nm	Y-Pos	= 13 μm	Head type	= EZ2-AFM
Sv	= -218.97nm	Z-Plane	= 0 μm	Scan head	= 10-07-239.hed
Sm	= 92.192pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 68.968nm	Date	= 05-09-2008	Controller S/N	= 023-06-252
Rq	= 84.162nm	Time	= 13:29:37	-- Module --	
Ry	= 369.62nm	-- Feedback --		Controller Board	= 2
Rp	= 217.67nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -151.95nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 32.947pm	I-Gain	= 1600	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.024kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image17

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.21 V
Area	= 618 μm^2	X-Slope	= 4.5 °	Error range	= 20 V
Sa	= 88.541nm	Y-Slope	= 6 °	-- Global --	
Sq	= 108.15nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 676.84nm	X-Pos	= -0.19 μm	Cantilever type	= ACLA
Sp	= 400.09nm	Y-Pos	= 13 μm	Head type	= EZ2-AFM
Sv	= -276.74nm	Z-Plane	= 0 μm	Scan head	= 10-07-239.hed
Sm	= 52.493pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 70.085nm	Date	= 05-09-2008	Controller S/N	= 023-06-252
Rq	= 81.274nm	Time	= 13:42:09	-- Module --	
Ry	= 272.63nm	-- Feedback --		Controller Board	= 2
Rp	= 119.09nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -153.54nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -68.361pm	I-Gain	= 1600	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.7 s	Vibration freq.	= 179.074kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

A-675-1 after heat treatment

Nanosurf Image Document

File: Image9

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.24 V
Area	= 629.9pm ²	X-Slope	= 350m°	Error range	= 20 V
Sa	= 51.993nm	Y-Slope	= 750m°	-- Global --	
Sq	= 71.199nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 879.89nm	X-Pos	= 11μm	Cantilever type	= ACLA
Sp	= 731.97nm	Y-Pos	= 0.46μm	Head type	= EZ2-AFM
Sv	= -147.91nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 1282.1fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 35.197nm	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	= 43.888nm	Time	= 12:28:58	-- Module --	
Ry	= 220.13nm	-- Feedback --		Controller Board	= 2
Rp	= 135.03nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -85.104nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 30.81nm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.074kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image8

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.29 V
Area	= 629.9µm²	X-Slope	= 350m°	Error range	= 20 V
Sa	= 32.942nm	Y-Slope	= 750m°	-- Global --	
Sq	= 44.709nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 371.31nm	X-Pos	= 11µm	Cantilever type	= ACLA
Sp	= 259.03nm	Y-Pos	= 0.46µm	Head type	= EZ2-AFM
Sv	= -112.28nm	Z-Plane	= 0µm	Scan head	= 10-07-239.hed
Sm	= -136.74fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 28.156nm	Date	= 09.09.2008	Controller S/N	= 023.06.252

Nanosurf Image Document

File: Image10

Parameter:

-- Area Roughness --		Lines = 256	Excitation ampl. = 0.25 V
Area = 629.9 μm ²	X-Slope = 350 m°	Error range = 20 V	
Sa = 36.056 nm	Y-Slope = 750 m°	-- Global --	
Sq = 51.933 nm	Rotation = -14.2 f°	Op. mode = Dynamic Force	
Sy = 1098.8 nm	X-Pos = 11 μm	Cantilever type = ACLA	
Sp = 907.61 nm	Y-Pos = 0.46 μm	Head type = EZ2-AFM	
Sv = -191.14 nm	Z-Plane = 0 μm	Scan head = 10-07-239.hed	
Sm = 31.799 pm	Overscan = 5 %	Software ver. = 1-5-1-0	
-- Line Roughness --		Const.Height-Mode = Disabled	Firmware ver. = 2-1-1-2
Ra = 39.772 nm	Date = 09-09-2008	Controller S/N = 023-06-252	
Rq = 51.093 nm	Time = 12:38:15	-- Module --	
Ry = 325.98 nm	-- Feedback --	Controller Board = 2	
Rp = 239 nm	Set point = 48 %	AFM Basic Module = 2	
Rv = -86.976 nm	P-Gain = 10000	AFM Dynamic Module = 2	
Rm = -822.03 fm	I-Gain = 1900	AFM Extension Module = 1	
-- Scan --		Video Module = 0	
Image size = 25 μm	Tip voltage = 0 V	Signal Module S = 0	
Scan direction = Up	Feedback mode = Free	Signal Module A = 0	
Time/Line = 0.6 s	Feedback algo. = Adaptive PI	Nanosurf Report = 0	
Points = 256	Vibration freq. = 179.084 kHz	Scripting Interface = 0	
	Vibration ampl. = 0.7 V		

A-675-4 after heat treatment

Nanosurf Image Document

File: Image11

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 629.9pm ²	X-Slope	= -1.25 °	Error range	= 20 V
Sa	= 111.75nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 142.88nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 940.82nm	X-Pos	= 11μm	Cantilever type	= ACLA
Sp	= 647nm	Y-Pos	= 0.46μm	Head type	= EZ2-AFM
Sv	= -293.82nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 214.63pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 105.3nm	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	= 148.5nm	Time	= 12:48:21	-- Module --	
Ry	= 673.67nm	-- Feedback --		Controller Board	= 2
Rp	= 483.4nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -190.27nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 190.26pm	I-Gain	= 1800	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.6 s	Vibration freq.	= 179.084kHz	Nanosurf Report	= 0

Nanosurf Image Document

File: Image13

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.26 V
Area	= 629.9 μm^2	X-Slope	= -1.75 °	Error range	= 20 V
Sa	= 209nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 249.99nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 1.457 μm	X-Pos	= 11 μm	Cantilever type	= ACLA
Sp	= 828.54nm	Y-Pos	= 0.46 μm	Head type	= EZ2-AFM
Sv	= -628.45nm	Z-Plane	= 0 μm	Scan head	= 10-07-239.hed
Sm	= 500.3fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 90.752nm	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	= 113.23nm	Time	= 13:05:51	-- Module --	
Ry	= 532.4nm	-- Feedback --		Controller Board	= 2
Rp	= 273.4nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -259.01nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 259.11nm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.6 s	Vibration freq.	= 179.094kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image15

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 629.9 μm^2	X-Slope	= -1.75 °	Error range	= 20 V
Sa	= 135.73nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 166.95nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 1206.3nm	X-Pos	= 11 μm	Cantilever type	= ACLA
Sp	= 757.7nm	Y-Pos	= 0.46 μm	Head type	= EZ2-AFM
Sv	= -448.59nm	Z-Plane	= 0 μm	Scan head	= 10-07-239.hed
Sm	= 65.204pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 130.66nm	Date	= 09-09-2008	Controller S/N	= 023-06-252
Rq	= 163.91nm	Time	= 13:22:46	-- Module --	
Ry	= 649.21nm	-- Feedback --		Controller Board	= 2
Rp	= 387.74nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -261.47nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= -88.952pm	I-Gain	= 1900	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Down	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.6 s	Vibration freq.	= 179.074kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

A-675-8 after heat treatment

Nanosurf Image Document

File: Image18

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.18 V
Area	= 618pm ²	X-Slope	= 2 °	Error range	= 20 V
Sa	= 267.45nm	Y-Slope	= 2 °	-- Global --	
Sq	= 336.37nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 2174.1nm	X-Pos	= -12μm	Cantilever type	= ACLA
Sp	= 1208.5nm	Y-Pos	= -8.7μm	Head type	= EZ2-AFM
Sv	= -965.6nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 66.861pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 249.53nm	Date	= 04-09-2008	Controller S/N	= 023-06-252
Rq	= 308.62nm	Time	= 18:01:05	-- Module --	
Ry	= 1158.8nm	-- Feedback --		Controller Board	= 2
Rp	= 601.64nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -557.21nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 28.829pm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.6 s	Vibration freq.	= 179.034kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image19

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.25 V
Area	= 618 μm^2	X-Slope	= 1 °	Error range	= 20 V
Sa	= 332.85 nm	Y-Slope	= 1 °	-- Global --	
Sq	= 406.47 nm	Rotation	= -14.2 °	Op. mode	= Dynamic Force
Sy	= 2309.7 nm	X-Pos	= -12 μm	Cantilever type	= ACLA
Sp	= 1345.6 nm	Y-Pos	= -8.7 μm	Head type	= EZ2-AFM
Sv	= -964.12 nm	Z-Plane	= 0 μm	Scan head	= 10-07-239.hed
Sm	= 209.53 μm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 311.47 nm	Date	= 04-09-2008	Controller S/N	= 023-06-252
Rq	= 404.73 nm	Time	= 18:11:06	-- Module --	
Ry	= 1655.4 nm	-- Feedback --		Controller Board	= 2
Rp	= 833.68 nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -821.72 nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 135.9 μm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25 μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.6 s	Vibration freq.	= 179.124 kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image20

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.2 V
Area	= 618pm^2	X-Slope	= -1 °	Error range	= 20 V
Sa	= 197.92nm	Y-Slope	= 1 °	-- Global --	
Sq	= 263.57nm	Rotation	= 90 °	Op. mode	= Dynamic Force
Sy	= 1.876μm	X-Pos	= -12μm	Cantilever type	= ACLA
Sp	= 1211.9nm	Y-Pos	= -8.7μm	Head type	= EZ2-AFM
Sv	= -664.08nm	Z-Plane	= 0μm	Scan head	= 10-07-239.hed
Sm	= 110.48pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 305.07nm	Date	= 04-09-2008	Controller S/N	= 023-06-252
Rq	= 399.55nm	Time	= 18:22:29	-- Module --	
Ry	= 1709.4nm	-- Feedback --		Controller Board	= 2
Rp	= 1084.2nm	Set point	= 48 %	AFM Basic Module	= 2
Rv	= -625.22nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 145.79pm	I-Gain	= 1500	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.7 s	Vibration freq.	= 179.054kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

A-675-16 after heat treatment

Nanosurf Image Document

File: Image5

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.18 V
Area	= 618pm ²	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 141.63nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 176.87nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 1105.3nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 621.28nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -484.01nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= -107.78fm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 161.94nm	Date	= 22-08-2008	Controller S/N	= 023-06-252
Rq	= 192.79nm	Time	= 19:23:50	-- Module --	
Ry	= 800.61nm	-- Feedback --		Controller Board	= 2
Rp	= 480.43nm	Set point	= 46 %	AFM Basic Module	= 2
Rv	= -320.18nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 1.6087fm	I-Gain	= 1400	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.4 s	Vibration freq.	= 179.074kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

Nanosurf Image Document

File: Image6

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.17 V
Area	= 618pm ²	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 146.1nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 179.96nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 1127.2nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 646.37nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -480.85nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 164.41pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 150.83nm	Date	= 22-08-2008	Controller S/N	= 023-06-252

Nanosurf Image Document

File: Image7

Parameter:

-- Area Roughness --		Lines	= 256	Excitation ampl.	= 0.18 V
Area	= 618pm^2	X-Slope	= 1.5 °	Error range	= 20 V
Sa	= 183.66nm	Y-Slope	= 2.5 °	-- Global --	
Sq	= 227.7nm	Rotation	= -14.2f°	Op. mode	= Dynamic Force
Sy	= 1595.9nm	X-Pos	= 0fm	Cantilever type	= ACLA
Sp	= 780.47nm	Y-Pos	= 0fm	Head type	= EZ2-AFM
Sv	= -815.47nm	Z-Plane	= 0fm	Scan head	= 10-07-239.hed
Sm	= 26.789pm	Overscan	= 5 %	Software ver.	= 1-5-1-0
-- Line Roughness --		Const.Height-Mode	= Disabled	Firmware ver.	= 2-1-1-2
Ra	= 151.36nm	Date	= 22-08-2008	Controller S/N	= 023-06-252
Rq	= 175.7nm	Time	= 19:39:12	-- Module --	
Ry	= 736.93nm	-- Feedback --		Controller Board	= 2
Rp	= 393.13nm	Set point	= 46 %	AFM Basic Module	= 2
Rv	= -343.8nm	P-Gain	= 10000	AFM Dynamic Module	= 2
Rm	= 1648.9fm	I-Gain	= 1400	AFM Extension Module	= 1
-- Scan --		Tip voltage	= 0 V	Video Module	= 0
Image size	= 25μm	Feedback mode	= Free	Signal Module S	= 0
Scan direction	= Up	Feedback algo.	= Adaptive PI	Signal Module A	= 0
Time/Line	= 0.5 s	Vibration freq.	= 179.044kHz	Nanosurf Report	= 0
Points	= 256	Vibration ampl.	= 0.7 V	Scripting Interface	= 0

APPENDIX C: Potentiodynamic Polarization Data for Non-abraded Samples

OC, N-0

080717_ti64_0_0_OC, E vs t

PowerSuite -- 2.58

Page #1

CP, N-0

080717_ti64_0_0_CP, E vs log(I)

PowerSuite -- 2.58

Page #1

OC, N-600-1

080606_ti64_2_OC, E vs t

PowerSuite -- 2.58

Page #1

OC, N-600-8

080605_ti64_1_OC, E vs t

PowerSuite -- 2.58

Page #1

APPENDIX D: Minitab Calculations for HRc Data

One-way ANOVA: HRc RT, HRc 600, HRc 675

Source	DF	SS	MS	F	P
Factor	2	13.78	6.89	3.11	0.057
Error	36	79.88	2.22		
Total	38	93.66			

S = 1.490 R-Sq = 14.72% R-Sq(adj) = 9.98%

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
HRc RT	7	34.329	2.216	(-----*-----)
HRc 600	16	34.281	1.354	(-----*-----)
HRc 675	16	33.087	1.236	(-----*-----)

-----+-----+-----+-----+-----
32.80 33.60 34.40 35.20

Pooled StDev = 1.490

One-way ANOVA: HRc 675 versus Time(675)

Source	DF	SS	MS	F	P
Time(675)	3	7.597	2.532	9.09	0.002
Error	12	3.342	0.279		
Total	15	10.939			

S = 0.5278 R-Sq = 69.45% R-Sq(adj) = 61.81%

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
1	4	33.400	0.668	(-----*-----)
4	4	34.075	0.377	(-----*-----)
8	4	33.675	0.189	(-----*-----)
16	4	32.225	0.699	(-----*-----)

-----+-----+-----+-----+-----
32.00 32.80 33.60 34.40

Pooled StDev = 0.528

One-way ANOVA: HRc 600 versus Time

Source	DF	SS	MS	F	P
Time	3	2.365	0.788	1.62	0.238
Error	12	5.855	0.488		
Total	15	8.220			

S = 0.6985 R-Sq = 28.77% R-Sq(adj) = 10.96%

Pooled StDev = 0.699

One-way ANOVA: HRc 600, C6

Source	DF	SS	MS	F	P
Factor	1	0.36	0.36	0.20	0.659
Error	21	37.69	1.79		
Total	22	38.05			

S = 1.340 R-Sq = 0.94% R-Sq(adj) = 0.00%

Pooled StDev = 1.340

One-way ANOVA: HRc 675, C6

Source	DF	SS	MS	F	P
Factor	1	4.72	4.72	2.45	0.132
Error	21	40.41	1.92		
Total	22	45.14			

S = 1.387 R-Sq = 10.46% R-Sq(adj) = 6.20%

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
HRc 675	16	33.344	0.854	-----+-----+-----+----- (-----*-----)
C6	7	34.329	2.216	(-----*-----) -----+-----+-----+----- 32.90 33.60 34.30 35.00

Pooled StDev = 1.387

One-way ANOVA: HRc 675, HRc 600

Source	DF	SS	MS	F	P
Factor	1	12.625	12.625	19.77	0.000
Error	30	19.159	0.639		
Total	31	31.785			

S = 0.7992 R-Sq = 39.72% R-Sq(adj) = 37.71%

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
HRc 675	16	33.344	0.854	-----+-----+-----+----- (-----*-----)
HRc 600	16	34.600	0.740	(-----*-----) -----+-----+-----+----- 33.00 33.60 34.20 34.80

Pooled StDev = 0.799