The Effect of Autoclave Exposure on SilcoTek surfaces

Technical Insight

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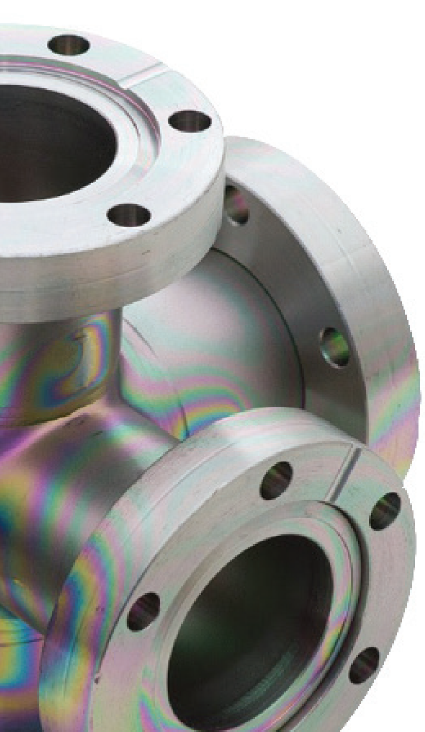


Synopsis

There is a general lack of understanding on the effect of steam and/or autoclave exposure to the surface and bulk properties of SilcoTek surfaces. This study will report on these effects via measurement of surface contact angles, FT-IR spectroscopy, and corrosion test comparisons.

Background

An autoclave is often used to sterilize surfaces via an exposure to saturated steam over a period of time. Understanding the effect of steam exposure on SilcoTek surfaces is important since many customers and potential customers may use steam for cleaning and sterilization purposes. This TI is a summary of findings on the effect of autoclave exposure on various surfaces offered by Silcotek, using contact angles, thickness, and FT-IR spectroscopy as characterization methods. 316 QC coupons were used for these characterization methods. This will be a living document, as the before/after exposure coupons will be archived, and future comparative analyses will be possible. For example, SilcoTek 316 corrosion coupons with SilcoTek corrosive-resistant coatings were also exposed to autoclave conditions and will be evaluated via ASTM G-31 hydrochloric acid corrosion exposure protocol and EIS spectroscopic analysis.

Experimental

The coatings/treatments evaluated for autoclave exposure were: Notak, RD5-SiN, Silcolloy 1000, Dursox, Dursan, SilcoNert 2000 and SilcoKlean 1000. 6 Coupons of each treatment were prepared per SilcoTek SOPs (or per the latest optimized processes per R&D development for Notak and RD5-SiN) on SilcoTek 316L QC coupons for a total of 42 QC coupons.

Six (6) 316L corrosion coupons were also coated with each of the following: Dursox, Dursan, Silcolloy and RD5-SiN for a total of 24 corrosion coupons. After treatment, the following measurements were made on all 316 QC coupons: DI water contact angle, hexadecane contact angle (for Notak coupons only), thickness (via Filmetrics F40 spectrometer) at the general center of the coupon, and FT-IR spectroscopy. ASTM G-31 and EIS corrosion characterization will be conducted and evaluated at a later date.

Afterward, half of the coupons, three from each process (QC and corrosion coupons) for a total of 33, were sent to Namsa in Northwood, Ohio for autoclave exposure. All coupons were treated simultaneously in one chamber with the following conditions of exposure:

- Cycle Type: Gravity
- Exposure Pressure Range: 16.4-17.8 psig
- Conditioning Time: 10 minutes, 7 seconds
- Exposure Temperature Range: 121.0-123.2°C
- Exposure Time: 60 minutes
- Total Cycle Time: 1 hour 10 minutes 40 seconds

The treated coupons were returned to SilcoTek and QC coupons were recharacterized via the same measurements prior to autoclave exposure.

Data and Discussion

Contact angle evaluation:

Table 1: Average contact angle differences, before and after autoclave exposure, with deionized water and hexadecane (Notak only).

Surface	DI Water Before	DI Water After	Decrease	Hxdcn Before	Hxdcn After
Notak	120°	120°	0°	83°	82°
Silcolloy	70°	29°	41°		
Dursox	51°	40°	11°		
Dursan	88°	78°	10°		
SN2000	82°	70°	12°		
SK1000	84°	75°	9°		
RD5-SiN	25°	22°	3°		

Table 1 illustrates changes in contact angles of seven different SilcoTek surfaces after exposure to standard autoclave conditions. Silcolloy, which is an unfunctionalized amorphous silicon deposition had the most dramatic change, with a water contact angle before exposure of 70° to a post exposure contact angle of 29°. The degree of change is not surprising, since Silcolloy has a silicon hydrides at the surface that can be surface oxidized to create Si-O-Si hydrophilic moieties, as confirmed in the FT-IR data discussed below (Silcolloy is, however, very stable against bulk oxidation).

On the other end of the scale is Notak. Notak is not a deposition, but a direct functionalization of the substrate surface (316 stainless steel in this case) with organofluoro molecules to provide a stable, low-energy surface which is hydrophobic and oleophobic. (No other SilcoTek surfaces are oleophobic and therefore only Notak was analyzed for hexadecane contact angles.) For hydrophobicity, the Notak coupons showed no change in contact angle after autoclave exposure. For oleophobicity, the Notak coupons had an average loss of 1 degree after autoclave exposure. RD5-SiN is significantly hydrophilic and showed a slight decrease in hydrophilicity after autoclave exposure. The spread of hydrophilic contact angle measurement values was rather large (13°), however, so the slight decrease after exposure is marginally significant. The other surfaces, Dursox, Dursan, SN2000 and SK1000 all showed similar decreases in water contact angle of 9-12°. For those depositions, there was a small degree of hydroxylation as illustrated in the FT-IR data discussed below.

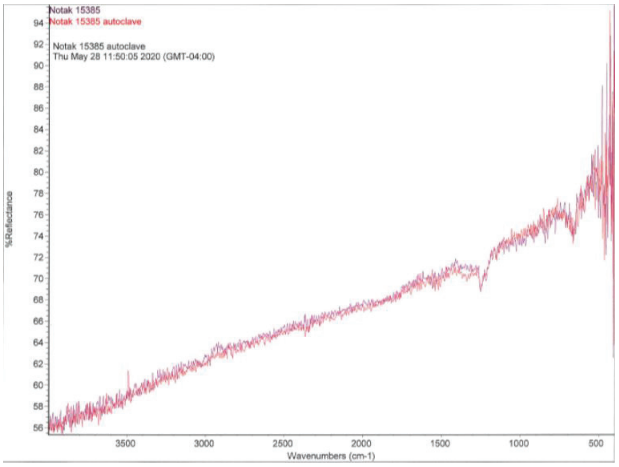
Film thickness evaluation:
Table 2: Average thickness (nm) differences, before and after autoclave exposure.

Surface	Thickness Before	Thickness After	Variance
Silcolloy	400	380	-20
Dursox	664	667	3
Dursan	486	506	20
SN2000	215	219	4
SK1000	211	214	3
RD5-SiN	839	824	-15

The measurement of coating thicknesses was accomplished with a Filmetrics F40 instrument. The F40 uses spectrophotometric absorbance data to non-destructively measure film thickness to a sensitivity of 4nm-40um. The spot size of the instrument is 10um and is difficult to train on to the exact same spot with repeated measurements. Therefore, measurements before and after autoclave exposure on the exact same spot is unlikely and some variation should be expected since the coating itself may have a significant thickness variance throughout due to thermal uniformity and proximity to other parts and fixturing during deposition. Accordingly, the measurement variances in Table 2 are not considered significant. There was no visible loss under magnification during measurement and the differences listed are considered within the expected variance of the instrument and technique.

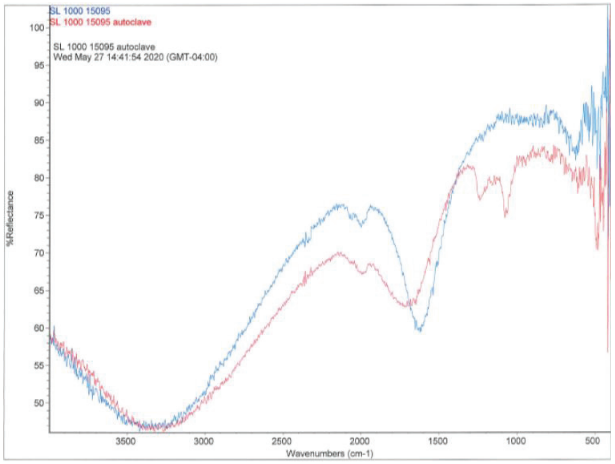
Grazing angle FT-IR evaluation:
FT-IR spectra were obtained with a Thermo/Nicolet 380 FT-IR equipped with a Smart SAGA grazing angle attachment, allowing FT-IR spectra of thin films and surfaces on reflective substrates. The following spectra provide FT-IR data before and after autoclave exposure for each surface.

Notak



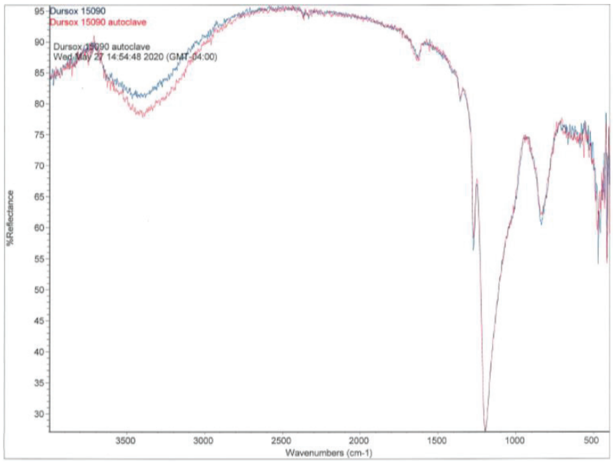
Notak FT-IR analysis: There was no significant change observable via FT-IR between before and after autoclave exposure for the Notak surface, showing Notak to be stable under the autoclave exposure conditions used.

Silcolloy



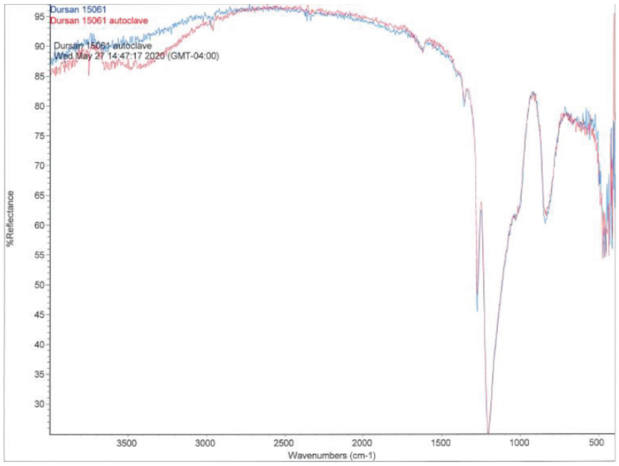
Silcolloy FT-IR analysis: Exposure to the autoclave conditions created Si-O-Si moieties visible in the 1050-1250 cm-1 region. This verifies the cause of loss of water contact angle mentioned above.

Dursox



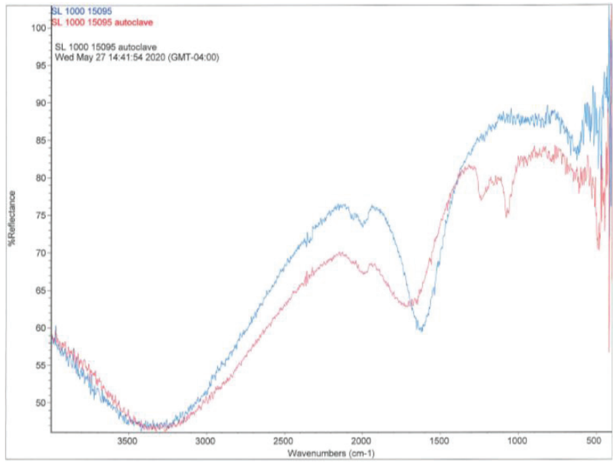
Dursox FT-IR analysis: There was little significant change observable via FT-IR between before and after autoclave exposure for the Dursox surface. There was a slight increase in Si-OH moieties in the broad 3400 cm-1 region, accounting for the slight decrease in water contact angle mentioned above.

Dursan



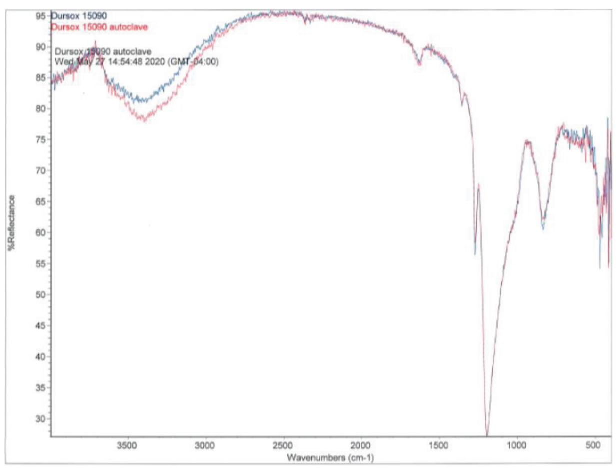
Dursan FT-IR analysis: There was little significant change observable via FT-IR between before and after autoclave exposure for the Dursan surface. There was a slight increase in Si-OH moieties in the broad 3400 cm-1 region, accounting for the slight decrease in water contact angle mentioned above.

SilcoNert 2000



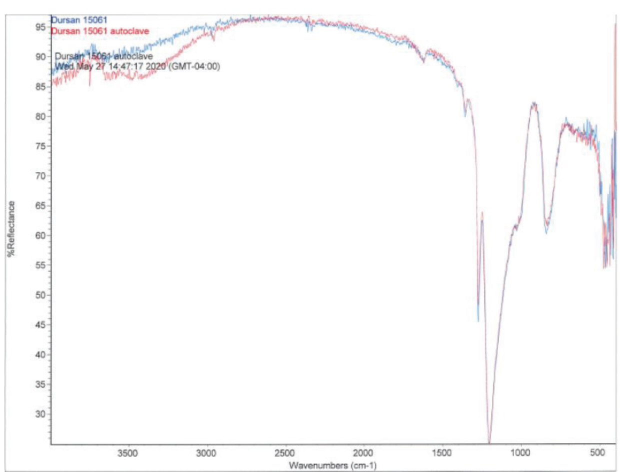
SilcoNert 2000 FT-IR analysis: There was little significant change observable via FT-IR between before and after autoclave exposure for the SilcoNert 2000 surface. There was a slight increase in Si-OH moieties in the broad 3400 cm-1 region, accounting for the slight decrease in water contact angle mentioned above.

SilcoKlean 1000



SilcoKlean 1000 FT-IR analysis: There was little significant change observable via FT-IR between before and after autoclave exposure for the SilcoKlean 1000 surface. There was a slight increase in Si-OH moieties in the broad 3400 cm-1 region, accounting for the slight decrease in water contact angle mentioned above.

RD5-SiN



RD5-SiN FT-IR analysis: There was no significant change observable via FT-IR between before and after autoclave exposure for the RD5-SiN surface, showing RD5-SiN to be stable under the autoclave exposure conditions used.

Conclusions

The exposure of several SilcoTek surfaces to autoclave conditions revealed the following:

- Notak surface energy measurements and FT-IR spectra were unaffected, signifying excellent resilience to autoclave conditions
- RD5-SiN surface energy measurements and FT-IR spectra were unaffected, signifying excellent resilience to autoclave conditions
- Dursan, SilcoNert 2000, SilcoKlean and Dursox showed some sign of surface hydroxylation via autoclave exposure via slight decreases in water contact angle and slight increases in Si-OH functionality via FT-IR
- Silcolloy 1000 was significantly surface-oxidized by autoclave exposure, with a substantial decrease in water contact angle and a measurable increase in Si-O-Si absorbance via FT-IR data.
- None of the SilcoTek depositions revealed any significant loss of coating thickness after autoclave exposure.

Further testing on the effect of autoclave exposure on the corrosion resistance of Silcolloy, Dursan, Dursox and RD5-SiN coatings will be reported at a later date and addended to this document.

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