

**Research Report on the Impeding
Influence of SurTec 101 on the
Successive Nitride Process**

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In cooperation with the IWT Bremen (Stiftung Institut für Werkstofftechnik) was investigated, whether the application of SurTec 101 has a negative effect on the following nitration process (formation of barrier layers).

Problem

The product in test was SurTec 101, a sprayable soak cleaner with temporary corrosion resistance.

After cleaning, residues of cleaner may remain on the surface of the treated part. Especially in case of no or poor rinsing. SurTec 101 was tested regarding to its potential impediment on the following gas nitriding process (formation of barrier layers).

Description of the Product

SurTec 101 is a liquid cleaner, foamless and sprayable above 40 °C. It is suitable for cleaning different materials as steel, stainless steel, non ferrous metals, aluminium and zinc. Zinc electroplated and chromated parts as well as anodised aluminium also are not attacked. Magnesium parts have to be tested before treating them in SurTec 101, since some alloys are not stable enough.

SurTec 101 works demulsifying and bath care of the cleaning bath is possible to extend the life time, e.g. oil separator, membrane filtration (ultra or micro filtration).

Due to its special composition, SurTec 101 also can be applied in ultra sonic, pressure flooding (injection flood washing, hydroson) and hydrovac, vaccuclean and flexiclean processes (= boiling in a vacuum, or inserting air / inert gas in a vacuum).

On steel parts, after drying remains a rinsable corrosion protecting film (temporary corrosion resistance).

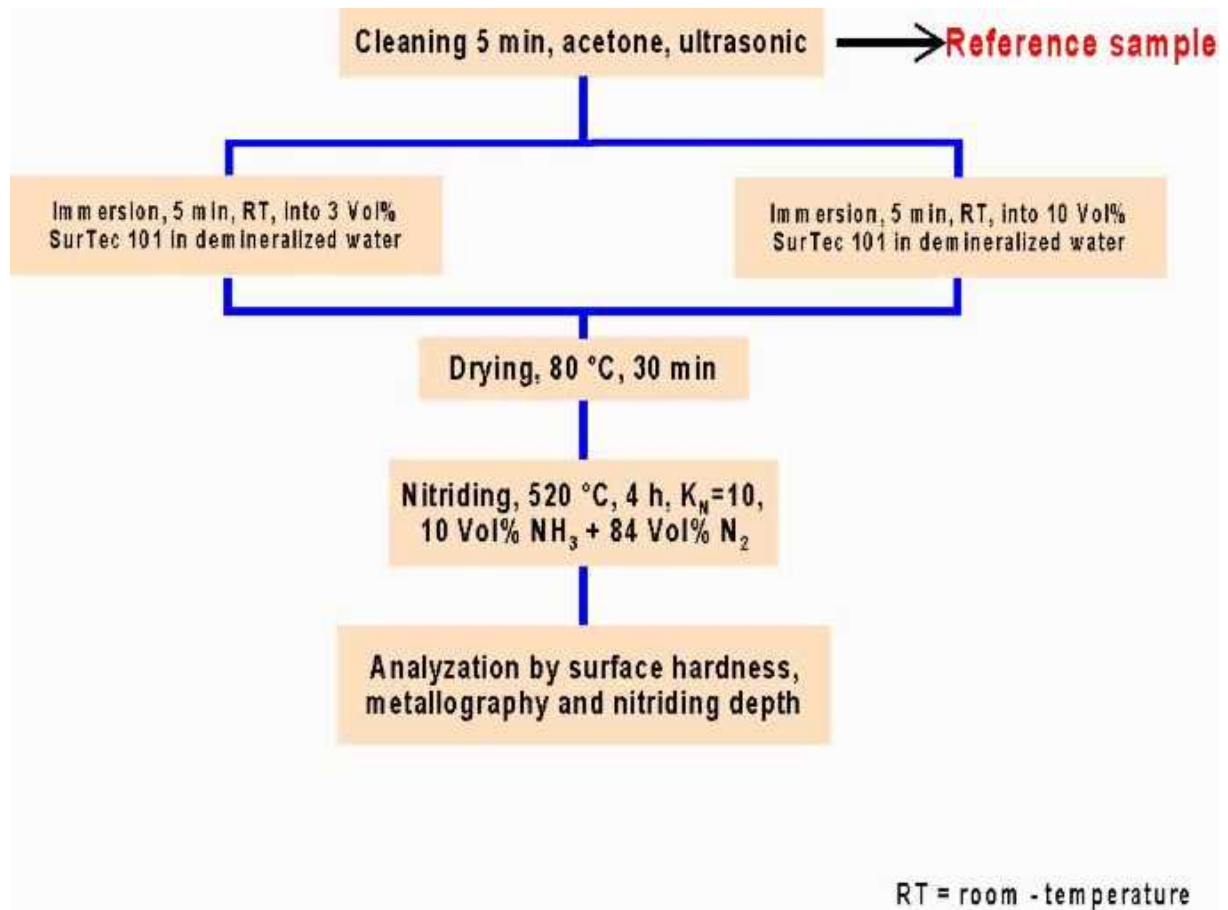
Concentration:	Cleaning without rinsing	1-2 Vol%
	Cleaning with rinsing	2-3 Vol%
	Conservation	0.5-1 Vol%

For further informations please consult our product information sheet SurTec 101.

Test Procedure

The test material was 34CrAlMo5. All tested samples were round shaped with a diameter of 25 mm and a height of 5 mm.

The test method is described in figure 1.



To have equal start conditions, all test parts were cleaned for 5 min in acetone and ultrasonic at room temperature. This also was taken as reference samples.

The cleaner SurTec 101 was applied to the samples as follows:

Solutions of 3 Vol% and 10 Vol% cleaner in deionised water were prepared. The samples were immersed for 5 min at room temperature mixing well with a magnetic stirrer. The drying was done at 80 °C for 30 min in a drying cupboard, until the water was evaporated. In each solution two samples were prepared.

The nitriding process was done in a inert quartz glass reactor at 520 °C for 4 h ($K_N=10$). Successively, the reactor was rinsed for about 10 min with N_2 , it was heated up (30 min) and the nitration itself was done in a (gas) mixture of 16 Vol% NH_3 and 84 Vol% N_2 . The parts were cooled down to room temperature (during 30 min), while rinsing with N_2 .

The nitriding result was controlled by metallographic sections, measuring the Vickers hardness and analysing the nitriding depth. To determine the hardness, each sample had been measured at 5 different sites, equally distributed. The average value and the standard deviation was calculated.

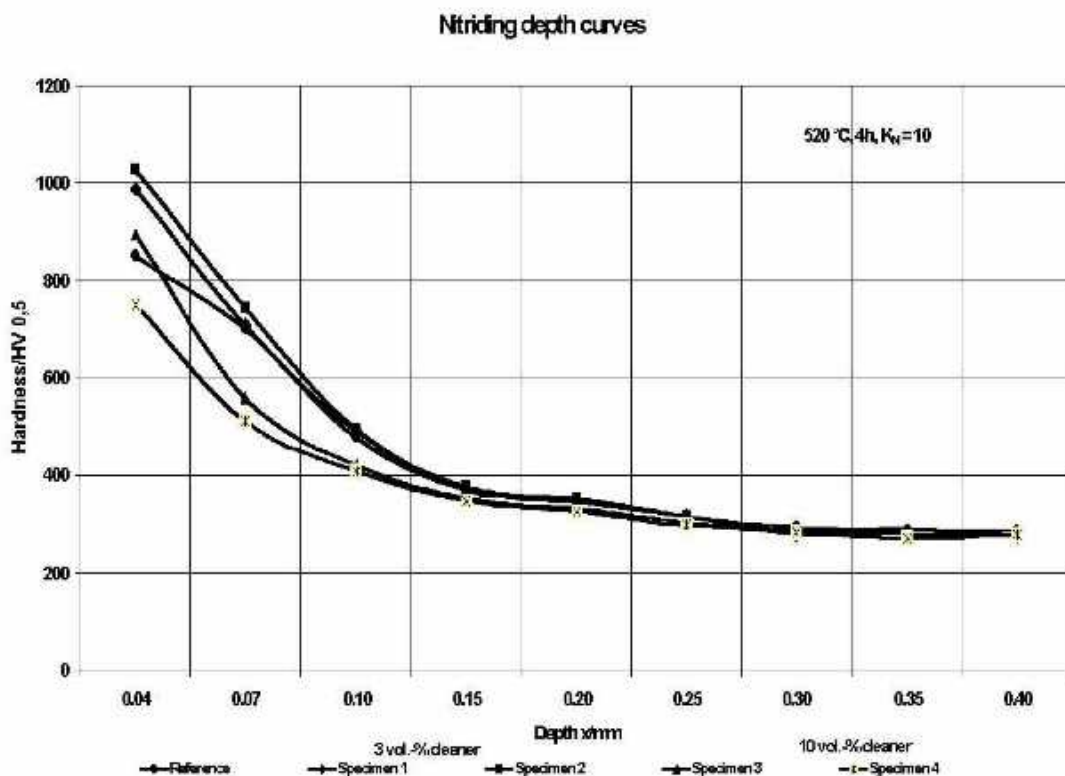
Results

Hardness (Vickers) and nitriding depth of reference and tested samples after nitration (520 °C, 4 h, $K_N=10$) are summarised in table 1.

Sample	Hardness (HV 0.5)	Nitriding Depth (mm)
Reference	1110 ± 25	0.23
Sample 1, 3 Vol% cleaner	1000 ± 35	0.22
Sample 2, 3 Vol% cleaner	1100 ± 50	0.23
Sample 3, 10 Vol% cleaner	1070 ± 45	0.22
Sample 4, 10 Vol% cleaner	1060 ± 60	0.21

The hardness of the bulk is about 270 HV0.5. The surface hardness of the 3 samples immersed in 10 Vol% cleaner are between 1000 and 1100 HV0.5. These values as well as the standard deviations are in the same ranges as the reference sample (1110 ± 25 HV0.5). Also concerning the nitriding depth the values of reference and immersed samples are similar, 0.21 to 0.23 mm.

The nitriding depth of all samples is shown in picture 2. It shows clearly, that reference and the samples of 3 Vol% cleaner (sample 1 and 2) differ only a little bit from one another. The samples immersed into 10 Vol% cleaner yielded in slightly smaller hardnesses.



Both, the results of surface hardness and nitriding depth, prove 3 Vol% cleaner not impeding the nitration, whereas 10 Vol% cleaner weakly seems interfering the nitration, however not totally impeding.

To demonstrate these results, pictures of the sections of all samples after the nitriding process (in 3 different scales) are shown in the following. The smallest scale (100:1) gives a view of a larger area, to see whether the layer is homogenous and whether there are soft spots on the sample. The bigger scales (500:1 and 1000:1) show the formation of the connection layer at the surface.

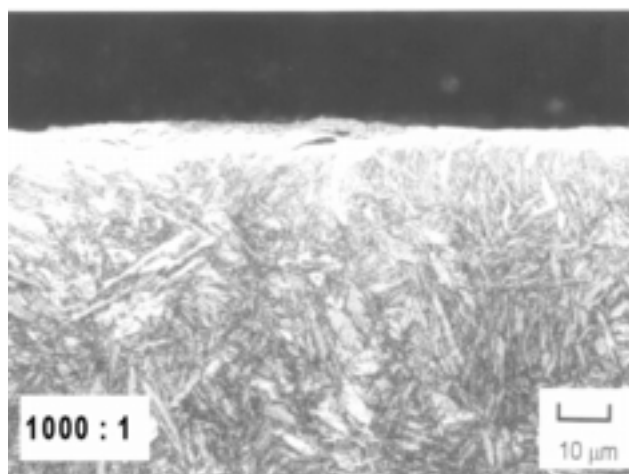
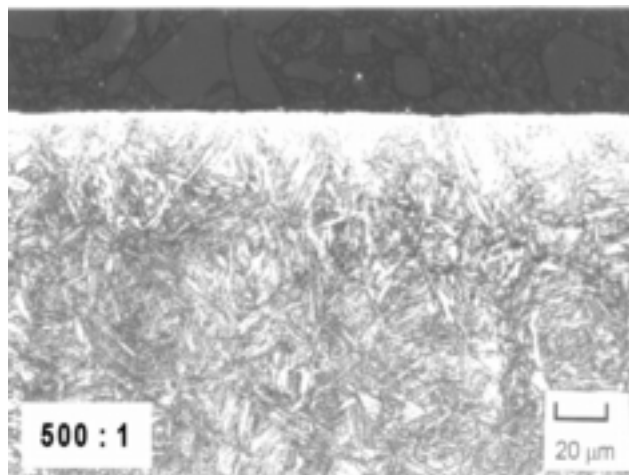
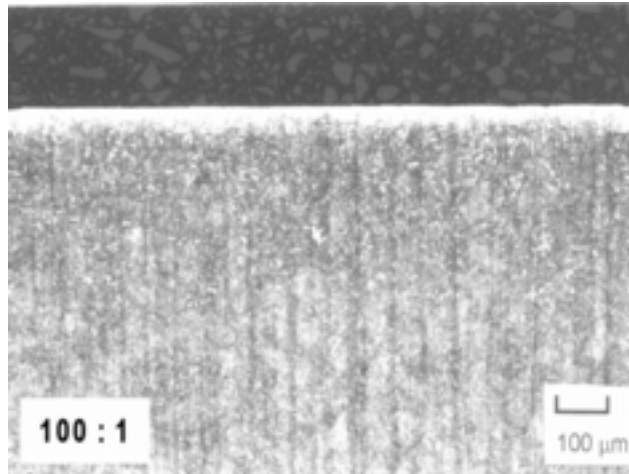
All samples show a white etched homogenous connection layer. Beneath that is the diffusion layer of about 200 μm , clearly visible on all samples, this proves that all samples adsorbed the nitrogen.

At the reference as well as at samples 1 and 2 (immersed in 3 Vol% cleaner), the connection layer is about 30 μm thick. It consists of iron nitrides (pure white layer). The last 5 μm additionally contain chromium nitrides, which are responsible for the high hardness of the surface. The connection layer of sample number 3 (immersed into 10 Vol% cleaner) is thinner compared to the others, it is only about 10-20 μm thick. The thinnest connection layer can be observed at sample number 4. Here, the white iron nitride layer is only about 2-3 μm thick. This very thin layer seems to be irregular, but is formed regular actually. Further tests with cleaner concentrations of more than 10 % would be interesting. However, such high concentrations have no relation to practice and the tests are quite expensive.

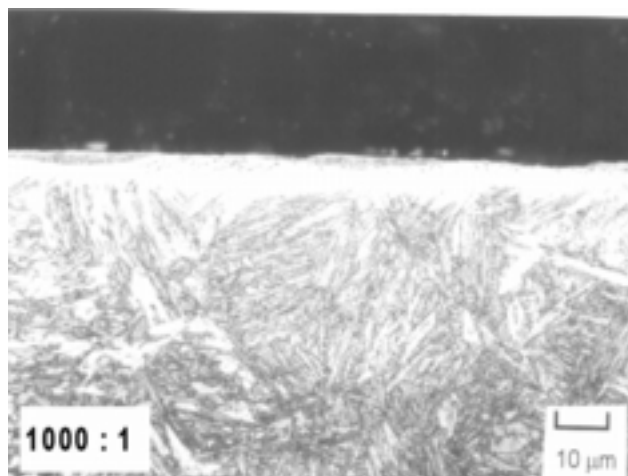
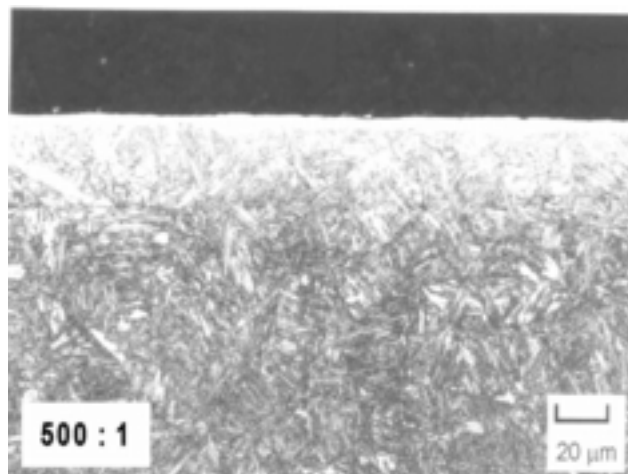
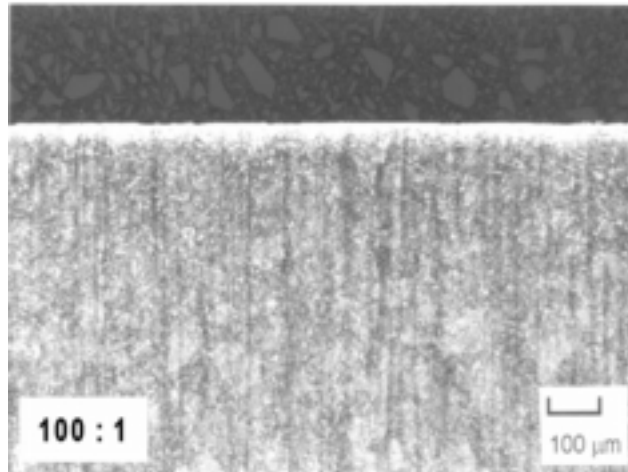
The diffusion layer is formed on all samples in a similar way.

Section pictures

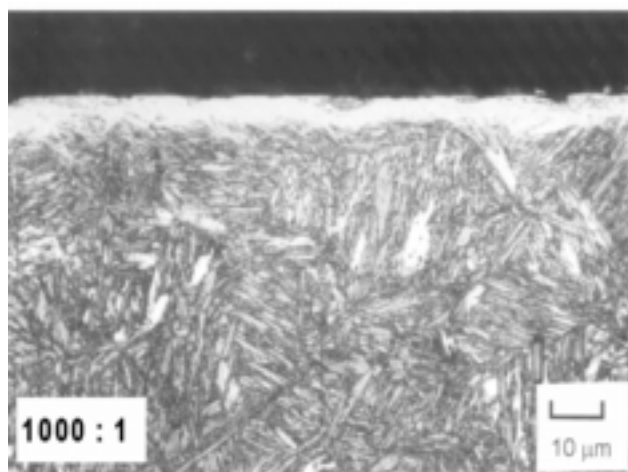
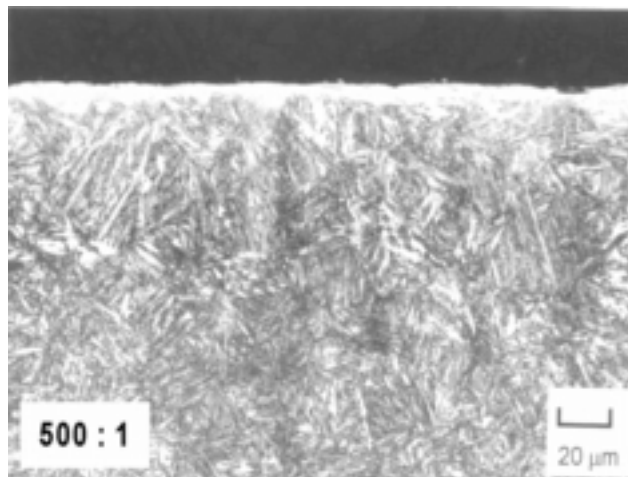
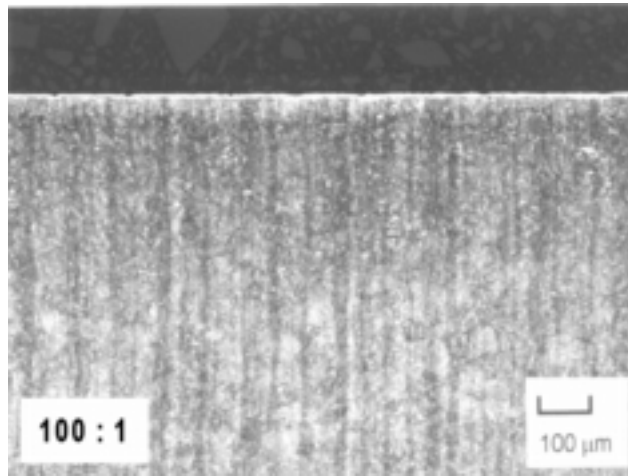
reference sample, etched in 3 Vol% HNO₃



sample SurTec 101 (3 Vol%), etched in 3 Vol% HNO₃



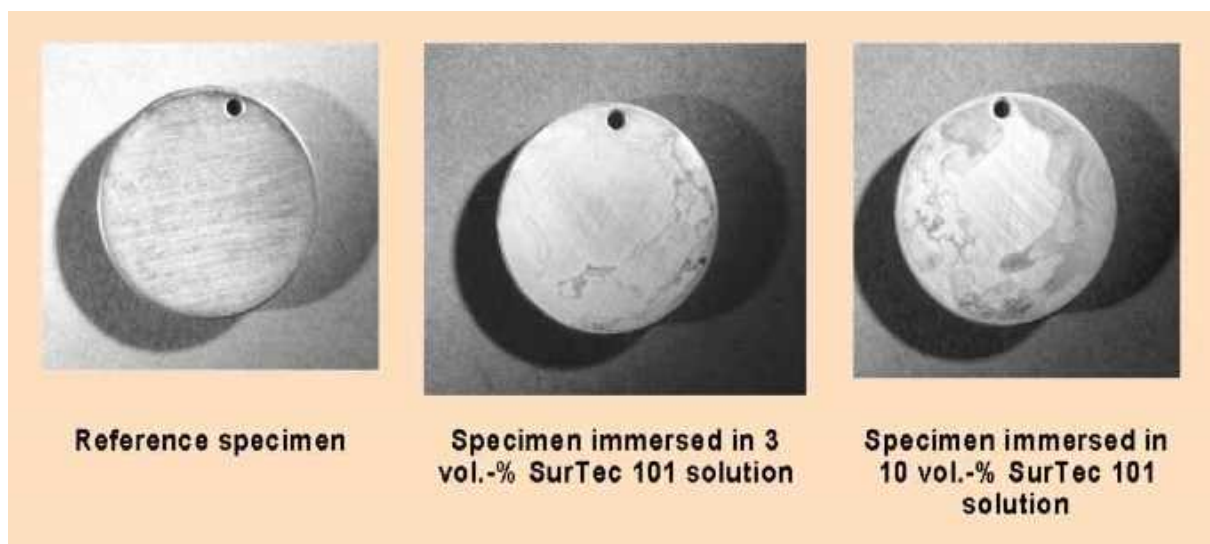
sample SurTec 101 (10 Vol%), etched in 3 Vol% HNO₃



Conclusion

The surface hardnesses of the samples immersed into cleaner solutions are only little lower as the hardness of the reference sample. All values are in between the standard deviation. The standard deviations of the immersed samples are a little higher than of the reference sample, but values up to 60 HV0.5 are not caused by a soft spot fault. Also the homogenous connection layers support this fact. The nitriding depths of all samples are in the same range as the reference sample.

The section pictures prove that all samples adsorbed nitrogen. Furthermore, on all samples a connection layer and beneath that a diffusion layer was formed. The thickness of the connection layer is lower at the sample immersed in 10 Vol% cleaner (sample 3). Perhaps the nitrogen adsorbance had been slower at this sample. The high concentration of cleaner ingredients on top of the part's surface, was the reason also for the colour changes after the nitration, being visible at picture 3.



It was found that a high concentration of cleaner on top of the part's surface may lead to a restricted adsorbance of nitrogen. However, all samples adsorbed a certain quantity of nitrogen, which is proved by the surface hardnesses and the nitriding depth. All measured values are in between the allowed ranges. Hence, there was no complete impediment of the processes being necessary to nitride (adsorption and dissociation of ammonia and nitrogen diffusion).

The presence of cleaner residues on the surface of a part leads to visible spots, which make the optical appearance less appealing.

Sum

It has to be noted that all described trials took place without a following rinsing step. Furthermore, cleaner concentrations of 10 Vol% are far more (3-5 times more) than concentrations of SurTec 101 usually applied.

Thus, working with SurTec 101 according to the product information sheet, the nitration is not influenced. Even at cleaner concentrations of 5 Vol% and no rinsing, no problems in the following nitration should occur.

Cleaner concentrations above 10 Vol% and no rinsing lead to a restricted nitriding process, but they do not interfere it completely (no formation of a barrier layer).

Practical experiences

SurTec 101 is used by several companies and by a producer of heat treatment plants doing hardening as a job shop. SurTec 101 is applied in different processes as spraying (one and two chamber lines), continuous lines (cleaning and rinsing as spraying process) and hydrovac (cleaning, rinsing and conserving).

After the cleaning, the parts are treated in different heat treatment processes as gas nitriding, plasma nitriding and up-carboning. In the users opinion, SurTec 101 cleans better than conventional cleaners. And nearly no problems in the heat treatment coming from an insufficient cleaning grade are detected. This fact leads to higher process security.

The life times of the cleaning baths are extended up to about 3 months using an oil separator and up to about 6 months using micro filtration (reducing costs).

Caused by the long service life of the baths, demineralised water has to be used to compensate the evaporation losses to prevent increasing of salts. Otherwise barrier layers may be formed on account of hardness and other ingredients of the tap water.

Because of the excellent demulsifying properties of SurTec 101, the recycling rate of the cleaner is high, hence the cleaner consumption and the costs for chemistry are reduced.