

## **Dephosphating of Parts before Heat Treatment**

At the Example of Screws

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2. edition

Before reshaping steel wire, e.g. forming the thread of screws, it has to be phosphated. The phosphate layer allows a better sliding and gives a temporary corrosion protection. These phosphate layers have to be removed completely before tempering. Otherwise glass like and diffusion inhibiting iron phosphorases will be formed during the heat treatment. They are very brittle and being present, they can result in blisters and faults whilst successive zinc plating.

By specific bath maintenance and good monitoring of the sequence of treatment, dephosphating baths can be increased both, life time and efficiency. This Technical Letter advices procedures being tested in praxis for increasing the service life.

Iron wire as base material for the production of screws passes several steps like forming, drawing for reducing the diameter or rolling of threads. For better properties and for temporary corrosion protection, the wire has to be phosphated (zinc phosphating) and treated with support compounds, such as draw-aids or water insoluble soaps.

The phosphate layer and the support compounds have to be removed completely before the following heat treatment (glowing,-possibly tempering-, quenching and annealing). If not, crack products of the support compounds will be formed as well as glass like iron phosphate layers.

These residues can behave diffusion inhibiting at thermochemical diffusion processes like nitrifying. At following galvanic processes (e.g. zinc plating) they can lead to blisters or faults, since they can hardly be removed during the pretreatment but by pickling over a long period. However, strong and long pickling leads to a high diffusion rate of hydrogen into the bulk with all its disadvantages (see *SurTec Technical Letter 8*). Thus, the time for pickling should be as short as possible.

Similar problems occur, if the oil of the quenching process is not removed completely.

The dephosphating is done in a highly alkaline solution ( $\text{pH} > 11$ ). Yet, the applied support compounds cause problems.

Due to organics containing chloride being banned (disposal, AOX-problem) from production, the chlorinated paraffines (draw supporters) have been replaced by highly ester containing compounds. Esters improve the tribological properties of supporting compounds indeed.

They are synthesized by reaction of carbonic acids and alcohols. Also natural oils (triglycerides) are esters (native esters, e.g. rape or root oil) composed of glyceride and long chained fatty acids (carbonic acids).

Via hydrolysis in aqueous alkaline solutions, these esters are separated into soaps and alcohols. This process of saponification is accelerated by temperature and mixing.

The crack product soap usually behaves emulsifying and prevents oil to demulsify in the cleaning bath. Furthermore, the water soluble soaps generate and stabilize foam and are causing problems. Additionally, these soaps can react with magnesium or calcium from water and with iron or zinc from parts forming insoluble chalk or metal soaps, which can precipitate on the parts and on the process equipment.

In the following, hints for bath care and examples for the process sequences are given. It will be shown how the cleaning baths will work more efficient having an extended service life. The possibility of a waste water free process is described.

## **1. Pre-Cleaning**

To reduce the saponification to a minimum, before the dephosphating bath should be a low alkaline ( $\text{pH} < 9$ ), demulsifying cleaning with successive rinsing. For instance SurTec 131 (builder) + SurTec 089 (surfactant for soak application) or SurTec 086 (surfactant for spray application, foam reduced). For easier or automatic dosage (by conductivity), both compounds are liquid.

Even in the weak alkaline medium a few soaps are formed, however, they can be destroyed (and removed) by adding demulsifiers as SurTec 930. The demulsified oil and the small amounts of smud (zinc phosphate, metal soaps, chalk soaps), can be removed in an oil separator being installed in a bypass. The oil separator has to be provided with a sloping bottom and an outflow to let off the deposited smud. Alternatively, a slope clarifier can be used. The demulsified oil has to be removed continuously to prevent further saponification.

Due to the zinc phosphate layer having a crystalline structure, the supporting compounds can not be removed to 100 %. During the pre-cleaning, the quenching oils can also be removed.

## 2. Dephosphating

To remove the phosphate layer, highly alkaline (pH > 12) products containing complexing agents, and being free of surfactants are used. For instance SurTec 198 or SurTec 194 (alkaline compound) + SurTec 419 (complexing agent). The complexing agents are biodecomposable and the metal complexes (zinc, iron) can be removed easily in usual waste water treatment.

For easier or automatical dosage (by conductivity), both compounds are liquid.

If a pre-cleaning is not possible (e.g. not existant in the present process line) surfactants have to be added to the dephosphating bath (e.g. SurTec 089, SurTec 086). The soaps being formed can be destroyed completely by using demulsifying agents like SurTec 930.

Depending on the thickness of the phosphate layer and on the throughput, big amounts of smut can be built up. They can be removed by slope clarification, separators or decanters. Demulsified oil can be removed by an additional oil separator. Again, the demulsified oil has to be removed continuously to prevent further saponification.

## 3. Rinsing, corrosion protection

In the successive rinses, water soluble corrosion inhibitors as SurTec 534 or SurTec 533 can be added. Both products prevent corrosion while storing. They evaporate at about 180-200 °C and do not disturb the following heat treatment or thermochemical diffusion processes, respectively.

## 4. Examples for the processes sequence

Pre-cleaning:

SurTec	concentration [Vol%]	temperature [°C]
131 + 089	3-4 + 0.5	50-70

Dephosphating in succession of pre-cleaning:

<b>SurTec</b>	<b>concentration [Vol%]</b>	<b>temperature [°C]</b>
198	5-10	50-70

alkaline compound and complexing agent separately:

<b>SurTec</b>	<b>concentration [Vol%]</b>	<b>temperature [°C]</b>
194 + 419	5-10 + 2-4	50-70

Dephosphating without pre-cleaning:

<b>SurTec</b>	<b>concentration [Vol%]</b>	<b>temperature [°C]</b>
198 + 089	5-10 + 0.5	50-70

alkaline compound and complexing agent separately:

<b>SurTec</b>	<b>concentration [Vol%]</b>	<b>temperature [°C]</b>
194 + 419 + 089	5-10 + 2-4 + 0.5	50-70

Rinsing, corrosion protection:

<b>SurTec</b>	<b>concentration [Vol%]</b>	<b>temperature [°C]</b>
533 or 534	1-2	50-70

Drying

### 5. Example of a waste water free process line

For the process technique, different types of processes can be applied: chain, barrel, rommel or snail processes.

A waste water free process could be established as follows:

