Dephosphating of high strength components before heat treatment

By Stefan Lenzer, global product manager metallurgy, MacDermid GmbH

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Dephosphating of high strength components before heat treatment

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High strength fasteners must be dephosphated before heat treatment to prevent phosphate residues having a negative influence on the performance of the heat treatment process. This article looks at the process of phosphating and dephosphating as well as application and quality control.

Phosphating and cold forming

In order to carry out the cold forming of steel, components such as fasteners, wire and pipes are usually phosphated and coated with lubricants. The phosphate layer serves as a lubricant carrier and as a barrier between the work piece and the tool.

The principle is based on the formation of phosphate layers, which are anchored firmly in the metal surface and provided with special lubricants. Through the combination of the adherence and separation effects of the carrier layers on the one hand, and the separation and gliding effects of the special lubricants on the other hand, high degrees of metal forming can be attained with excellent surface quality, long tool service life and moderate press capacities.

After forming, the cold formed components will have a 'phosphate pressing pattern' on their surface. This consists of zinc or the zinc calcium phosphate coating and a lubricant (e.g. metal soaps, polymers, molybdenum disulphide, graphite).

After cold forming fasteners are heat treated. This process can be negatively affected by the phosphate pressing pattern. This causes an increased sensitivity to cracking and intergranular corrosion. Additionally any phosphate residue present on the surface during the non-oxidising annealing treatment also leads to a diffusion of phosphorus into the marginal zone. This contributes to the sensitivity of the components to stress corrosion cracking and the probability of failure increases. This is especially true with high-tensile fasteners, for example Class 12.9 and higher.

Dephosphating

To avoid these problems this residual phosphate/lubricant layer must be removed before the heat treatment is commenced. According to DIN EN ISO 898-1 a detectable, ascertainable white zone, enriched with phosphorus is not permitted. Additionally the Volkswagen specification of VW 60250 high strength bolts states bolts of strength Class 10.9 and higher must be dephosphated before heat treatment.

The phosphate pressing pattern can be mechanically removed by blasting or by chemical means such as acid or alkaline stripping treatments. Due to the risk of hydrogen induced brittle fracture, acid processes are often prohibited. Therefore alkaline processes are preferred.

Processes

The simplest alkaline dephosphating systems are based on mixtures of strong alkalis (i.e. sodium hydroxide or potassium hydroxide) in combination with a complexing agent. The effectiveness of these simple solutions is quickly reduced by contamination. Very quickly the application time and the bath concentration must be increased in order to achieve a consistently good result. Also these solutions tend to produce foam, which then solidifies and is dragged through the entire system. Conventional alkaline cleaning solutions are not the answer either. Since substantial amounts of lubricants build up in the bath and tend to solidify or turn to a gel when cooled below 65°C.

Modern dephosphating, such as KeyKote CL 400, are formulated so that these undesirable effects do not readily occur. KeyKote CL 400 can also cope with the build-up of forming lubricants and soaps without creating excessive foam. Additionally this alkaline system cleans components in this same process step.

Plant engineering

Dephosphating is usually carried out in barrel immersion plants, dip spin systems or Archimedes screw systems operating at more than 65°C. The type of equipment is highly dependent on the required throughput and the part geometry. In barrel immersion plants the application time is generally more than 10 minutes. whereas in dip spin systems 3 minutes – 6 minutes (see Table 1) and in screw 3 minutes – 10 minutes application times are the norm. Dip spin systems are highly effective, but require a high performing dephosphating product like KeyKote CL 400 – which can dephosphate in the short application time, has a low tendency to foam and has a low impact on the waste water.

The dosage of the chemical concentrates can be carried out remotely via metering pumps. The adjustment of the pump can be based on the production rate, average product usage and analysis of the solution concentration. This means that plant operators have limited contact with the concentrates, increasing worker safety and improving process control.

Table 1: Example of process flow // AT= ambient temperature

Pos.	Process	Parameter	Time
1	Degreasing	>65°C	4 mins
2	Dephosphating	>65°C	4 mins
3	Rinsing	AT-65°C	2 mins
4	Rinsing	AT-65°C	2 mins
5	Hot rinse	>65°C	2 mins
6	Drying, hot air	120°C	2 mins

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Floating oil can be removed from the process solution. The classic oil separator configuration has proven to be very effective but even a simple oil skimmer mounted directly in the process bath also works well. A common dip-spin procedure is as follows:

Application

The product KeyKote CL 400 offered by MacDermid is a liquid concentrate based on a special blend of potassium hydroxide and soft complexing agents, which can be treated in the usual wet chemical wastewater treatment plant.

Depending on the requirements, the product is combined with a surfactant such as KeyKote CL ADD 501 (see Table 2) to give increased emulsifying power.

Used at extremely low concentration KeyKote CL 400 has an excellent absorption capacity for oils, fats and soaps and has an extremely long bath life. KeyKote CL 400 also avoids the solidification (jellifying) of a soap lubricant 'contaminated' working bath at a lowered temperature (during down times).

Due to its special formulation KeyKote CL 400 is ideally suited for the use in drum, dip spin and even screw systems without creating problematic excess foam. The working parameters and system settings depend on the respective plant and the components that are being dephosphated.

Table 2: Application parameters of KeyKote CL 400

	Degreasing	De-Phosphating
Working concentration KeyKote CL 400	30-100 mL/L	100-150 mL/L
Operating temperature	35°C – 80°C	60°C – 80°C
Optional:		
KeyKote CL ADD 501		
Surfactant addition depending on the requirements	2-20 mL/L	

Quality control

The dephosphating bath is usually analysed by a simple acid / base titration. The proportion of soaps and oils can be determined by an "acid splitting" reaction. A bath sample is mixed with mineral acid and after reaction the floating oily substance is measured and recorded as percentage by volume. This gives the concentration for the contaminant. Furthermore, the zinc and complexing agent concentrations can be analysed and if necessary additions made separately in order to increase the bath life.

The dephosphating performance is determined colorimetrically by the so-called "blue test". A defined surface of the component is immersed in a chemical solution, which will react with any residual phosphate present to form a blue colour. The intensity of the colour is proportional to the amount of phosphate present.

Summary

The effective removal of the phosphate layer prior to the heat treatment is of critical importance for the quality of the parts produced. Modern equipment and increased quality levels require powerful dephosphating products. KeyKote CL 400 from MacDermid is a tried and tested modern dephosphating product and satisfies the criteria outlined in this article.

By using KeyKote CL 400 the quality of your dephosphating process can be improved. In particular users of KeyKote CL 400 report improved consistency through factors such as longer/ more predictable bath life and automation in maintaining solution concentration. ■

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