TUFFTRIDE®-QPQ-PROCESS

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Technical Information

TUFFTRIDE® - QPQ - Process

Salt bath nitrocarburizing by the TUFFTRIDE process has been applied in a wide range of industries throughout the world for many decades. It is used to improve the wear resistance, the fatigue strength and – in particular when combined with the oxidative cooling – the corrosion resistance of components made from steel, cast iron and sintered iron materials. In many cases the TUFFTRIDE process is used as an alternative to other surface engineering processes such as case hardening, galvanic (e.g. hard chrome plating), and other coating processes (plastic coating, painting, laser coating etc.), also plasma or gas nitrocarburizing with equally good or improved quality and greater economy.

Carrying out the process

Compared with other nitrocarburizing processes, the TUFFTRIDE Q, QP and QPQ processes are very easy to carry out. As is usual when treating components in salt baths, the parts are first preheated to about 350°C in air. Nitrocarburizing takes place in a so-called TF 1 bath at 480 - 630°C, the standard temperature is usually 580°C.

The salt melt mainly consists of alkali cyanate and alkali carbonate. It is operated in a pot made from special material, and the pot is fitted with an aeration device. The active constituent in the TF 1 bath is the alkali cyanate. During the nitrocarburizing process a reaction takes place between the surface of the components and the alkali cyanate, resulting in the formation of alkali carbonate. By adding specific amounts of the non-toxic regenerator REG 1, the nitriding active constituents are again produced in the salt melt and the activity of the TF 1 bath is kept within very strict tolerances (Fig. 1).
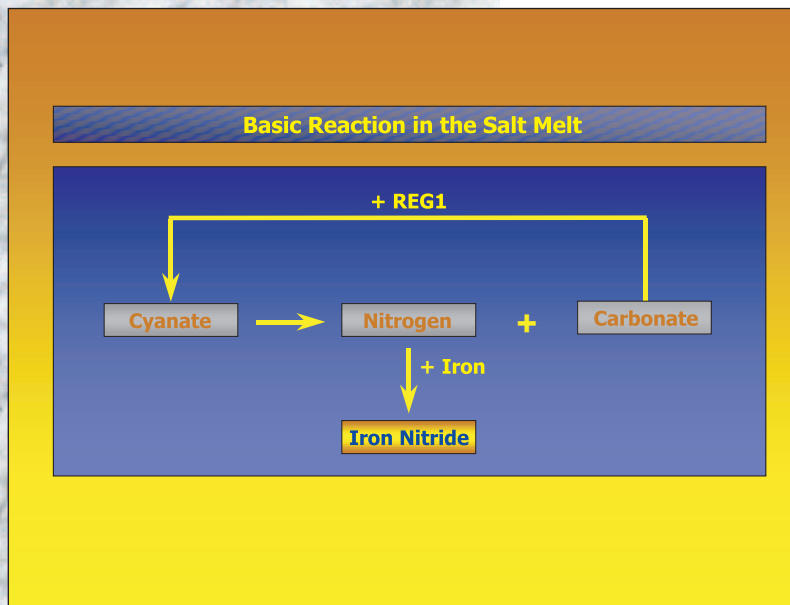

Basic Reaction in the Salt Melt

+ REG1

Cyanate → Nitrogen + Carbonate

+ Iron

Iron Nitride

Fig. 1

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As the regeneration takes place without any change in the volume of the nitrocarburizing bath, part of the melt does not have to be bailed out. Drag-out losses which occur when the parts are removed from the bath are supplemented with TF 1 replenisher salt. Unlike gas nitriding or gas nitrocarburizing, the substances – TF 1 and REG 1 - needed for the TUFFTRIDE QPQ process, do not contain constituents classified as toxic or harmful to the environment.

A specially developed cooling bath (AB 1 bath) is used for carrying out the oxidative treatment after salt bath nitrocarburizing. During this treatment a black iron oxide layer (magnetite) is produced on the surface of the treated parts, which greatly enhances the corrosion resistance. The temperature of the cooling bath is 350-400°C. Apart from the oxidative effect, the bath has a positive influence on the dimensional stability of the cooled components.

Thereafter, the parts are cooled to room temperature and then cleaned (TUFFTRIDE Q process).

If the surface of the components after nitrocarburizing is not smooth enough for certain applications, depending on the size and shape of the parts, various polishing methods can be used to reduce the roughness (TUFFTRIDE QP process). Some proven methods are:

- Lapping with emery cloth grade 360 or finer;
- Polishing or continuous microfinishing with special plastic discs similar to centreless polishing, or on an automated lathe fixed between centre pieces or clamped in;
- Polishing in a vibrating drum. This method is primarily used for small and thin parts;
- Blasting with glass beads size 40-70 µm in diameter. To prevent edges being excessively rounded off, or the thickness of the compound layer reduced, the pressure should not exceed 4 bar;
- Automated blasting with metal shot, if possible the diameter should be less than 1 mm.

Mechanical processing can, however, partly reduce the corrosion resistance gained. For this reason, in many cases an oxidative post treatment in an AB 1 bath is carried out after polishing.

This complete process sequence is shown in Fig. 2 and is in fact the TUFFTRIDE QPQ process. QPQ means Quench Polish Quench and comprises TUFFTRIDE treatment with oxidative cooling, mechanical processing and oxidative post treatment in a salt melt.
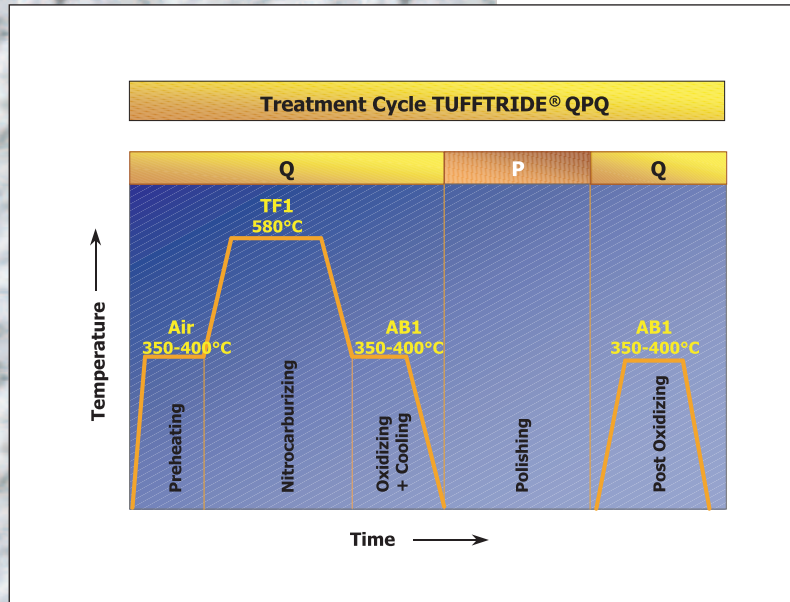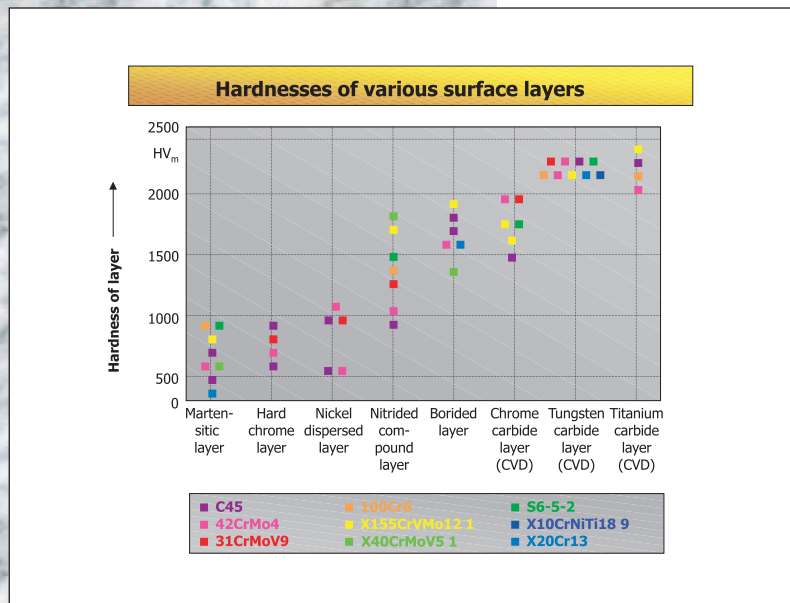
Composition and thickness of the nitride layer

Compound layer

During salt bath nitrocarburizing by the TUFFTRIDE process a nitrocarburized layer is formed consisting of the outer compound layer (ε-iron nitride) and the diffusion layer thereunder. The formation, microstructure and properties of the compound layer are determined by the base material.

The compound layer consists of compounds of iron, nitrogen, carbon and oxygen. Due to its microstructure, the compound layer does not possess metallic properties. It is particularly resistant to wear, seizure and corrosion, as well as being stable almost to the temperature at which it was formed. Compared with plasma or gas nitrocarburizing, compound layers with the highest nitrogen content can be obtained by the TUFFTRIDE process. Layers with a high nitrogen content give better protection against wear, and in particular corrosion, than those with a low content.

Depending on the material used, the compound layer will have a Vickers hardness of about 800 to 1500 HV. Fig. 3 shows a comparison of the surface layers produced by various processes and their hardness.

Fig. 2

Fig. 3

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In the metallographic analysis of salt bath nitrocarburized components, that part of the total layer known as the compound layer is defined clearly from the diffusion layer thereunder as a slightly etched zone. During the diffusion of atomic nitrogen the compound layer is formed. The growing level of nitrogen results in the limit of solubility in the surface zone being exceeded, which causes the nitrides to precipitate and form a closed compound layer.

In addition to the treatment parameters (temperature, duration, bath composition), the levels of carbon and alloying elements in the materials to be treated influence the thickness of layer obtainable. Although the growth of the layer is lower the higher the content of alloy, the hardness however increases to an equal extent.

The data shown in Fig. 4 were determined in a TF 1 bath at 580°C. With the usual treating durations of 60-120 minutes, the compound layer obtained was 10-20 µm thick on most qualities of material.

Diffusion layer

The depth and hardness of the diffusion layer are largely determined by the material. The higher the alloying content in the steel, the lower the nitrogen penetration depth at equal treating duration. On the other hand, the hardness increases the higher the alloying content.

Obtainable compound layer thickness in relation to the treating time

Compound layer thickness
TUFFTRIDE TF1 580°C

C15 - C45
QT steel
Hot work steel
12 % Cr-steel GGL

Compound layer thickness in µm

Treating time in the TF 1 - bath (hrs)

Fig. 4

In the case of unalloyed steels, the crystalline structure of the diffusion layer is influenced by the rate of cooling after nitrocarburizing. After rapid cooling in water, the diffused nitrogen remains in solution. If cooling is done slowly, or if a subsequent tempering is carried out, some of the nitrogen could precipitate into iron nitride needles in the outer region of the diffusion layer of unalloyed steels. This precipitation improves the ductility of nitrocarburized components. Unlike unalloyed steels, part of the diffusion layer of high alloyed materials can be better identified metallographically from the core structure, due to the improved etchability.
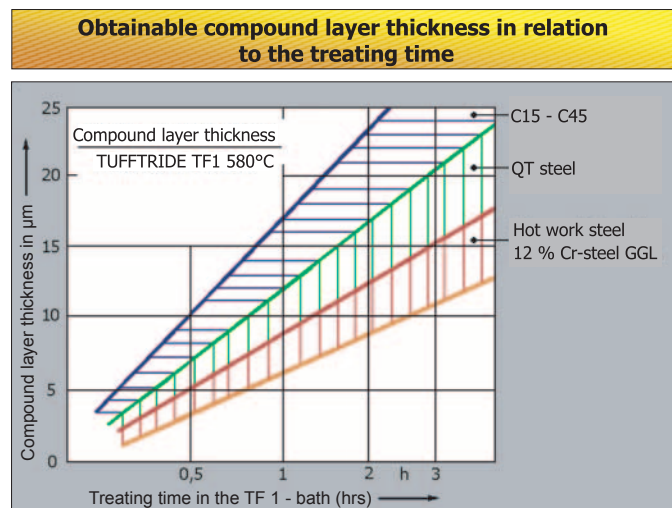
Total nitriding depth after salt bath nitriding of various steels in relation to the treating time
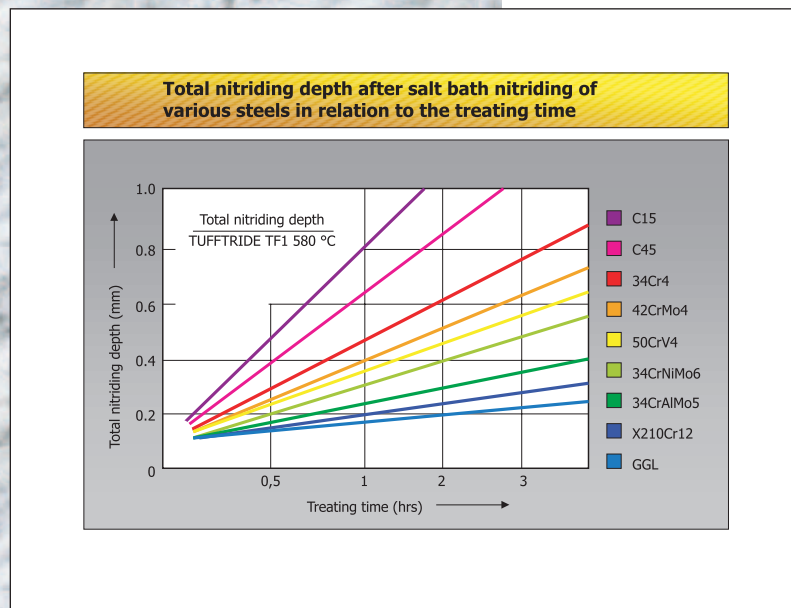
Fig. 5

But the actual nitrogen penetration is also considerably deeper than the darker etched area visible metallographically.

Cooling does not influence the formation of the diffusion layer to any noteworthy extent. Fig. 5 shows the depth of nitration on various materials in relation to the nitrocarburizing duration.

Surface hardness and tensile strength

The surface hardness obtainable by TUFFTRIDE treatment is mainly influenced by the composition of the material. The higher the content of nitride-forming alloying elements (Cr, Mo, Al, V, Mn, Ti, W) the greater the surface hardness. Fig. 6 gives the average tensile strength and surface hardness of salt bath nitrocarburized steels.

Steel		Core strength after hardening and tempering Temper. temperature (N/mm^2)		Surface hardness 90 min 580°C TUFFTRIDE		
Name	Steel number	2 hours	6 hours	HV 1	HV 10	HV 30
Ck15	1.1141	600	550	350	300	200
C45W3	1.1730	750 - 850	700 - 800	450	350	250
Ck60	1.1221	750 - 900	700 - 800	450	350	250
20MnCr5	1.7147	800 - 950	800 - 900	600	450	400
53MnSi4	1.5141	850 - 950	800 - 900	450	400	350
90MnV8	1.2842	1000 - 1200	900 - 1100	550	450	400
42CrMo4	1.7225	900 - 1200	900 - 1100	650	500	450
X19NiCrMo4	1.2764	900 - 1100	900 - 1000	600	500	450
55NiCrMoV6	1.2713	1200 - 1400	1150 - 1300	650	550	500
56NiCrMoV7	1.2714	1300 - 1500	1250 - 1400	650	550	500
50NiCr13	1.2721	1200 - 1350	1100 - 1200	600	500	450
X20Cr13	1.2082	1000 - 1200	1000 - 1200	> 900	600	450
X35CrMo17	1.4122	1000 - 1200	1000 - 1200	> 900	700	550
X210Cr12	1.2080	1500 - 1700	1400 - 1600	> 800	600	450
X210CrW12	1.2436	1500 - 1800	1400 - 1650	> 800	600	500
X165CrMoV12	1.2601	1400 - 1900	1400 - 1700	> 800	650	500
45CrMoW58	1.2603	1500 - 1800	1400 - 1700	800	700	600
X32CrMoV33	1.2365	1700 - 1800	1600 - 1750	> 900	850	700
X38CrMoV51	1.2343	1700 - 1900	1500 - 1700	> 900	850	700
X37CrMoW51	1.2606	1700 - 1900	1600 - 1800	> 900	800	700
X30WCrV53	1.2567	1700 - 1900	1600 - 1800	> 900	850	750
X30WCrV93	1.2581	1500 - 1800	1500 - 1700	> 900	850	800

Fig. 6

Changes in component properties through the QPQ treatment

Corrosion resistance

To determine the corrosion resistance of samples and components, a salt spray test (German Standard DIN 50021) and a total immersion test (German Standard DIN 50905/part 4) are often carried out.

In the simple salt spray test the parts are subjected to a fine mist of a 5% solution of sodium chloride at 35°C. This test is referred to in the German Standard as SS.

Fig. 7 shows the results of a salt spray test conducted in accordance with DIN 50021 SS on hard chrome plated piston rods and TUFFTRIDE nitrocarburized ones made from unalloyed steel C35. The piston rods were either hard chrome plated to a layer thickness of 15-20 μm or salt bath nitrocarburized for 90 minutes to obtain a compound layer 15-20 μm thick. In the case of the salt bath nitrocarburized piston rods, different variants such as nitrocarburizing plus oxidative cooling, with and without lapping, as well as the QPQ treatment were tested. After being sprayed for 40 hours, the first corrosion spots occurred on the chrome plated piston rods. After 180 hours the rods showed very heavy corrosive attack over a large area. All nitrocarburized piston rods, however, were still free from corrosion after 40 hours and even after 180 hours the QPQ treated piston rods showed no signs of rust.

Fig. 8 shows the corrosion resistance measured in a DIN 50021 SS salt spray test of samples made from material C45 after each stage of treatment. Fig. 9 shows the respective surface roughness of the samples.
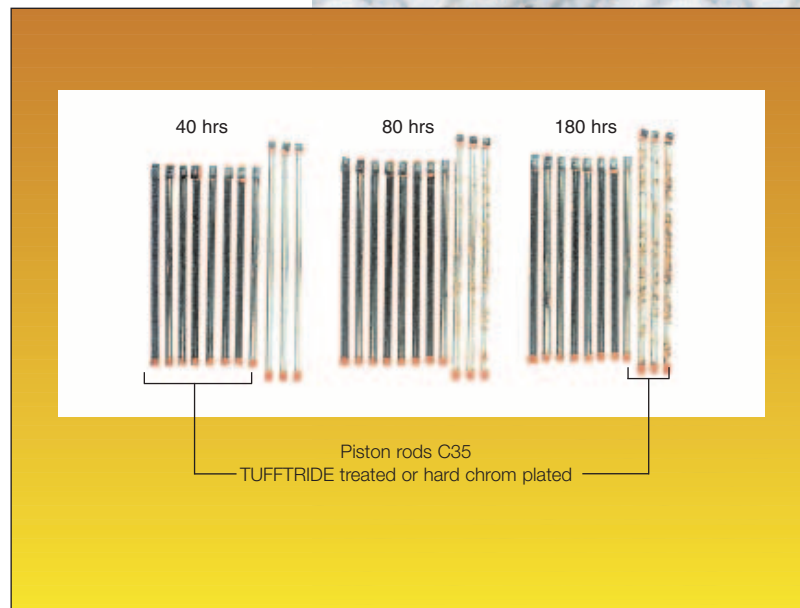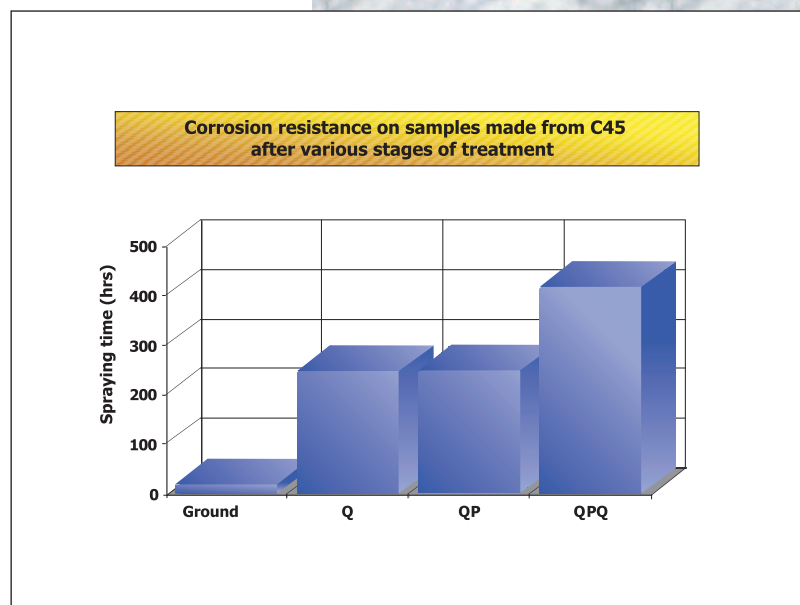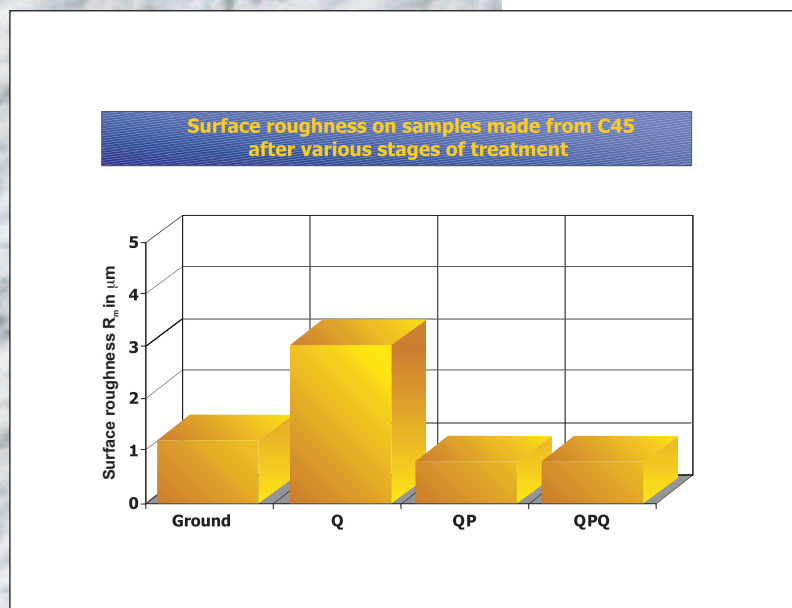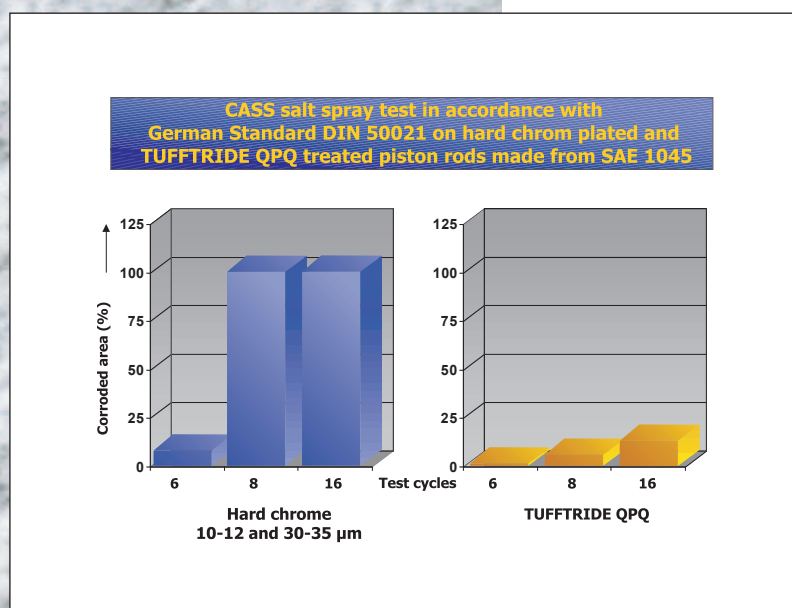

40 hrs   80 hrs   180 hrs

Piston rods C35
TUFFTRIDE treated or hard chrom plated

Fig. 7

Corrosion resistance on samples made from C45 after various stages of treatment

Spraying time (hrs)

Ground   Q   QP   QPQ

Fig. 8

Surface roughness on samples made from C45 after various stages of treatment

Surface roughness R_m in μm

| Ground | Q | QP | QPQ |

Fig. 9

CASS salt spray test in accordance with German Standard DIN 50021 on hard chrom plated and TUFFTRIDE QPQ treated piston rods made from SAE 1045

Corroded area (%)

Hard chrome 10-12 and 30-35 µm

Test cycles: 6, 8, 16

TUFFTRIDE QPQ

Fig. 10

In the ground condition, corrosion occurred after only a short time. After 90 minutes salt bath nitrocarburizing followed by oxidation in the cooling bath the corrosion resistance was over 200 hours. Lapping does not change the resistance of the samples. After oxidative post treatment in the cooling bath (25 mins. at 370°C), figures of over 400 hours will be obtained.

The most stringent corrosion test under DIN 50021 is the CASS test in which the test solution additionally contains acetic acid and copper chloride, and the temperature is raised to 50°C. Fig. 10 shows the results obtained in a comparison between QPQ treated piston rods and hard chrome plated ones with layer thicknesses of 10-12 µm and 30-35 µm.

The test was performed by the Material Testing Institute in Darmstadt, Germany under the following conditions:

Spray solution
5 % NaCl + 0.26 g $CuCl_2$/l;
pH 3.11-3.3;
Temperature 50°C
(1 test cycle = 1 hour).

After QPQ treatment, the corrosion resistance is much better than after hard chrome plating. After 16 hours the QPQ treated samples merely showed corrosive attack on about 10 % of the surface.

For the total immersion test (DIN 50905/part 4) a solution of 3 % common salt and 1 % hydrogen peroxide (H_2O_2) is used as the corrosive medium. Prior to being dipped into the solution, the samples are degreased.

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Fig. 11 shows the results obtained on samples made from C45 treated by different surface engineering processes after a total immersion test lasting 2 weeks and carried out in accordance with the Standard.

In the first horizontal column there is the treatment and the average weight loss per m² and 24 hours on the QPQ sample.

With a weight loss of 0.34 g/m², this sample is much better than the galvanic or chemically coated ones.

In the case of 12 µm hard chrome and even 45 µm double chrome plating, the weight loss was around 7 g/m² and was thereby more than 20 times greater than that of the nitrocarburized samples.

In the age hardened condition, the 20 µm nickel layer showed a weight loss of 2.9 g/m². Only the Triplex layer containing 37 µm copper, 45 µm nickel and 1.3 µm chrome is comparable with the TUFFTRIDE QPQ salt bath treated sample.

Total immersion test (DIN 50905, Part 4) Weight loss of C45 samples treated by various processes after 2 weeks immersion	
Layer or treatment	**Weight loss in g/m² per 24 h**
90 min QPQ	0.34
12 µm Hard chrome	7.10
Double chrome: 20 µm Soft chrome 25 µm Hard chrome	7.20
Nickel: 20 µm Kanigen, age hardened	2.90
Triplex: 37.0 µm Copper 45.0 µm Nickel 1.3 µm Chrome	0.45
Medium: 3 % NaCl, 0.1 % H_2O_2 Material: C45	

Fig. 11

Wear resistance and running properties

Due to the intermetallic composition of the compound layer, the friction and the tendency to weld with a metallic counter-partner are reduced. Excellent sliding and running properties, as well as greater wear resistance, are the well-known advantages of TUFFTRIDE treated components.

Wear tests and practical application repeatedly confirm the superior wear resistance of salt bath nitrocarburized parts over traditional or induction hardened or hard chrome plated surfaces. In very many cases, the wear resistance of the compound layer is improved still further by an oxidative post treatment. For example, components such as transmission shafts, plug gauges and hydraulic aggregates have a longer service life after TUFFTRIDE treatment than after hard chrome plating.

The question is often raised as to the wear resistance of the diffusion layer. Fig. 12 shows a comparison of the wear behaviour of rocker arms treated by two different heat treatment processes. It shows the wear on the running surface of the rocker arm which run against a salt bath nitrocarburized camshaft made from chilled cast iron. Although the surface hardness of the case hardened rocker arm was slightly reduced by nitrocarburizing, the much improved wear resistance due to the presence of the compound layer, to approximately 80 hours running time is clearly visible.
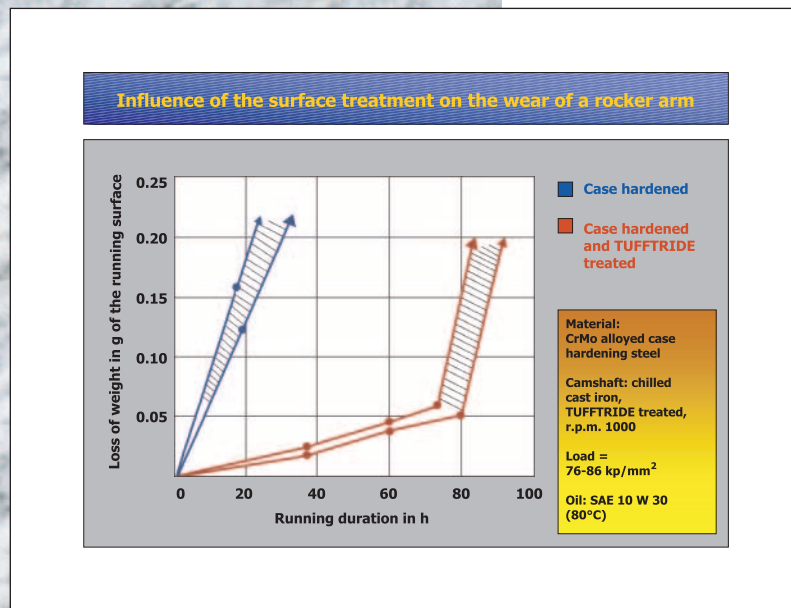
After 70-80 hours, the wear profile then runs parallel to that of the case hardened only rocker arm, which is attributable to the protection given by the diffusion layer. A spontaneous increase in wear after the loss of the compound layer was not observed.

This test again showed very impressively that a high surface hardness does not automatically mean that the protection against wear is also very high. It depends on the respective wear mechanism involved as to how a material or material partnering is to be assessed. Nitrocarburized running partners have proved themselves to be very good under adhesive wear conditions in particular. Their tendency to seize is much lower than that of other surface layers.

Fig. 13 shows the results according to Nieman-Rettig of scuffing load limit tests on gears. These data were established by applying torque to the tooth flank and increasing it until seizure occurred. Nitrocarburizing by the TUFFTRIDE process raised the scuffing load limit of the materials tested by 2-5 times.
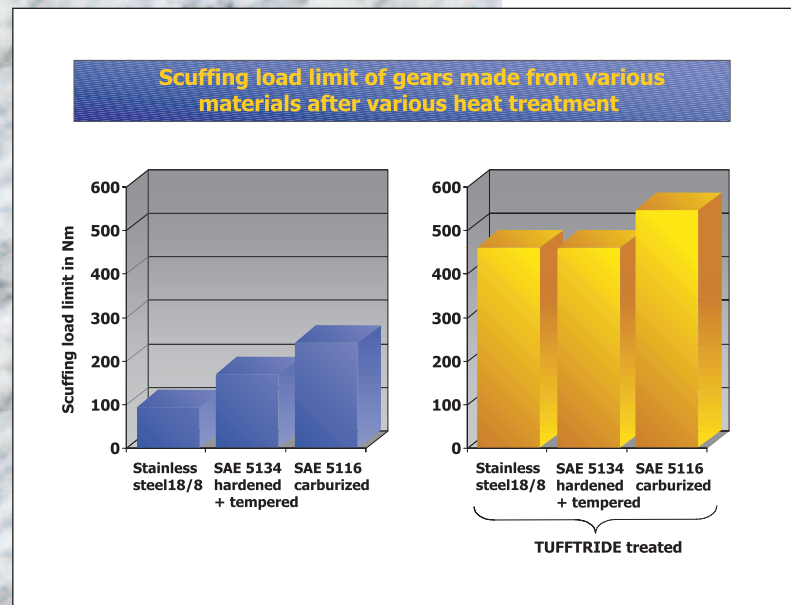

Fig. 12

Fig. 13

Another interesting factor in connection with the wear resistance and running properties is the friction coefficient of the outer surface layer. The interfacial reactions which occur during sliding are not so much determined by the absolute hardness of the running partner but by the material partnering, their microstructural composition, surface geometry and the lubricant used.

To determine the coefficient of friction, tests were carried out in our laboratory on the Amsler machine.

The tests were carried out with one disc running at 200 rpm against another disc which was fixed. Both parts were treated equally. To avoid adhesive wear, a load of 5-30 N was applied. Under greater loads the coefficient of friction increased with the load but in the range of 5-30 N it remained constant.

Fig. 14 gives an overview of the friction coefficient of different pairings under dry running conditions, and after being lubricated with oil, type SAE 30.
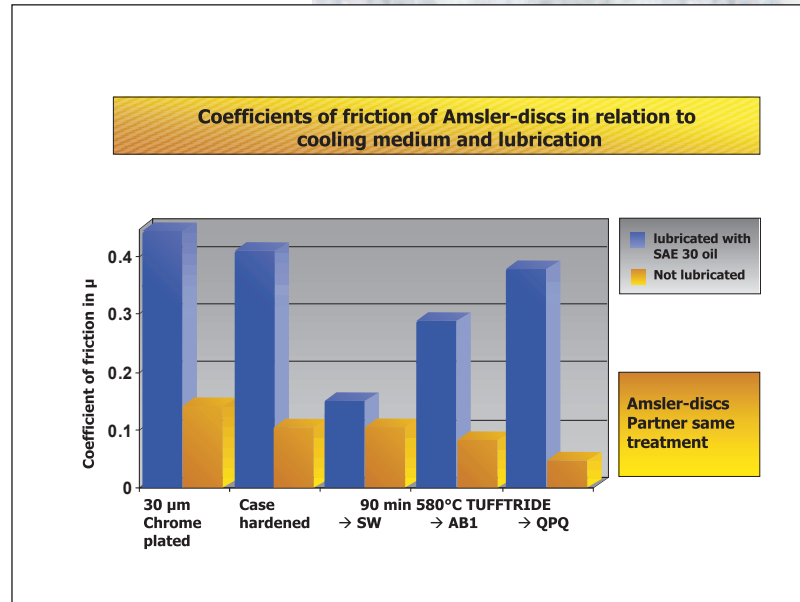

Fig. 14

After hard chrome plating, case hardening, and nitrocarburizing followed by water cooling or oxidative cooling in the AB 1 cooling bath, the samples tested had a surface roughness of around 4 μm. Only the surfaces of the QPQ treated samples were reduced to a surface roughness of Rm = 1 μm by polishing.

Under dry running conditions, nitrocarburized samples have a much lower coefficient of friction than case hardened or hard chrome plated ones. Due to the oxidation of the compound layer, the coefficient of friction of the nitrocarburized samples increases.

In the lubricated condition, the hydrodynamic load supporting film has to be taken into account. With the exception of the QPQ treated samples, there is more solid mass because of the surface roughness so that the results presumably lie within the mixed friction range. Under these test conditions, of all variants the QPQ nitrocarburized samples had the lowest friction coefficient.

The TUFFTRIDE treatment increases the rotating bending fatigue strength and the rolling fatigue strength of components. These are mainly influenced by:

• the level of nitrogen in the compound and diffusion layer,

• the thickness of the diffusion layer and

• the state of solution of the nitrogen on unalloyed steels.

Furthermore, the state of the microstructure and the strength are to be taken into consideration. Whereas with unalloyed steels the increase in fatigue strength is determined by the rate of cooling, with alloyed materials, however, it has no mentionable effect. The increase in fatigue strength possible after 1-2 hours TUFFTRIDE treatment is 100 % on parts made from unalloyed and low alloyed steels.

In this connection we would like to point out that hard chrome plating reduces the rotating bending fatigue strength of the base material. A similar situation prevails with electro galvanizing. Nitrocarburizing, however, always increases the fatigue strength.

Fig. 15 shows the results of a fatigue strength test conducted on notched samples made from material C45N. QPQ treatment increased the fatigue strength by more than 50 %. Hard chrome plating, however, reduced the fatigue strength by 20 %.
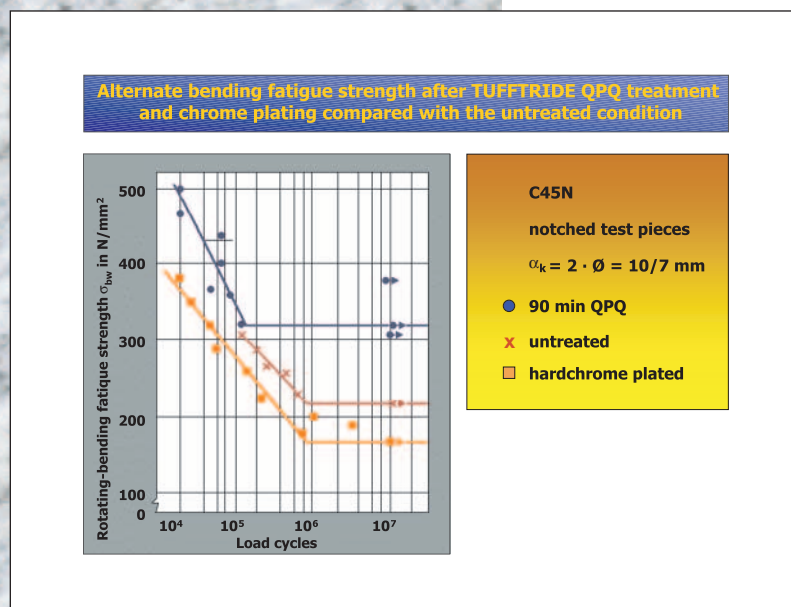

Alternate bending fatigue strength after TUFFTRIDE QPQ treatment and chrome plating compared with the untreated condition

C45N

notched test pieces

$\alpha_k = 2 \cdot \varnothing = 10/7$ mm

● 90 min QPQ

x untreated

□ hardchrome plated

Rotating-bending fatigue strength σ_{bw} in N/mm²

Load cycles

Fig. 15

Practical application of the TUFFTRIDE QPQ process

A variety of methods were used to test the resistance to corrosion and wear, and the fatigue strength of components treated by the environment-friendly QPQ salt bath nitrocarburizing process. These demonstrated the superiority of this process over hard chrome plating, nickel plating and other nitrocarburizing processes.

Fig. 16 shows quick-fit connections for joining hoses for liquid and gaseous media which used to be manufactured from corrosion-resistant steels. By using the QPQ salt bath nitrocarburizing process it was possible to change from the expensive base material to an unalloyed case hardening steel. In the QPQ treated condition the required corrosion and wear resistance is achieved without any problems.

Fig. 17 shows a section of a crankshaft made from material 42CrMo4 for high performance two-stroke engines. The QP treatment gave the parts the required resistance to wear and fatigue, and good running properties.

The components were polished automatically in an overhead rail shot blast machine with metal shot. Tests with gas or plasma nitrocarburized parts did not produce the desired results.

Fig. 16

Fig. 17

Fig. 18

Fig. 19

Fig. 20

Fig. 18 shows gas springs and QPQ treated piston rods. These parts are mainly built into tailgate flaps and engine bonnets of cars, as well as into the baggage flaps and cabin doors of aeroplanes.

It is reported that, compared with the hard chrome plating previously carried out, the corrosion and wear resistance is considerably improved. Furthermore, the cost of treating these parts was reduced by about one third.

Fig. 19 shows small parts such as fastenings for suitcases and hairdryer grilles. These parts are treated in bulk and in very large quantities by the QPQ process. A nitrocarburizing duration of 60 minutes plus oxidative cooling, vibratory polishing and oxidative post treatment are enough to obtain the required corrosion and wear resistance.

Representing the greatly diversified applications of the QPQ process are the typical components in Fig 20, which are nowadays treated in large quantities by the combined salt bath nitrocarburizing process. Most of the parts involved, for example windscreen-wiper shafts, v-belt tighteners and valves, are used in the automotive industry.

The QPQ process is also used for components in the aircraft industry, in off-shore technology, in the construction of plant and machinery, in energy technology, in the food industry as well as in the manufacture of textile machines, hydraulic aggregates and optical equipment.

TUFFTRIDE® plants and their economics

When choosing manufacturing processes, economics, quality and environment-friendliness are the most important criteria. Furthermore, the fact that they should be easy to carry out is of considerable importance. Compared with other nitrocarburizing processes, the TUFFTRIDE process is very easy to carry out. The treatment can be done in manually operated and fully automated plants.

Modern, computer-controlled plants are very versatile. Not only can they be adapted to meet fluctuating production but also permit different programs to run simultaneously.

The automated salt bath plant shown in Fig. 21 is ideally suited for the TUFFTRIDE treatment of mass produced parts in a production line and for commercial heat treating which, due to the different needs of the various customers, requires a heat treating plant to be highly adaptable.

Fig. 22 shows a schematic diagram of a TUFFTRIDE plant which, in accordance with modern technology, is operated effluent-free and which is equipped with an efficient extractor system and exhaust air cleaning plant. With this plant technology the environmental and work place regulations can be complied with in all industrial countries with no difficulty.
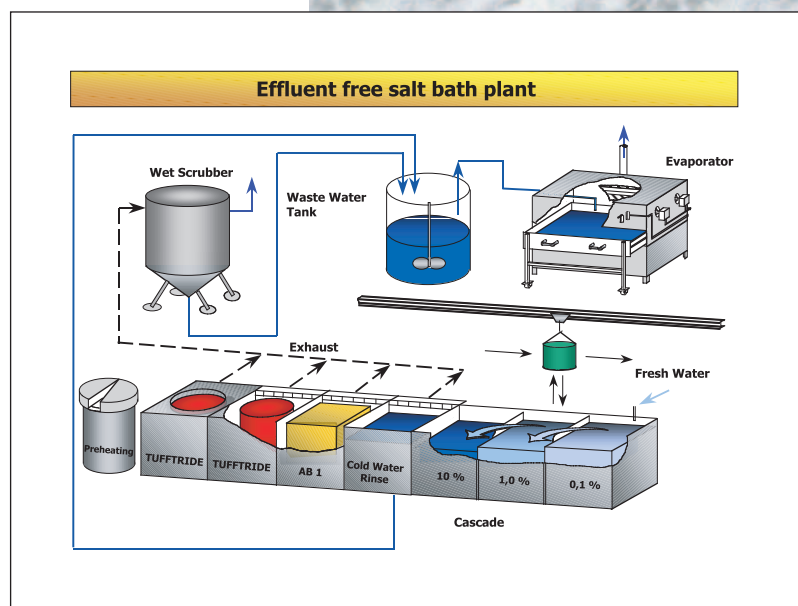

Fig. 21

Effluent free salt bath plant

Fig. 22

14

Cost comparison - Hard chrome vs. TUFFTRIDE QPQ

Cost comparative figure (%)

Legend:
- Hard chrome
- QPQ

Categories: Investment costs, Energy costs, Chemicals consumption, Treating costs
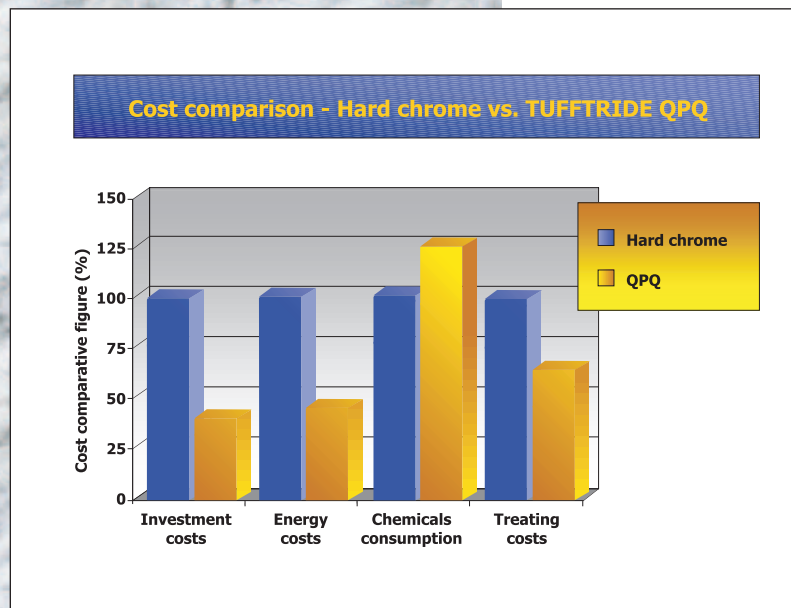
Fig. 23

A comparison of the economics of this process with other surface engineering processes also shows some favourable aspects. Cost comparisons made by various users showed that major savings can be made. As an example, please see the cost comparison in Fig. 23.

Low investment and energy costs also had a very beneficial effect on the economics of the process. A saving of 37 % was possible with the QPQ process over hard chrome plating.

Summary

In addition to the improvements in the properties such as wear protection, fatigue strength and sliding properties, the TUFFTRIDE treatment plus oxidative cooling and/or post treatment produces a major increase in the corrosion resistance. Results of tests and practical applications show that the quality of the treated components is often superior to that of electro galvanic layers and other nitrocarburizing processes. This opens a broad field of application for the TUFFTRIDE process, which can often be accompanied by expensive materials being replaced by lower-cost ones.

Due to the characteristics of the process, such as very good repeatability of high quality results, easy use and high flexibility it is being used to an ever-increasing extent in the metal processing industry all over the world. The process is very easy to carry out and does not require complicated plant technology. The parts can be treated in manually operated and computer-controlled plants. The plant itself operates effluent-free. The process is characterized by its extremely good environment-compatibility. Therefore environmental regulations can easily be complied with.

The TUFFTRIDE QPQ process is known in English-speaking and Asian countries under that name, in Europe and German-speaking countries as TENIFER QPQ and in the USA as MELONITE QPQ. TUFFTRIDE®, TENIFER® and MELONITE® are registered trademarks of Durferrit GmbH.

NOTES: