Dr. Joachim Boßlet

TUFFTRIDE®-/QPQ®-Process

Technical Information
Nitrocarburization in salt melts has firmly established worldwide. Not only the automotive industry employs TUFFTRIDE®/respectively QPQ® treated parts with great success, but also engineering and toolmaking, electronic-, oil- and hydraulic-industry as well as aviation. The key benefits are high wear resistance, fatigue strength and in particular the exceptionally high corrosion resistance. Both processes are to be seen as alternative to case hardening or galvanic processes as well as increasingly to gas- or plasma nitrocarburizing.

The TUFFTRIDE®/QPQ®-Process offers process specific advantages:

- highest temperature constancy
- fast and constant heat transfer
- very stable chemical composition
- shortest treatment cycles
- simple monitoring
- very flexible in use

Remarkable as well is the relative insensitivity towards machining residues on the parts to be treated. Consequently, extensive and costly pre-cleaning processes are not required.

In principle all sorts of ferrous materials, such as austenitic steels, cast iron or sintered material can be nitrocarburized in salt melts. The treatment temperature is in the range of 480 °C and 630 °C. Monitoring simply consists of checking:

- treatment temperature
- treatment time
- chemical composition of salt melt

Compared to other treatment media the salt melt provides exceptionally high nitrogen offer. The nitrocarburizing process immediately starts after immersion into the melt. Already after some minutes the formation of a compound layer can be detected.

Process procedure

Compared to other nitrocarburizing methods, to run this process is very easy. To begin with, the parts are preheated in air to approximate 350 °C. The nitrocarburizing itself is mostly performed at the standard temperature of 580 °C. The treatment time at this temperature is usually 1 to 2 hours.

The active elements in the nitrocarburizing bath are alkali cyanates. During the reaction on the part surface cyanate is transformed to carbonate, whereas the salt bath composition changes slowly. Continuous feed of a polymer regenerator effectuates the recycling of the forming carbonate into active cyanate directly within the melt and keeps the salt bath activity in narrow limits (Fig. 1).

![Principle of Regenerating](image-url)
As the regeneration takes place without change of bath volume, no bail out salts occur. The drag-out losses caused by taking out a treated load are supplemented by replenish salt (TUFFTRIDE® Process). Unlike gas nitriding or gas nitrocarburizing, neither the replenish salt for the nitrocarburizing or the oxidation bath (see below), nor the regenerator are classified as toxic or are harmful to the environment.

The oxidation treatment is performed after salt bath nitrocarburization in an especially developed cooling bath at a temperature range of 370 - 430 °C. During the treatment a black iron oxide layer (magnetite) is produced on the surface of the treated parts, which enhances substantially the corrosion resistance. Apart from the oxidative effect, the dimensional stability of the quenched component is positively influenced.

In case the corrosion resistance doesn’t play a decisive role, components and/or tools can, pending on the risk of cracking or distortion, either be cooled directly in water, by means of air blower, under nitrogen or in vacuum. Nowadays, the oil quench is also, regarded under safety aspects, rather questionable and not required any more.

Thereafter, the treated material is furthermore cooled to room temperature as well as cleaned within a well tempered and agitated washing cascade.

Is the surface roughness after nitrocarburization too high, pending on the seize and the shape of the parts, various polishing methods can be used.

- Lapping with emery cloth grade 360 or finer;
- Polishing or continuous micro-finishing with special polishing discs with the throughfeed method, similar to centreless polishing, or on an automated lathe fixed between centre pieces;
- Vibratory finishing; this treatment is primarily used for small and sheet metal components;
- Blasting with glass beads size 40 - 70 µm in diameter; to prevent edges being excessively rounded, or removal of the compound layer being avoided, the pressure should not exceed 4 bar;
- Automated blasting with metal beads whose diameter should be less than 1 mm.

Intermediate treatment, however, may partly reduce the gained corrosion resistance. For this reason, in many cases a second oxidative treatment is carried out. This complete sequence is shown in Fig. 2 for the QPQ® Process. QPQ® comprises TUFFTRIDE® treatment with oxidative cooling, mechanical processing and oxidative post treatment, using the same salt melt for both oxidative steps.
Composition and thickness of the nitride layer

During the TUFFTRIDE®-process a nitrocarburized layer is formed consisting of the outer compound layer ($\varepsilon$-iron nitride) and the diffusion layer thereunder. The formation, microstructure and properties of the compound layer are significant reliant on the base material. The formation, structure and properties of the compound layer are very much depending on the used basic material. Apart from iron some alloying elements, such as Cr, Mo, Al, V, Ti or W, are able to react with nitrogen. The so-called specia nitrides are formed within the compound layer as well as within the diffusion layer.

Compound layer

While diffusing atomic nitrogen the compound layer is formed. With rising nitrogen absorption the solubility limit of the boundary layer is exceeded and forms iron nitrides, in case of alloyed steel, additionally special nitrides and builds a closed compound layer. Depending on the nitrogen content it contains either $\varepsilon$-iron nitrides, $\gamma'$-iron nitride or a mixture of both. Compared to the classical nitriding the nitrocarburization enriches a small quantity of carbon in the compound layer and strictly speaking iron carbon nitrides are formed. Because TUFFTRIDE® posses by far most nitrogen offer the compound layers are almost monophase existing of $\varepsilon$-iron carbon nitride. Depending on the material used, the compound layer will have a Vickers hardness of about 800 to 1500 HV measured in the cross section.

The compound layer divides in a compact part and a porous part which is situated directly at the surface. The latter is also called pore zone. It is used as lubricant reservoir and supports the good dry-running operation properties of the treated parts. During oxidizing cooling the pores are almost filled with magnetite, which anchors optimal the protecting oxide layer. Same time, the residual compressive stress in the boundary area increases. 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 the obtainable layer. Although the layer growth is lower, the content of alloy is higher, the hardness however increases to an equal extent. Fig. 3 shows the determined correlation at a tempera-

![Compound layer thickness after TUFFTRIDE® treatment](image)

**Fig. 3**

- Mild steel
- QT steel
- Hot work steel
- 12 % Cr-steel
- Cast iron

<table>
<thead>
<tr>
<th>Compound layer thickness in $\mu$m</th>
</tr>
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<tr>
<td>Treatment time in h</td>
</tr>
<tr>
<td>0.5</td>
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<tr>
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<td>2</td>
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</tr>
<tr>
<td>4</td>
</tr>
<tr>
<td>5</td>
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</table>

Composition and thickness of the nitride layer
ture of 580 °C. With the usual treatment time of 60 to 120 minutes a compound layer of 10 - 20 µm is obtained on most qualities of material.

The metallographic analysis defines clearly the compound layer from the total layer as a slightly etched zone, followed by the diffusion layer (see Fig. 4).

**Diffusion layer**

The area below the compound layer is called diffusion layer. Due to the concentration decline from the edge to the core, the nitrogen content is not sufficient to form iron nitride. Obtainable depth and hardness of diffusion layer are dependant on the material.

In case of unalloyed steels, the crystalline structure of the diffusion layer is influenced by the rate of cooling after nitrocarburizing. After 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 first mentioned case a higher hardness is achieved, in the second one the ductility is increased.

In contrast, cooling has no noteworthy influence on the formation of the diffusion layer at alloyed steels. Nitrogen has already been precipitated as special nitride (i.e. chromium-, aluminium- or vanadium-nitride). Therefore, part of the diffusion layer can better be identified metallographically from the core structure, due to the improved etchability. The actual nitrogen penetration depth is pending on the content of alloying elements considerably higher than the visible darker etched area. **Fig. 5** shows the nitriding hardness depth according to DIN 50190-3 on various materials against treatment time.
Surface hardness and core 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. 7 gives reference values of core hardness and surface hardness for different steels.

Corrosion resistance

With upward trend the nitrocarburization is used for improvement of corrosion resistance of parts made from unalloyed steels. As well as for corrosion resistance it is important to produce preferably monophase ε-compound layers. Furthermore, there are two more factors of importance: on the one hand a sufficient thick ε-nitride layer of minimum 12 µm thickness and on the other hand the black iron oxide (magnetite), which is formed during the oxidizing cooling at the outer edge layer and within the pores.

The stress combination of corrosion and wear happens in practice quite often. Almost everywhere, where motional processes with corrosive strains take place, the use of nitrocarburization with oxidizing cooling unfolds. In case the demand system asks for softer running partners with lower surface finish, lapping or polishing is done to adjust the desired surface finish. In this connection it is important to remove as little as possible. This shall ensure that besides the sufficient thick compound layer as well parts of the pore seem remain preserved, to be able to obtain an optimal formation of the oxide layer as well as consistent dark coloring of the surface during the subsequent oxidizing finishing treatment (QPQ® Process). Remarkable is the fact, that the roughness of the polished surface hardly changes during second oxidation.

Improvement of component properties

Table: Improvement of component properties

<table>
<thead>
<tr>
<th>Component characteristics</th>
<th>main influence</th>
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<tr>
<td></td>
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<tr>
<td>wear resistance</td>
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CL  Compound Layer  DL  Diffusion Layer

Fig. 6
check parts in a salt mist of 5% sodium chloride at 35 °C. Even after a test period of 500 hours there were no corrosion attacks visible on the functional surface of the QPQ® treated piston rods. As well as after TUFFTRIDE®-treatment (incl. oxidizing cooling) a clear increasing of corrosion resistance on components is observed. Pending on the part geometry and its surface finish a holding time of up to 200 hours and more are possible in the salt spray test. In principal the corrosion resistance increases with declining surface roughness.

As test criterion the salt spray test according to DIN EN ISO 9227:2006 NSS was chosen to check parts in a salt mist of 5% sodium chloride at 35 °C. Even after a test period of 500 hours there were no corrosion attacks visible on the functional surface of the QPQ® treated piston rods.

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**Fig. 8** shows an overview in corrosion resistance of various galvanic layers compared to a QPQ®-layer.

<table>
<thead>
<tr>
<th>Name</th>
<th>Material number</th>
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<td>1500 - 1700</td>
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</tbody>
</table>

**Fig. 7**

**Fig. 8** shows an overview in corrosion resistance of various galvanic layers compared to a QPQ®-layer.
Even under these conditions a QPQ® treatment results in considerable better corrosion resistance. The first of the samples showed a corrosion attack after 92 hours and the last, after 159 hours. The average resistance was 114 hours. However, the chrome plated piston rods failed completely after 21 hours.

For the total immersion test according to DIN 50905-4 a solution of 3 % sodium chloride and 0.1 % hydrogen peroxide (H₂O₂) is used. Prior to being dipped into the solution, the samples are degreased. Fig. 10 shows the results obtained on samples made from C45 treated by different surface engineering processes after a test lasting 2 weeks.

With an average weight loss of 0.4 g/m² per 24 hours, the QPQ® treated samples were much better than the galvanic or chemically coated ones. In case of 12 µm hard chrome and even 45 µm double chrome plating, the weight loss was 20 times exceeded than that of the nitrocarburized samples. Only the Triplex layer containing 37 µm copper as well as 45 µm nickel and 1.3 µm chrome is comparable with the QPQ® treated samples.
Wear resistance and running properties

Excellent sliding and running properties, as well as high wear resistance, are the well-known advantages of TUFFTRIDE®-treated components. Due to the intermetallic composition of the compound layer, the friction and the tendency to weld with a metallic counterpart are reduced. Compared to case hardening the nitride layer possess a much better heat resistance. The gained hardness increase within the diffusion layer remains the same even at higher temperatures.

Many wear tests and practical applications confirm the advantages to other surface layers. Structure and composition of the compound layer (proportion N and C) influence the wear resistance considerably. Monophase carbon enriched ε-compound layers, which exist after TUFFTRIDE® treatment, gain very good results. Layers with low carbon or having high γ'-proportion were in most cases definitely worse.

In the outer zone of compound layer occurring porosity is no evidence for poor wear properties. In fact, better adhesion of the oil film can often lead to more beneficial running properties. Fig. 11 shows a gear made of C45N after service life of 700,000 km in a commercial vehicle. The removal by wear from the upmost monophase ε-compound layer was only 1 - 2 µm. Even the pore zone is still clearly visible.
Fig. 12 allows conclusions about the relative wear resistance of surface layers after hardening, nitrocarburization and boriding under adhesive stress. Although the borided samples had an approx. 1000 Vickers higher surface hardness as the TUFFTRIDE® treated ones, the latter had with factor 800 higher wear resistance and still 80-fold higher than the hardened samples. This investigation makes it clear that the hardness of the surface layer is not the only influencing factor in respect of wear properties.

Also scuffing is significantly reduced compared to other surface layers. Fig. 13 shows the results according to Niemann-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.

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 pairing, their microstructural composition, surface geometry and the lubricant used.

To determine the coefficient of friction, tests were performed on the Amsler machine. Trials were carried out with a disc running at 200 rpm against another disc being 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.

The tested samples 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. 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.

![Adhesive wear of different layers](source: Habig, BAM)
Under these test conditions, all variants the QPQ® nitrocarburized samples had the lowest friction coefficient.

The TUFFTRIDE® treatment increases the rotating bending fatigue strength as well as the rolling fatigue strength. These are mainly influenced by:

- the level of nitrogen in the compound and diffusion layer
- the thickness of the diffusion layer
- the state of solution of the nitrogen on unalloyed steels

Furthermore, the microstructure and the strength of the base material 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, it has no noticeable effect. An increase in fatigue strength is possible after 1 - 2 hours TUFFTRIDE® treatment of 100 % on notched parts made from unalloyed steels. With alloyed steels a general improvement of 30 to 80 % can be obtained.
Applications

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 is noted for zinc plating. 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%.

Industrial Application of the TUFFTRIDE®-/QPQ®-process

Beside the conventional applications, improving the wear and fatigue resistance by nitrocarburizing in salt melts, the corrosion protection gains more and more interest. Increasingly, the TUFFTRIDE® treatment in combination with oxidative post treatment respectively the QPQ® treatment is used as substitution for galvanic coating processes such as hard chrome plating, nickel plating, zinc plating etc., or used as substitution of corrosion resistant steels. Subsequent, there are given representative applications.

Tools made of hot working steel for extrusion (see Fig. 16), forging or die-casting achieve much better service life results after TUFFTRIDE® treatment. Reasoned by the non-metallic character of the compound layer the functional surface remains smooth for much longer. The affinity of adhesion is minimized and the metallic make-up is virtually avoided. User report that, compared to plasma- or gas nitrided resp. nitrocarburized extrusion dies, significantly better press performances are achieved and that tools can even be re-treated several times. Especially, the risk of chipping is considerably reduced. A further advantage are the short treatment times. The tools are much quicker ready for use. This also results in remarkable savings in tooling costs. As well as injection molds for plastic materials are successfully salt bath nitrocarburized.

Valves in combustion engines (see Fig. 17) are parts having to stand high standards in respect of thermal capacity, wear resistance and corrosion resistance. Compared to hard chrome plating the TUFFTRIDE® process offers cost savings to manufacturing costs, because inductive hardening and final grinding is not necessary. Furthermore, the necessity to manufacture the exhaust valves from inductive hardenable steel is not applicable. It can rather be produced from heat resisting austenitic steel.
The treatment times are, depending on the specification between 20 and 90 minutes. Pending on the plant seize batches vary between 2500 and 4000 pieces. This way, a productivity of less than 1 s per valve is realized. On the basis of short treatment times there are no big buffer capacities necessary even at changing geometries, steel grades or specifications.

For highly stressed 4 stroke engines, met in motorbikes respectively sports car industry, as well as two stroke engines for small planes or snowmobiles, the crank shafts and cam shafts are treated with the TUFFTRIDE® process. Despite or just because of the clearly visible pore seam in the compound layer the high demands are fulfilled without any problems. The pore zone facilitates the running-in wear behavior and offers good dry-running properties due to the micro lubrication slots effect. As well for big diesel engines for SUVs or commercial vehicles the crank shafts, tappets or steering wheels are nitrocarburized in big quantities in salt melts. Further applications are gear and differential parts.

The new generation of engines, which can alternatively be run by biofuel, the TUFFTRIDE® layer offers superior wear protection properties. In the meantime, more than 250 million valves per year with upward trend are treated in salt melts.
In the motorcycle industry also various TUFFTRIDE® respective QPQ® treated engine and power transmission parts are in use. Fig. 18 shows parts with better corrosion resistance than gas or plasma nitrocarburized ones. Owing to the short treatment time and the high flexibility there is given an optimal integration into the production flow with the according cost advantage.

The QPQ®-process finds constant growing application for piston rods, hydraulic cylinders or bushings. As material mild steel, unalloyed or low alloyed steel is used. The required testing time in the salt spray test is mostly 144 hours without corrosion point. In some cases there are even 400 hours required, which are also reached successfully.

Fig. 19 shows gas spring piston rods, which are utilized in the automobile and aircraft industry, for engineering or in office chairs. By substitution of the chrome layer considerable cost reductions were achieved. The QPQ® treatment is performed in fully automated plants. The combination of up to 4 nitrocarburizing furnaces in one treatment line allows cycle times of 0.5 - 0.6 s per piston rod.

The examples could be endlessly continued. The TUFFTRIDE®-/QPQ® process is also used for components in the aircraft, in the offshore technology, in the plant and machine construction, in energy technology, in the food industry, photo and computer industry as well as in the manufacture of textile machines or hydraulic aggregates.

**Applications**

Drive and clutch components for motor bikes

Piston rods for gas springs and dampers

Fig. 18  Fig. 19
To run the TUFFTRIDE®/QPQ® process it is easy compared to other nitrocarburizing processes, because there is no complicated plant technology required. The treatment can be done both manually and in fully automated plants. They are arranged in line in a modular concept (please see Fig. 23) and are consequently advanced under the aspect of

- **reduction of energy consumption**
- **process security**
- **operator friendliness**

Fig. 20 shows a nitrocarburization furnace of the newest generation. It has a pneumatic closeable, isolated lid and offers considerable energy savings at base load operation. Furthermore, this furnace is equipped with external placed, continuous working filtration device for cleaning the salt melt and also with a feed unit for regeneration.

**New generation of energy saving furnaces**

**Features:**

- Pneumatic closable lid
- TENOCLEAN® filtration device
- Regenerator dosing device
For hand operated plants also electronic systems are available, which record all relevant parameters and assort them to a batch documentary. Compared to the gaseous processes an online monitoring of the chemical parameters is not required due to the high stability of the melt. Daily analysis are allocated to the treated batches via computer.

Sometimes the remaining salt residues on the components are criticized but they only arise from insufficient cleaning equipment.

State of the art are meantime three to four step, heated and agitated washing cascades (please see down right of Fig. 23), which perform not only very good washing results, but allow considerable water savings.

**Fig. 21** shows a microprocessor monitored capsuled plant. Batches with different treatment parameters are operated by computer controlled guidance with a special software to guarantee an optimal batch flow. Big sliding doors allow easy access to the individual plant components. The feeding of the refill salt respective regenerator salt is done by according channels from outside.

**Fig. 22** shows exemplary the diagram for the control of the nitrocarburizing furnace.

Computer controlled and multi-purpose TUFFTRIDE® plant
Control of the TUFFTRIDE® furnace via PC

Effluent free TUFFTRIDE® plant
Environmental aspects

Against overwhelming prejudices, the authorization of a new plant "in the green" is not more complicated than for other nitrocarburizing processes. Without any problem the effective environmental as well as work place regulations will be met. The relevant plant components are equipped with an efficient exhaust device. Even although in most cases the legitimate allowance for air cleaning procedures is met the exhaust air is lead through a dry filter device or if applicable through a wet scrubber device, to respect environmental aspects (see Fig. 23).

In the year 2000 an ecological assessment of salt nitrocarburizing and gas nitrocarburizing was established. Failing sufficient provided data the originally planed plasma process could not be considered. For the study all for the process comprising energy and mass flow had been taken into account and set in relation to the quantity of treated goods. The evaluation had been effected by giving "harm points" for the material and energy input as well as for exhausts, waste water and wastage. This was done by consulting criterion of the Federal Office for Environment/Berlin. Fig. 25 shows the results of this comparison. It reflects that the salt bath nitrocarburizing process from ecological point of view tends to result in a better valuation than gas nitrocarburizing.

Ecobalance on Nitrocarburizing

<table>
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<th>Process</th>
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<tr>
<td>Gas furnace 3.6 m³</td>
<td>1000</td>
</tr>
<tr>
<td>Salt bath electr. heated</td>
<td>3000</td>
</tr>
<tr>
<td>Salt bath gas heated</td>
<td>4000</td>
</tr>
</tbody>
</table>

Evaluation contains contamination through:
- Deposition space
- Acidification
- Nutrient enrichment
- Form. of photo oxidants
- Effect on ecotoxicity
- Effects on health
- Ozone degradation
- Greenhouse effect
- Resource Consumption

Source: J. Buchgeister
Summary

In addition to the significant improvement of wear protection, fatigue strength and sliding properties, the TUFFTRIDE® treatment plus oxidative cooling or QPQ® treatment produces a major increase in corrosion resistance. Results of tests and industrial 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, in which expensive materials can often be substituted. Due to the characteristics of the process, such as

- excellent reproducibility, on high quality level
- shortest treatment times
- most negligible distortion
- high flexibility

The TUFFTRIDE®/QPQ® is the most widely used nitrocarburizing process. The process is very easy to carry out and does not require complicated plant technology. Electronic monitoring and documentation of the process sequence up to automated procedure, efficient devices for filtering the melt as well as for subsequent cleaning of the treated parts are state of the art today. The plant itself is run effluent-free. Environmental specifications can be complied without difficulty.

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