OPTIMIZED HEAT TREATMENT AND NITRIDING PARAMETERS FOR A NEW HOT-WORK TOOL STEEL

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Abstract
Thyrotherm 2999 EFS SUPRA is a new hot-work tool steel designed for forging tools, which are exposed to intensive wear. Industrial application tests demonstrated the necessity to optimize the heat treatment recommendations for such tools. The study describes the influence of the austenitizing temperature on the hardness and ductility of the steel, the influence of the hardened cross section and of the quenching medium on the ductility of Thyrotherm 2999 EFS SUPRA. The results of the investigations allowed to derive the conclusion that an optimum balance of hardness and ductility can be achieved if tools are hardened from 1100 °C.

Nitriding is a very popular technique to prevent surfaces of tools against wear. The specific chemical composition of Thyrotherm 2999 EFS SUPRA requires an adaptation of the nitriding parameters in order to avoid a drastic embrittlement. The report gives a survey on the nitriding behavior of Thyrotherm 2999 EFS SUPRA.

Keywords: Hot-work tool steel, forging tool, heat treatment, austenitization, carbide solution, hardness, ductility, nitriding.

INTRODUCTION
The hot-work tool steel Thyrotherm 2999 EFS SUPRA has been developed for hot-forming applications, which impose extreme mechanical and thermal impacts on the tools. Among the steel’s outstanding properties, which have been described before [1] the steel’s hot-strength, wear resis-
tance and thermal conductivity are of great benefit for a high productivity of forging dies. The excellent wear resistance of Thyrotherm 2999 EFS SUPRA, first described in an independent study of the University of Hannover [2, 3], Germany, encouraged various customers in the forging industry to conduct application tests. The results gained in these application tests will be presented in another paper on this conference [4].

The results of the industrial application trials clearly demonstrated that an optimization of the heat treatment recommendations for tools as well as a profound knowledge of the nitriding behavior of Thyrotherm 2999 EFS SUPRA would further improve the performance of hot forming tools. The results of these optimizations will be described here.

**FAILURE ANALYSIS ON DEFECT TOOLS**

A forging die for pliers, made of Thyrotherm 2999, was hardened and tempered to 47–48 HRC and finally plasma nitrided (nitriding depth 0.30 mm). During operation it failed after only 10 forging strokes. Metallographic examinations of the tool revealed the required hardened and tempered martensitic microstructure (Fig. 1). Also there was a high content of undissolved carbides visible within the grains and on the grain boundaries.

A second example is a press forging die made of Thyrotherm 2999 EFS SUPRA used to forge titanium fasteners (Fig. 2). The die was hardened and tempered to 46 HRC and finally nitrided. It failed after approximately 700 strokes. In contrast to the tool described before its microstructure consisted of a homogeneous martensite, which was free of undissolved carbides and carbide precipitations on grain boundaries. But the metallographic examinations revealed an intensively nitrided surface with an average thickness of the compound layer of 12 µm, which was partially spalled. In the diffusion zone of approx. 0.30 mm thickness a very intensive network of precipitations on the grain boundaries was detected.

These examples demonstrate two negative influences on the toughness of tools: a high content of undissolved carbides as well as an intensively nitrided surface. The desired improvements in the performance of the tools required optimized heat treatment and nitriding recommendations for tools of Thyrotherm 2999 EFS SUPRA.
INVESTIGATIONS

IMPROVEMENT OF HEAT-TREATMENT

The intention of this investigation was the optimization of the heat-treatment parameters for forging tools of Thyrotherm 2999 EFS SUPRA with respect to a wellbalanced relation of hardness and ductility. To a high degree these
properties are controlled by the heat-treatment, especially by the solution of carbides during austenitization.

First the hardening and tempering behavior of Thyrotherm 2999 EFS SUPRA was studied. The chemical composition of Thyrotherm 2999 EFS SUPRA is listed in Table 1. Samples were hardened in a salt bath from temperature between 1075 and 1200 °C for 15 min / oil and double tempered in the temperature range from 400 °C to 700 °C.

In the second step it was intended to achieve results, which can easily be transferred to industrial applications. Therefore the experiments were conducted on steel bodies with cross sections of 60 mm × 60 mm, 100 mm × 100 mm, 200 mm × 200 mm and 240 mm length each. These bodies of Thyrotherm 2999 EFS SUPRA were hardened according to the parameters listed in Table 2 and double tempered to 46 HRC. Unnotched impact bending samples (dim. 7 mm × 10 mm × 55 mm) were then taken from the center region of these bodies in longitudinal and transverse directions (Fig. 3). Additionally a slice was taken from each body for hardness measurements and metallographic examinations.

Hardness profiles over the cross sections of the bodies give information about the through-hardenability. Metallographic examinations were conducted on a slice located between longitudinal and transverse impact bending samples. The impact bending samples were tested at room temperature.

The investigation is focused on these influences:

- The influence of austenitizing temperature
Table 2. Heat treatment parameters for bodies of Thyrotherm 2999 EFS SUPRA (M.T. = Martempering)

<table>
<thead>
<tr>
<th>Studied influence of</th>
<th>Body dim. in mm</th>
<th>Hardening</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardening temperature</td>
<td>60 mm × 60 mm</td>
<td>1075 °C/30 min / N₂ (6 bar)</td>
<td></td>
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<tr>
<td></td>
<td>1100 °C/30 min / N₂ (6 bar)</td>
<td></td>
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<td></td>
<td>1125 °C/30 min / N₂ (6 bar)</td>
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<td></td>
<td>1150 °C/30 min / N₂ (6 bar)</td>
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<tr>
<td></td>
<td>1175 °C/30 min / N₂ (6 bar)</td>
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</tr>
<tr>
<td></td>
<td>1200 °C/30 min / N₂ (6 bar)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hardened cross section</td>
<td>60 mm × 60 mm</td>
<td>1100 °C/30 min / N₂ (6 bar)</td>
<td>Vacuum</td>
</tr>
<tr>
<td></td>
<td>100 mm × 100 mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>200 mm × 200 mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quenching medium</td>
<td>60 mm × 60 mm</td>
<td>1100 °C/30 min / N₂ (1 bar)</td>
<td>Vacuum</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1100 °C/30 min / N₂ (3 bar)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1100 °C/30 min / N₂ (6 bar)</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>1100 °C/30 min / M.T. 180 °C/air</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1100 °C/30 min / M.T. 540 °C/air</td>
<td>Salt bath</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Quenching</td>
</tr>
</tbody>
</table>

- The influence of the hardened cross section
- The influence of the quenching medium

on hardenability, tempering response, through-hardenability, carbide solution, grain size, and ductility.

**PARAMETER STUDY ON NITRIDING RESPONSE**

The steel compositions in Table 1 demonstrate that Thyrotherm 2999 EFS SUPRA can be distinguished from other hot-work tool steels by its high content of molybdenum. This element is rather reactive with nitrogen. As the results of the industrial trials clearly pointed out the parameters of the nitriding processes have to be adapted to the steel’s chemical composition. In this part of the investigation the nitriding behavior of Thyrotherm 2999 EFS SUPRA was therefore studied in comparison to the well-known hot-

work tool steels 1.2344 and 1.2367. The chemical compositions of these steels are also given in Table 1.

For this investigation oversized unnotched impact bending samples (dim. 8 mm × 12 mm × 55 mm) were taken from these steels in longitudinal direction, hardened and tempered to 46 or 50 HRC respectively and then nitrided according to Table 3. These samples were then grinded on three of their longitudinal sides to their final dimension so that one side remained completely nitrided. In the impact bending tests the samples were positioned in such a way that the nitrided side was exposed to tension on the impact of the pendulum hammer (Fig. 4).

In metallographic examinations the constitution of the nitrided layers was examined and micro-hardness profiles were measured.
RESULTS OF INVESTIGATIONS

IMPROVEMENT OF HEAT TREATMENT

The first study focused the hardenability of Thyrotherm 2999 EFS SUPRA. The correlation of hardness and hardening temperature for Thyrotherm 2999 EFS SUPRA is shown in Fig. 5. The highest hardness – 62 HRC – can be achieved after hardening from 1200 °C. The corresponding tempering curves indicate that an increasing austenitizing temperature raises the secondary hardness maximum to a certain extent so that the maximum secondary hardness is 62 HRC.

The austenitizing temperature directly influences the steel’s microstructure (Fig. 6). On the one hand increasing hardening temperatures directly reduce the amount of undissolved carbides, on the other hand temperatures above 1125 °C cause a rapid grain growth.

The influence of the hardening temperature on the ductility was determined on steel bodies of the dim. 60 mm × 60 mm × 240 mm, which were heat treated to 46 HRC. All bodies investigated revealed even hardness profiles over their cross sections. Increasing the hardening temperature from
1075 to 1100 °C improves both the ductility of longitudinal and transverse samples and thus also the isotropy of the steel (Fig. 7). A further increase of the hardening temperature to 1125 °C starts to impair the ductility again. Higher hardening temperatures then reduce the ductility dramatically. The progressing carbide solution with rising hardening temperature and the improvement of ductility coincide. The improvement of ductility is directly related to the decreasing carbide content whereas the later dramatic loss of ductility is due to the intensive grain growth. This fact can easily be seen in the fractures of tested impact bending samples (Fig. 8). Samples hardened from temperatures well above 1125 °C reveal an intergranular fracture with a very coarse structure. It can be concluded that a hardening temperature of 1100 °C offers the best balance of hardness and ductility and should therefore not be exceeded.

In the following studies all samples were hardened from 1100 °C. In order to determine the influence of the hardened cross section on the ductility steel bodies with the cross sections 60 mm × 60 mm, 100 mm × 100 mm, and 200 mm × 200 mm were vacuum-hardened and tempered to 46 HRC (Table 2). The ductility in the core of the bodies decreases with growing dimensions of the bodies (Fig. 9). All bodies achieved even hardness profiles without any significant loss of hardness in the center of the larger bodies (Fig. 10). Significant differences in the microstructure of the bodies could...
not be found. They all revealed a martensitic microstructure without any indications of a bainitic microstructure.
The quenching medium directly controls the cooling rate of a tool, which is being hardened, and can thus influence the mechanical properties of the steel. Today’s vacuum furnaces offer a great variety of quenching facilities. In most cases pressurized nitrogen gas is used as quenching medium and the gas pressure controlling the cooling rate. In this study bodies of 60 mm × 60 mm were vacuum hardened with nitrogen pressure of 1, 3, and 6 bar. Additionally two bodies were austenitized in a salt bath and martempered in a salt bath of 180 or 540 °C respectively. The results of the impact bending tests (Fig. 11) show that among the three vacuum hardened bodies the ductility
Figure 9. Influence of the cross-section on the ductility of Thyrotherm 2999 EFS SUPRA.

Figure 10. Hardness profiles on cross-sections of heat treated steel bodies of Thyrotherm 2999 EFS SUPRA.

varies only slightly. The fact that lower gas pressures lead to similar results can be contributed to the size of the three bodies. Although a martempering
in a 180 °C salt bath gave higher ductility values than the 540 °C salt bath. It should be restricted to experiments as it might create high tensions, which could destroy the tool.

Figure 11. Influence of the quenching medium on the ductility of Thyrotherm 2999 EFS SUPRA.

NITRIDING PARAMETERS

The nitriding behavior of Thyrotherm 2999 EFS SUPRA was studied in comparison to the hot-work tool steels 1.2344 and 1.2367. The results of impact bending tests conducted on gas nitrided samples are shown in Fig. 12. The nitriding treatment drastically lowers the ductility of these three steels. A prolonged process time even enforces this embrittlement. It is obvious that the embrittlement of samples with a hardness of 50 HRC is more intensive than that of samples with 46 HRC. The figure also demonstrates that the embrittlement is directly related to the total nitriding depth, which increases with the process time.

The evaluation of the impact bending tests of salt bath nitried samples leads to a similar result (Fig. 13). Here – as well as in gas nitrided samples – Thyrotherm 2999 EFS SUPRA shows the most intensive reaction with nitrogen, which has to be respected in tool design.

Fig. 14 displays characteristic photomicrographs of the gas nitrided samples. All samples reveal a small white compound layer, which grows with increasing process time. Differences between the three steels with respect to
Optimized Heat Treatment and Nitriding Parameters for a New Hot-work Tool Steel

the thickness of the compound layer were not seen. Parallel to the nitrided surface the steels develop fine precipitations on grain boundaries, which are increasing with the nitriding time. As the micrographs do not show significant differences of these steels further investigations are required.

Differences in the nitriding behavior of the three steels can be seen in Fig. 15, which gives an idea of the microstructures after salt bath nitriding. They all develop precipitations on the grain boundaries parallel to the nitrided surface but the extension of the precipitation zone is largest in Thyrotherm 2999 EFS SUPRA. Precipitations after a 3-hour salt bath treatment are larger than after 20 hours of gas nitriding. These micrographs explain the low ductility values.

Characteristic hardness profiles of gas and salt bath nitrided samples are shown in Fig. 16. Essential differences between the three steels cannot be seen in these graphics. This explains that the extreme embrittlement of Thyrotherm 2999 EFS SUPRA is directly related to the formation of precipitations on grain boundaries.

CONCLUSIONS

Investigations of the hardening and tempering behavior of Thyrotherm 2999 EFS SUPRA come to the conclusion that an optimum balance of hardness and ductility can be achieved after hardening from 1100 °C as this austenitizing temperature guarantees a sufficient carbide solution. Higher temperatures should be avoided as they cause a rapid grain growth, which drastically reduces the ductility. An influence of the hardened cross-sections on the through-hardenability and ductility could not be seen in these experiments.

Nitriding is a common method to protect surfaces of tools against wear. This treatment generally reduces the ductility of a hot-work tool steel. Due to its high content of molybdenum Thyrotherm 2999 EFS SUPRA intensively reacts with nitrogen. Comparisons of microstructures of nitrided samples showed that Thyrotherm 2999 EFS SUPRA tends more to form precipitations than other hot-work tool steels (e.g. 1.2344 and 1.2367) do. These precipitations – preferably aligned parallel to the nitrided surface – drastically reduce the ductility and should therefore be kept on a minimum. As Thyrotherm 2999 EFS SUPRA has an excellent "natural" wear resistance [2, 3, 4] the necessity of nitriding should always be considered carefully. As described in [4] tools of Thyrotherm 2999 EFS SUPRA can reveal a better
performance as those of other, nitrided hot-work tool steels. If nevertheless a nitriding treatment of a tool of Thyrotherm 2999 EFS SUPRA seems to be unavoidable the process should be controlled carefully and the formation of precipitations in the nitrided layer be kept at an absolute minimum.

REFERENCES


Figure 12. Ductility and total nitriding depth of gas nitrided samples of three hot-work tool steels.
Figure 13. Ductility and total nitriding depth of salt bath nitrided samples of three hot-work tool steels.
**Optimized Heat Treatment and Nitriding Parameters for a New Hot-work Tool Steel**

<table>
<thead>
<tr>
<th>Nitriding time: 4h</th>
<th>20h</th>
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<tbody>
<tr>
<td>1.2344 50 HRC</td>
<td></td>
</tr>
<tr>
<td>1.2367 50 HRC</td>
<td></td>
</tr>
<tr>
<td>Thyrotherm 2999 50 HRC</td>
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</table>

*Figure 14.* Microstructure of gas nitried samples of the three steels (50 HRC).
Figure 15. Microstructure of salt bath nitrided samples of the three steels (50 HRC).
Figure 16. Hardness profiles of saltbath and gas nitrided samples of the three steels (Hardness: 50 HRC).