Article

Advanced complex analysis of the thermal softening of nitrided layers in tools during hot die forging

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Abstract: This article is devoted to the issues of thermal softening of materials in the surface layer of forging tools. The research covers numerical modeling of the forging process, laboratory tests of tempering of nitrided layers and the analysis of tempering of the surface layer of tools in the actual forging process. Numerical modeling was supported by measuring the temperature inside the tools with a thermocouple inserted into the tool to measure the temperature as close to the surface as possible. The modeling results confirmed the possibility of tempering the die material. The results of laboratory tests made it possible to determine the influence of temperature on tempering at different surface layer depths. Numerical analysis and measurement of surface layer microhardness of tools revealed the destructive effect of temperature during forging on the tempering of the nitrided layer and on the material layers located deeper below the nitrided layer.

Keywords: thermal softening; nitrided layer; hot forging

1. Introduction

Tools in the die forging processes are subjected to many destructive factors that contribute to their premature wear. The reasons can be found in the extremely difficult working conditions of the tools, which are subjected to cyclically changing high temperatures and pressures. The pressures are accompanied by the movement of the forging material on the tool surface. This creates a friction pair in which the surface of tools is subject to abrasive wear, which is further intensified by hard oxide particles flaking off the surface of hot charge material. Forging tools work at high temperatures, which are also cyclically variable, the surface tool layer is subjected to alternating compressive and tensile stresses while cooling the cavity. As a result, structural changes occur in the material. Initially, there is a temporary strengthening of the tool material during plastic deformation of the die in subsequent cycles. This strengthening decays as a result of thermally activated processes causing progressive coagulation of carbides and die recrystallization. This leads to the phenomenon of heat checking and thermo-mechanical fatigue, which causes cracking on the die surface. In the cracked zone, accelerated oxidation takes place, crack propagates deeper from the surface as well as its widening, which over time contributes to the formation of the first abrasive grooves. Immense amounts of heat transferred from the forging to the tools can also lead to overheating of the tool surface layer. This is the cause of tempering and, consequently, loss of hardness of the tool material, where one of the basic features that forging tools should have for hot forging is high hardness while maintaining sufficient crack resistance [1].

Therefore, by definition, forging tools should be resistant to the tempering effects of high temperatures that occur during forging. The tempering mechanism is dependent on the diffusion of carbon and alloying elements and is closely related to temperature and time. The influence of temperature and tempering time on steel hardness is presented by the tempering parameter M – J.H. Hollomon and L.D. Jaffe [2]:

$$M = T(C + \log t) \quad (1)$$

where:

T – tempering temperature on the absolute scale, K

t – tempering time in seconds,

C – constant depending on carbon concentration

Usually, the notion of temperability is limited to expressing the dependence of hardness on the tempering temperature at a constant time of this operation, and such relationships are determined under laboratory conditions. Graphs showing the dependence of hardness on the tempering temperature for individual tool steel grades are published in the technical literature and material data sheets [3]. In actuality, however, often the operating temperature range of the tool exceeds the tempering temperature range of the tool material. This may cause physical and structural changes in the material and thus disqualify the tool from further use. Moreover, exposure to high temperature is accompanied by high pressures, which may additionally decrease the hardness due to tempering.

One of the basic measures aimed at improving the performance of the surface layer of parts, including forging tools, is nitriding. By increasing the surface layer hardness, nitriding increases the tool resistance to abrasion, fatigue strength and corrosion resistance. Observations of many industrial forging processes in which nitrided tools were applied have proven that this treatment makes it possible to increase tool service life several times [4],[5]. Nevertheless, nitriding by itself does not protect the material from the adverse effects of high temperature. Attempts made for this purpose concerned the use of hybrid layers such as a nitrided layer/PVD coating, where the purpose of the coating, apart from increasing resistance to abrasive wear, is also insulation protecting the material against tempering under the influence of high temperature. Due to the very small thickness, the insulating effect is very short and, moreover, when the PVD coating is damaged, the tool material is no longer protected [6].

Another method of increasing the tool resistance to exposure of high temperature is the use of hard faced layers, where the use of a hard faced layer resistant to tempering in the temperature range of the tool with a relatively large thickness effectively insulates the basic metal [7].

Special lubricant/coolants are also used, which reduce friction between the tool and the forged part as well as cool the tool. In addition, after application, these agents create a lubricating film that effectively reduces the amount of heat that enters the tool from the hot workpiece material. For steel forging graphite-based lubricant/coolants are most often used, the heat transfer coefficient values of individual media vary depending on the type of graphite used [8].

Despite efforts to reduce the effect of heat on the tool surface, tempering of the forging tool surface layer is commonly observed in forging processes. This article deals with the research on the phenomenon of tempering of nitrided layers, as the most commonly used to increase the durability of forging tools.

2. Materials and Methods

In the research part, laboratory tests of the tempering of nitrided layers were first carried out, and then a comprehensive analysis of the tempering process under the actual conditions of the forging process was undertaken. This analysis was supported by numerical modeling and measurements of tool working conditions.

2.1. Testing tempering of nitrided layers under laboratory conditions

The tests were carried out on samples made of X37CrMoV5-1 hot work tool steel, subjected to quenching and tempering, the same as in the case of forging tools consisting in hardening and double tempering at 600°C. The hardness after heat treatment was 43-45 HRC. Then the samples were gas nitrided. To reproduce the effect of the temperature that occurs in the actual process on the nitrided layer, the treated samples were annealed at the temperature of 500, 550, 575, 600, 625, 650, 675 and 700°C. The adopted annealing time was 2 h and 4 h. During forging, the tool remains in contact with the hot material for a period of approx. 1 to 1.3 seconds, which means that for 10,000 forging cycles it remains in contact with the hot forging for a period of time of up to 4 hours. After sample annealing, the hardness distribution as a function of the distance from the surface was determined. The hardness was measured using LECO LM100AT microhardness tester. The measurement was performed using the Vickers method with a load of 100 g. The purpose of these tests was to experimentally demonstrate the effect of specific temperatures on the hardness of nitrided layers and the die core material. The test result was the development of tempering curves of the surface layer at different depths. The obtained curves can be useful in numerical modeling of the nitrided layer in the aspect of modeling forging tool wear.

2.2. Analysis of tool working conditions in a selected industrial forging process

The analyzed forging process is carried out on a Massey 2500T press with the speed of 60 strokes per minute in 3 operations. All tools are preheated to a temperature of approx. 250°C by prolonged contact with preheated preforms.

The dies are made of X37CrMoV5-1 hot work tool steel, quenched and tempered (quenching and double tempering at 600°C) to a hardness of 43 to 45 HRC and gas nitrided.

Each of the three operations involves top and bottom tools, each of which has slightly different durability. The preliminary forging tools (2nd operation) are characterized by the lowest durability, usually they are destroyed on average after approx. 7000 to 8000 cycles.

The key parameter affecting the tempering of dies is their working temperature. In order to determine the actual temperature prevailing on the surface and in the surface layer of the tools, the temperature was measured using a thermal imaging camera (before and after the forging process) and measured with a thermocouple inserted through an additional hole in the tool, approx. 4 mm below the surface. Fig. 1 shows schematically the measurement system with the place of measurement marked.

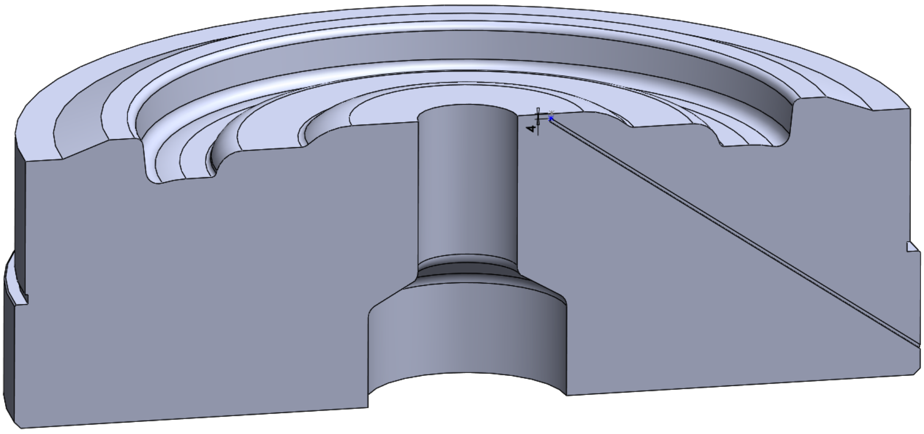
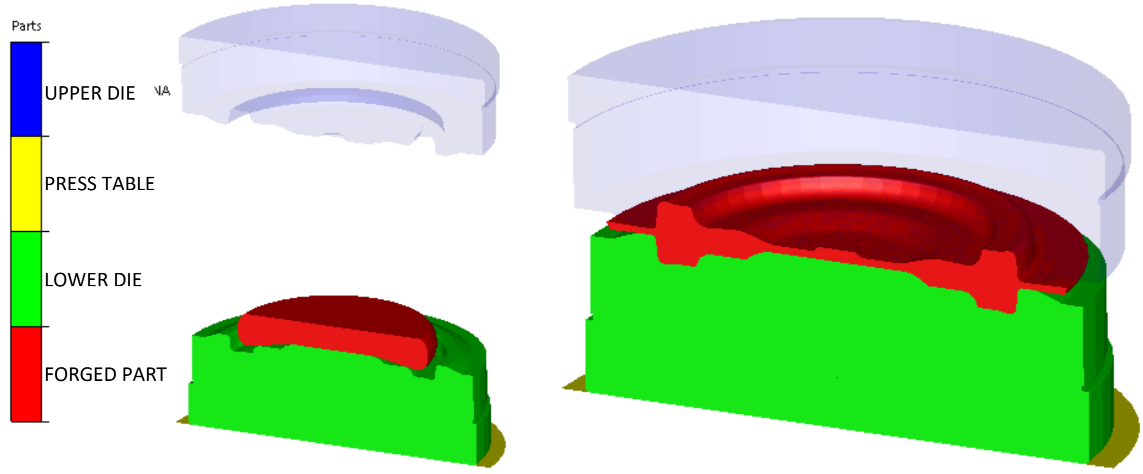


Figure 1. Method of temperature measurement in the tool during die forging.

As the temperature measurement with a thermal imaging camera is not possible at the moment of contact with the forging, when the temperature on the tool surface is the highest, numerical modeling was proposed to determine this value. Similarly, measuring with a thermocouple makes it possible to measure the temperature at a certain depth below the surface which is a valuable piece information but does not give the answer to what the maximum temperature in the surface layer is. Nevertheless, the data from both measurements served as boundary values for building and adjusting the numerical model so that the model was as close as possible to actual conditions.

Measurements using thermal imaging and the sensor, i.e. a thermocouple inserted into the die enable the determination of the die surface temperature, but not during contact with the hot forging (only before and after forging) and the temperature at a depth of 4 mm below the surface. These are valuable measurement methods, yet they cannot determine the maximum temperatures in the tool surface layer during forging. Therefore, these temperatures should be determined using numerical modeling. The obtained temperature measurement results can be used as boundary values for the developed model [9].

The axially symmetric numerical model of the successive cycles of 2 forging operations was developed using the Forge NxT 3.0 program. In the simulation, the forging and the bottom die insert were developed as deformable bodies. The top die and the bottom table were created as a non-deformable body in order to reduce the computation time and because this study deals with the temperatures on the bottom die. The general view of the model before and after the 2nd forming operation is shown in Figure 2.



(**a**) (**b**)

Figure 2. General view of the FEM model: (**a**) before; (**b**) after 2nd forging operation.

Based on the observation of the actual process, the single forging cycle was divided into stages and the average times of each stage were determined. The forging time of one forging (3 operations) takes approx. 14 seconds. The forging cycle in the second operation consists of the following stages:

1. Contact with the bottom die – 0.37 s
2. Deformation – 0.06 s
3. Contact with the bottom die before transfer – 0.58 s
4. Free self-cooling – 2 s
5. Lubrication – 0.7 s
6. Free self-cooling for the next cycle – 10.29 s

Based on the temperature measurement in the actual process, the following boundary conditions were adopted for the model development:

- the initial charge temperature is the same as after the forging was transferred from the 1st to the 2nd forging operation – it was exported from the simulation of the first operation (upsetting),
- die initial temperatures – bottom – 250°C, top – 350°C (its temperature does not change during calculations),
- heat transfer coefficients in contact – 14 kW / m^2 x °K between the dies and the forging,
- heat transfer coefficient of the bottom die working surface with the environment during lubrication/cooling 2.6 kW / m^2 x °K, during free self-cooling 15.5 W / m^2 x °K
- friction coefficient – Coulomb model marked in the program as water + graphite - $\mu = 0.15$.

2.3. Analysis of the influence of working temperature on the phenomenon of tool thermal softening in a selected industrial forging process

The influence of the working temperature on the tool thermal softening phenomenon was tested on tools that had worked a different number of forging cycles in the range from 1500 to 8000. First, the hardness distributions of the surface layer as a function of the distance from the surface were determined. Hardness measurements were carried out in the same place where the temperature during forging was measured and determined in modeling. Then, based on the results obtained, the surface layer was divided into 4 characteristic zones – layers at a depth of 0.05 mm, 0.1 mm, 0.15 mm and 0.2 mm. The next step was to assign the maximum temperatures that occur in individual zones and exposure times to temperatures above 600°C. The determined values allowed for the analysis of the impact of the holding time and temperature on the change in hardness and structural changes in the nitrided layer and in the layers directly under the nitrided layer.

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3. Results

The first part presents the results of the tempering of nitrided layers under laboratory conditions. Then, the results of a comprehensive analysis of the actual working temperature of tools in the forging process are included. This analysis was supported by numerical modeling of the forging process. The effect of the tests are the measuring results of microhardness and microstructure in actual tools used the forging process. These tests determined the effect of heat on the tempering of individual material layers in the nitrided surface layer of forging tools.

3.1. Test results for tempering of nitrided layers under laboratory conditions

The samples were held at temperatures ranging from 500-700°C for 2 and 4 hours. After annealing, the hardness in the near-surface area was measured. The hardness measurement results for samples held at different temperatures are shown in Fig. 6 and Fig. 7.

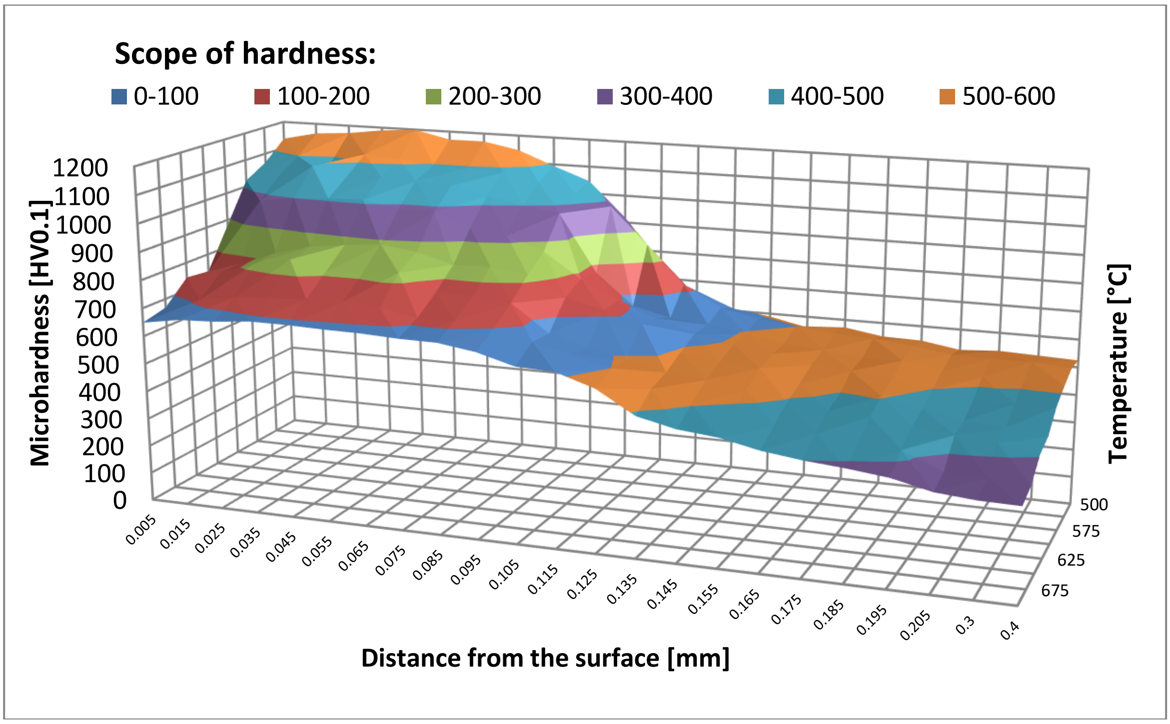

Figure 3. Hardness in the surface layer of samples after annealing for 2 hours as a function of the distance from the surface.

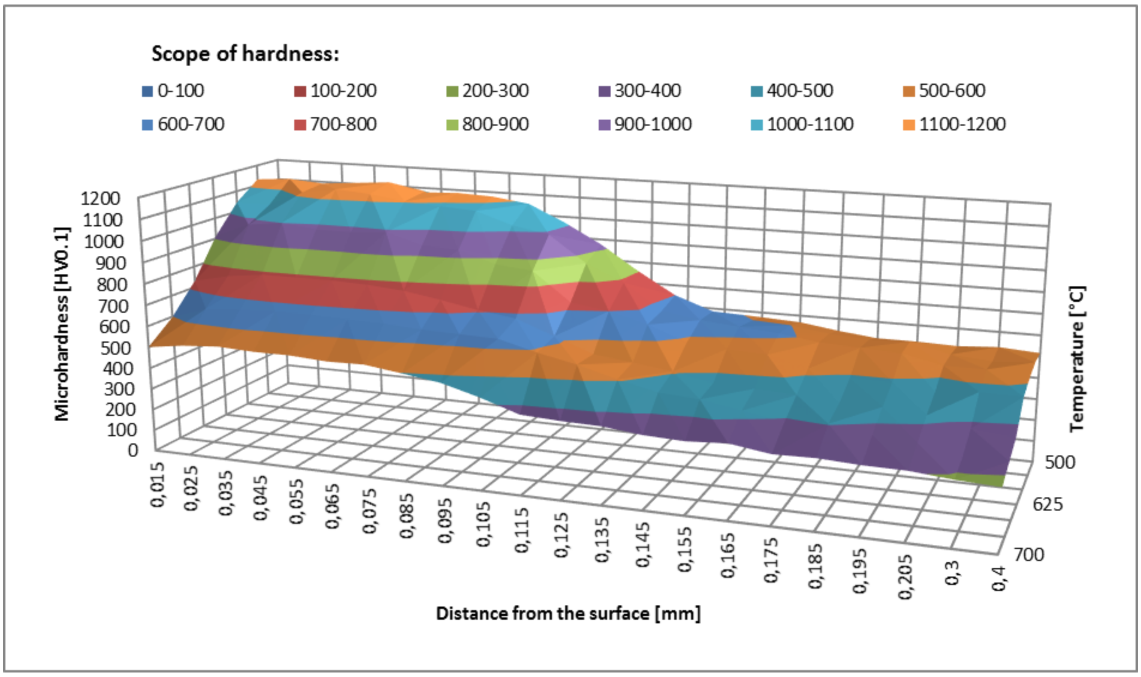

Figure 4. Hardness in the surface layer of samples after annealing for 4 hours as a function of the distance from the surface.

The hardness measurement results show a distinct tempering effect in the nitrided layer and below the hardened layer in the core material. As temperature increases, hardness decreases, the greatest decrease being recorded at temperatures above 600°C. The temperature of 600°C can be considered critical (the one that must not be exceeded), which is consistent with the data contained in the literature [10] and with the guidelines of tool steel manufacturers. Changes in the material hardness are also related to the holding time. By comparing the graphs in Fig. 3 and Fig. 4, a conclusion may be drawn that hardness depends on temperature and time. The results of nitrided layers tempered under laboratory conditions can be used as a reference in the evaluation of the tempering process taking place in actual forging processes. Based on these results it is possible to approximate at what temperature and for what period of time a forging tool has worked. This comparison can be distorted in the layers closest to the surface because in this zone the tempering is affected by the high pressure exerted and a number of physicochemical factors that do not exist under laboratory conditions during annealing.

3.2. Results of temperature analysis of tools in the die forging process

In order to determine the value of the tool working temperature, the measurement data was used first. Temperature measurement results include photos from a thermal imaging camera and data from the measurement using the thermocouple. Fig. 5 shows photos from a thermal imaging camera measured on the die working surface just before forging and immediately after removing the forging from the die surface.

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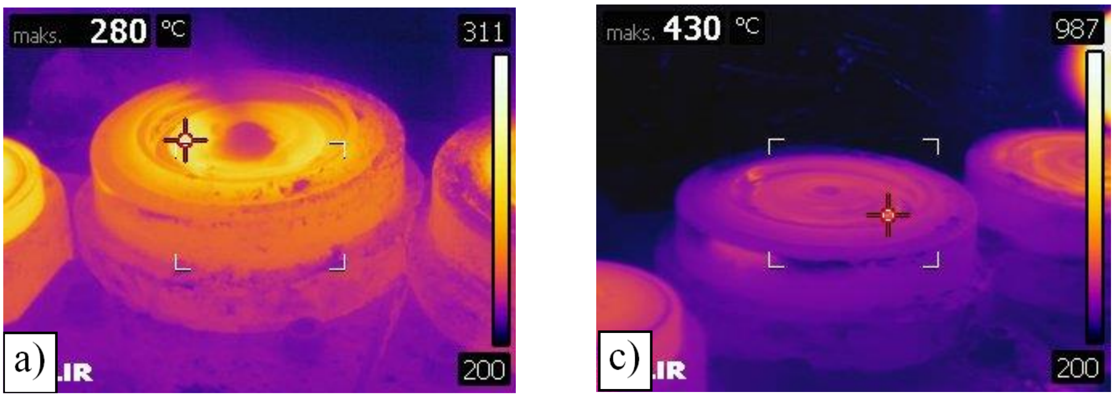

(**a**) (**b**)

Figure 5. Thermogram of the die working surface: (**a**) just before forging, after lubrication and cooling; (**b**) immediately after removing the forging from the die surface.

The results of the thermal imaging measurement indicate the presence of very high temperature amplitudes on the surface (over 150°C difference during one forging cycle). These differences result mainly from heating during contact with the forging and intensive cooling by spraying aqueous graphite suspension. During forging, the temperature may be much higher, which is also indicated by the results of the temperature measurement in the tool, measured with a thermocouple inserted into the tool, in accordance with the methodology described in section 2. The measurement results for a single forging cycle, measured approx. 4 mm below the surface, are shown in Fig. 6, for the period of the first 10 minutes of tool operation after pre-heating to 250°C.

The results of the tool temperature measurement at a depth of 4 mm in a single forging cycle show variation in the range of 420 to 426°C. These are very small changes compared to the changes in the range of 280 to 430°C observed on the surface in the measurement using a thermal imaging camera. The results presented in Fig. 6 demonstrate that the tool operating temperature stabilized after approx. 41 forging cycles. Due to the nature of the hand forging process, the temperature cycles are irregular.

The performed measurements made it possible to determine the temperature on the die surface, but not during contact with the hot forging (only before and after forging). Also, the measurement using a thermocouple only showed a temperature at 4 mm below the surface. These results are valuable, but numerical modeling was used to determine the maximum temperatures in the tool surface layer during forging. The obtained temperature measurement results were used to determine the boundary conditions for the developed model.

In order to compare the temperature measured with a thermocouple at a depth of 4 mm from the tool working surface with the results of numerical computation, a sensor was indicated in the model that reads the temperature from a selected place in the numerical simulation – its location corresponded to the position of the thermocouple measuring tip in actual tests. Figure 6 shows a comparison of the temperature curves from the actual forging process with the simulation results.

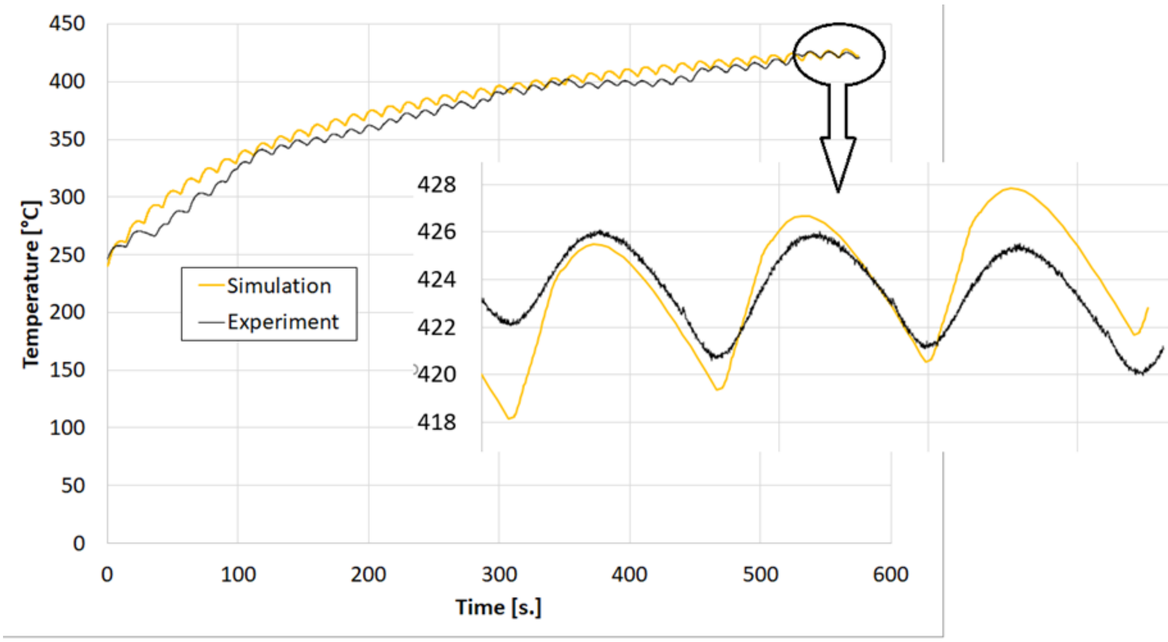

Figure 6. Comparison of temperature graphs from thermocouple measurements in the at a depth of 4 mm below the surface and from a sensor placed in the same place in the simulation.

Having analyzed the graphs of Fig. 6, it can be noticed that these waveforms correlate well with each other, after stabilizing the temperature during tests and simulations – after about 41 forging cycles.

In order to determine the temperature on the surface and at the depths of 0.05 mm, 0.1 mm, 0.15 mm, 0.2 mm, sensors were placed there in the numerical model. The temperature graphs in the last (41st) forging cycle in these areas are shown in Fig. 7.

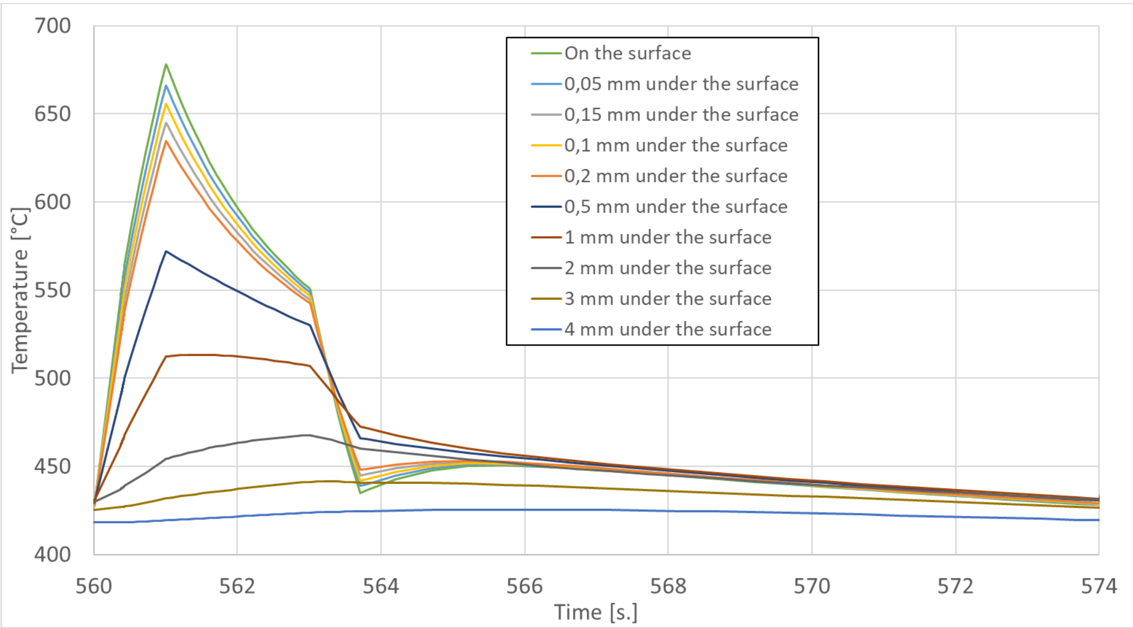

Figure 7. Temperature graph in the last (41st) cycle in the die in the second forging operation.

As shown by the temperature waveforms in Fig. 7, during forging, the temperature rises locally on the surface from approx. 430° C to approx. 680°C. So high a temperature can lead to tempering of the surface layer of forging tools.

The temperature distribution after 41 forming cycles in the last forging step on the bottom die insert is shown in Fig. 8. The figure also shows the location of the sensors from which the temperature was read in the numerical simulation.

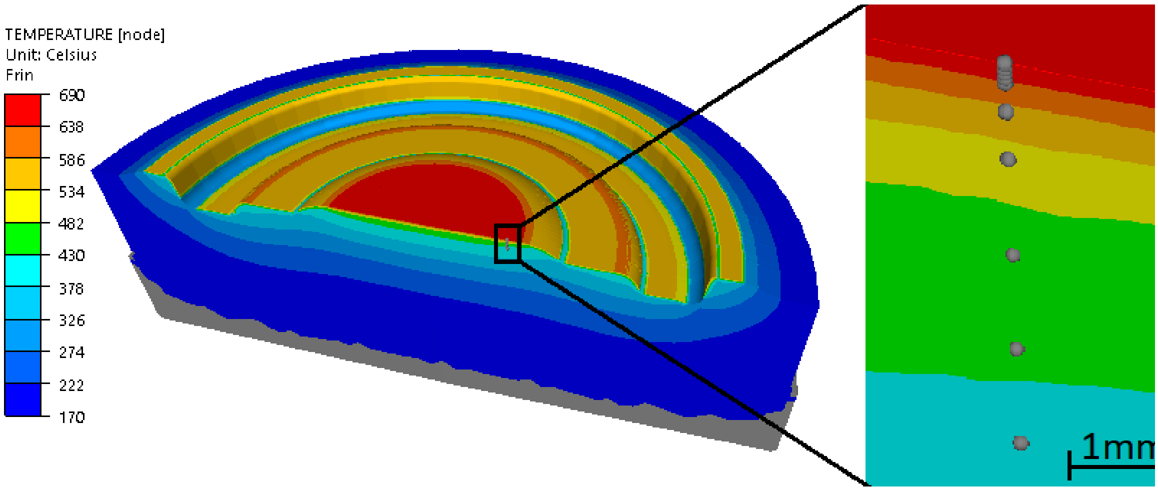

Figure 8. Temperature distribution in the last (41st) of 2 forging operations – the last forming step and the location of sensors in the numerical model

As can be seen in Fig. 8, the highest temperature occurs in the central part of the bottom die under consideration and this is where die tempering is expected.

3.3. Test results for tempering of nitrided layers under the conditions of the actual forging process

The influence of temperature and tool holding time on tempering was tested for 4 representative forging tools which have worked 1500, 3000, 7000 and 8000 forging cycles. Fig. 9 shows the result of the microhardness measurement in the surface layer of these tools, measured perpendicularly to the surface, according to the vector for which the tool working temperature was also determined.

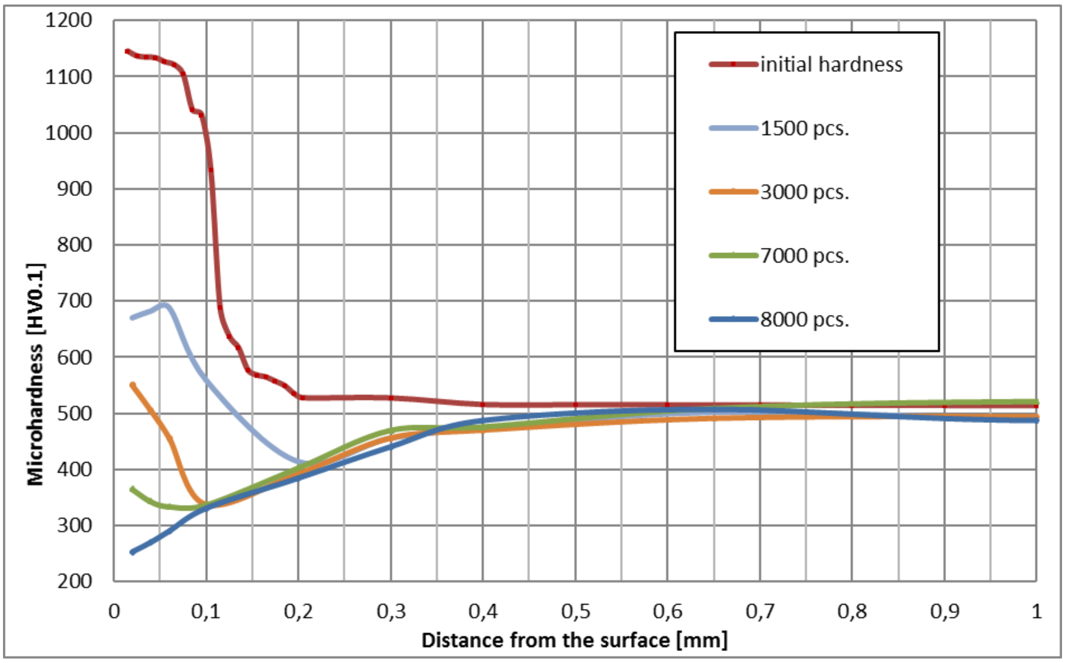

Figure 9. Results of microhardness measurement of nitrided surface layers after forging

The results shown in Figure 14 confirm the effect of heat at depths of up to 0.5 mm. This depth is much greater than the effective thickness of the nitrided layer obtained with the commonly used

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regular gas nitriding technology. These results reveal premature (after 1,500 forgings) tempering of the nitrided layer and of the layers of material below the nitrided layer. Based on these data, 4 characteristic zones were determined, in which a detailed analysis of temperature changes over time as well as changes in the microstructure and properties of the surface layer was carried out. The designated zones are layers of material at a depth of 0.05 mm, 0.1 mm, 0.15 mm and 0.2 mm.

In each of the zones, the maximum operating temperature and the of exposure time to the temperature above the maximum operating temperature of the specification, i.e. above 600°C, were determined based on the temperature analysis presented in Figure 7. These results are summarized in Table 1.

Table 1. Working conditions of the forging die at different depths.

Depth [mm]	Max. working temperature [°C]	Tempering time at temperature above 600°C [min]				
		in 1 cycle	in 1500 forging cycles	in 3000 forging cycles	in 7000 forging cycles	in 8000 forging cycles
0	678.05	1.31 s	33	66	153	175
0.05	666.1	1.18 s	30	59	138	157
0.1	655.56	1.01 s	25	51	118	135
0.15	645.11	0.90 s	23	45	105	120
0.2	634.67	0.71 s	18	36	83	95

The analysis of the influence of both of the aforementioned factors on the tempering of forging dies was carried out in the 4 mentioned zones. For each zone, a graph of temperature versus time, initial hardness and hardness after 1500, 3000, 7000 and 8000 cycles was determined. Moreover, an examination of the microstructure was made to verify changes in the microstructure of the surface layer.

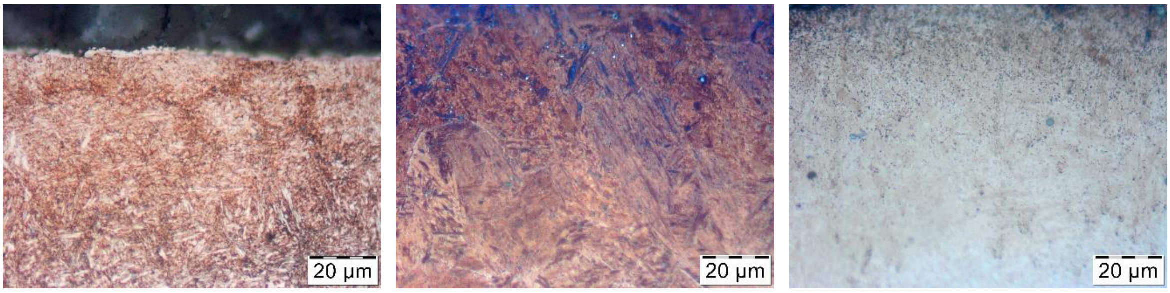
The first zone is a layer approximately 0.05 mm below the surface. There, hardness of approx. 1140 HV was obtained by nitriding. The maximum working temperature in this place was approx. 666.1°C and the holding time at a temperature above 600°C was 30 to 157 minutes. The results are shown in Table 2.

Table 2. Hardness of dies at indicated analyzed points.

Depth [mm]	Max. working temperature [°C]	Hardness [HV]				
		initial	after 1500 forging cycles	after 3000 forging cycles	after 7,000 forging cycles	after 8,000 forging cycles
0.05	666.1	1130	685	481	338	279
0.1	655.56	981	559	337	337	330
0.15	645.11	572	476	365	370	357
0.2	634.67	530	414	392	403	384

Based on the data obtained, the effect of the maximum operating temperature is much more significant than the effect of the holding time at temperatures above 600°C. The layer operating at a temperature of 666.1°C was ultimately softened significantly below 300 HV. The hardness of the layers which were not held so intensely decreased to 330 HV at 655°C, 357 HV at 645°C and 384 HV at 635°C (Table 2). The hardness reduction phenomenon was gradual and dependent on the holding time, as evidenced by the results presented in Table 1. Holding for 2 hours under laboratory conditions did not lead to such a radical decrease in temperature, while after 4 hours both at a depth of 0.05 mm and 0.1 mm, a decrease in hardness to 600-700HV was observed (Fig. 4). The reason for such a discrepancy may be the operating temperature in the forging process being temporary slightly exceeded due to cooling errors or difficulties in removing the forging from the die cavity. Then, in the case of such a high operating temperature, additional unexpected die heating can easily lead to overheating even above 700°C.

The thermal softening process in nitrided layers at a depth of 0.05 mm and 0.1 mm at a temperature of approx. 666.1°C led to a hardness decrease below the limit value of 500 HV only between 1500th and 3000th forging cycle, which corresponds to the holding time of approx. 1 hour. Under laboratory conditions, such intensive tempering took place at the temperature of 700°C (Fig. 3). Changes in the microstructure during tempering were also analyzed. Fig. 10 below shows the microstructure of the nitrided layer as delivered and tempered at 675°C for 2 hours, and the nitrided layer in the tool after 3000 forging cycles at approx. 666.1°C.

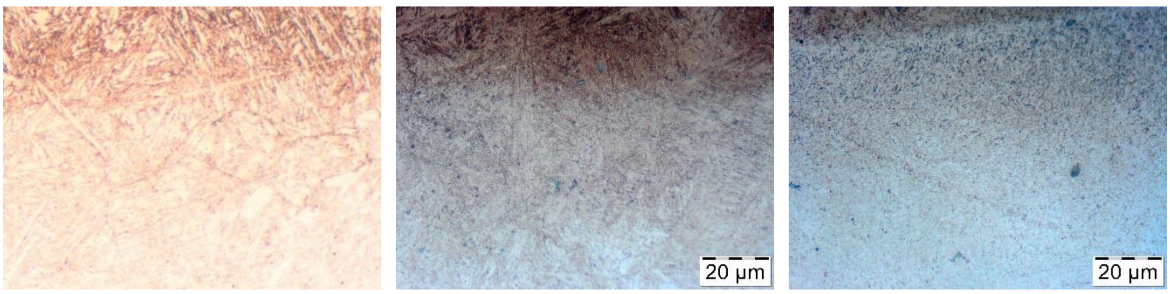


(**a**) (**b**) (**c**)

Figure 10. The microstructure of the nitrided layer: (**a**) as delivered; (**b**) held at 675°C for 2 hours; (**c**) after 3000 forging cycles at approx. 666.1°C.

The tempering phenomenon leads to the coagulation of cementite and other carbide precipitates, which can be observed in the nitrided layer in the form of small black dots. In the case of the die-derived sample (Fig. 10c), coagulated carbide precipitates are more numerous at the near-surface layer where annealing at higher temperature occurs. In the case of a sample annealed entirely in laboratory conditions, these changes are visible in its entire volume.

On the other hand, in the layers lying at a depth of 0.15 mm and below, where the hardness was initially approx. 550 HV, overheating led to tempering and a reduction in hardness of 350 to 400 HV. The reason for this change is undoubtedly holding at a temperature in the range of 600 to 645°C for 30 to 120 minutes. Also, annealing under laboratory conditions at similar temperatures for 2 hours at a similar depth led to a reduction of hardness to 350 to 450 HV (Fig. 3). Similar to the nitrided layers, the microstructure was also analyzed in this zone. Fig. 11 below shows the view of the microstructure of the layer at a depth of approx. 0.2 mm (below the nitrided layer) as delivered and tempered at 625°C for 2 hours, and the same layer of material from a die that worked 3000 forging cycles at approx. 625°C.



(a) (b) (c)

Figure 11. The microstructure at a depth of approx. 0.2 mm (below the nitrided layer) a) as delivered, b) held at 625°C for 2 hours b) after 3,000 forging cycles at approx. 625°C.

In the microstructure of the non-annealed nitrided layer, precipitates of cementite and other carbides in the material are sparse and dispersed (Fig. 11a). In contrast, in the case of the laboratory annealed sample (Fig. 11b), numerous coagulated precipitates appear, which are located throughout the entire volume of the material. In the sample coming from the forging die, these precipitates are mainly located directly under the nitrided layer, which proves that these parts of the material are overheated. This is confirmed by the results of temperature measurement and hardness measurement, which show that the material is thermally softened during forging at a depth of up to 0.5 mm below the surface.

4. Discussion and conclusions

The research on the thermal softening process of the surface layer of nitrided dies during hot forging lead to the following conclusions:

The tests conducted have shown that in the hot forging processes carried out in accordance with the adopted technology, the surface layer of working tools is overheated locally. The material in the surface layer on the surface and directly below the surface is temporarily heated to a temperature above 600°C and tempering occurs. Overheating occurs in the short time during contact with the forging and therefore cannot be observable. However, overheating effects are visible, because the surface layer is tempered to a depth of 0.3 mm. Consequently tempering leads to a decrease in the die hardness, which causes accelerated wear due to abrasion and plastic deformation.

The working temperature and the holding time are of have a paramount effect on the tempering phenomenon. The critical parameter in this case is exposure to high temperatures above 600°C. It was observed that even if the exposure time is very short, even short thermal cycles significantly decrease hardness when the material temperature exceeds 600°C. For higher temperatures in the range of 650 to 700°C, diffusion phenomena occur much faster and steel hardness decreases even below 300 HV.

The nitriding does not protect against the tempering phenomenon, but only delays the material softening process. Furthermore, during forging tempering occurs in the nitrided layer and in the layers deeper under the nitrided layer. Below the nitrided layer, tempering occurs relatively quickly and a soft layer is formed with a hardness below 400 HV. This may cause plastic deformation of the entire surface layer and cracking of nitrided layers due to the loss of base metal stability.

One should be aware of the real risk of overheating and use more effective cooling, as well as reduce the time of contact with the hot material of forgings. Moreover, it is recommended that the average temperature observed on the die surface should not exceed 400°C (excluding the moment of contact with the forging). Then the risk of reaching critical forging values exceeding 600°C is reduced.

In forging processes carried out in short (sometimes automatic) cycles, tool materials or hard faced layers with greater resistance to high temperature should be used. A thin layer with a minimum thickness of 1 mm as more resistant to tempering should suffice, because overheating in this case concerns only the surface layer at a depth of 0.5 mm..

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