INDUSTRIAL EXPERIENCES WITH CONTROLLED NITRIDING USING A ZEROFLOW METHOD

Steel nitriding using the ZeroFlow method allows precise forming of nitrided layers with respect to the required phase structure, zone thicknesses, and hardness distribution. In addition, the nitriding furnace uses simpler, inexpensive process adjustment and control system.

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This article presents experiences from steel nitriding using a ZeroFlow method in industrial conditions. Steel nitriding using this method allows precise forming of nitrided layers with respect to the required phase structure, zone thicknesses, and hardness distribution. The method is characterized by several times lower consumption of process gases compared with currently used controlled nitriding processes.

What is the ZeroFlow Method?

Controlled gas nitriding using the ZeroFlow process[1] based on carrying out the nitriding process using a unary atmosphere (only NH_3) is more economical and environmentally friendly than currently used controlled nitriding processes, which use binary atmospheres (NH_3 + diss. NH_3 and $NH_3 + N_2$). The adjustment of atmosphere chemical composition in the retort, and therefore the control of nitriding potential K_n, is performed by temporarily stopping and reactivating NH_3 feeding into the furnace. The amount of NH_3 fed into the furnace is adjusted (and controlled) using a gas analyzer (e.g., NH_3 or H_2 analyzer). Experimental work[2] shows that the ZeroFlow method is characterized by much lower consumption and emission of gases, requires a simpler control system, and allows forming the layer phase structure with the same precision as in processes with binary atmospheres.

Fig. 1 — Horizontal furnace for nitriding using ZeroFlow process.

Using ZeroFlow in Industrial Conditions

Figure 1 shows the furnace used for ZeroFlow nitriding in industrial practice at the Bodycote facility in Swiebodzin, Poland. More than 150 processes of industrial nitriding were performed during 18 months of operation. Applications included crankshafts, camshafts, toothed wheels from structural steels for toughening (5140, 4140, NIT135M), dies for pressure aluminum casting and tools for plastic forming from tool steels (H11, H13, D2) have been nitrided.

Nitriding crankshafts for sport car engines. Technology was developed to nitride crankshafts made of 4340 alloy steel using ZeroFlow method for a company making sports car engines (Fig. 2). User requirements for treated parts were set high regarding the layer phase structure, thickness of layer zones, thickness of effective precipitate zone, and hardness, as well as dimensional and geometrical changes. Requirements for the nitrided layer included:

- A 5 to 6 µm thick compound zone (white layer)
- Layer effective thickness with core hardness of 50+ HV at 0.4 mm
- Precipitation zone effective thickness with hardness 600HV at 0.15 mm
- Surface hardness of approximately 63 HRC

The first stage involved working out the nitriding recipe including simulation of the kinetics of nitrided layer growth[3]. In the calculations, a double-stage nitriding process was assumed when using temperatures of 490 and 530°C, and simultaneously a triple-stage nitriding if considering nitriding potential (K_n).

Figure 3 shows the simulation of effective layer thickness growth; the required effective thickness at 400HV (core hardness was 350HV) is obtained in the layer after approximately 29

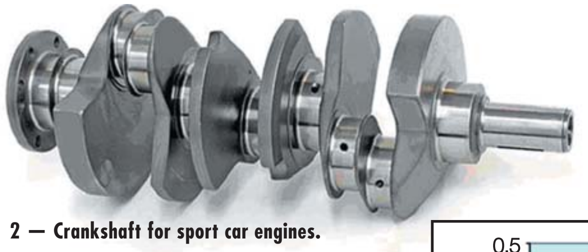

Fig. 2 — Crankshaft for sport car engines.

hours of nitriding (490ºC, 2 h, and 530ºC, 27 h). In further calculations, the nitriding potential K_N was selected to obtain the required compounds zone thickness (5 to 6 μm) in 29 hours. Those conditions were fulfilled using by potential K_n = 25 in the first stage of process, 0.6 in the second stage, and = 0.4 in the third stage. Times of individual stages are 2, 15, and 12 h, respectively (Fig. 4).

Metallographic examination of the crankshafts nitrided (ZeroFlow method) using the determined parameters was conducted. Photomicrographs of test sections are shown in Fig. 5, and the hardness distribution in the transverse section is shown in Fig. 6. Figure 5 shows that the total thickness of nitrided layer is about 0.4 mm and the compound zone thickness is 5.7 to 6.2 μm, which is consistent with expectations. The same effective thickness (GefHV400) was obtained using the hardness distribution measurement method; the effective thickness for hardness 600HV is approximately 0.16 mm, which is also consistent with expectations (Fig. 6).

Note that to prevent part deformation, the crankshaft was treated while in a special fixture. Furnace heating and cooling time were extended, resulting in only a small amount of deformation, which did not exceed the allowed deviations.

Figure 7 show the course of the most important process parameters: temperature, ammonia feed intensity, and nitriding potential in the retort (Fig. 7 b is a enlarged portion of Fig. 7a). The most interesting aspect is the ammonia consumption in the ZeroFlow process. In the analysis, a few stages have been separated: purging, and the first, second, and third nitriding stages.

Air used for surface activation is removed from the retort using a vacuum pump (the furnace and retort are designed for use in vacuum conditions), and replaced with ammonia. Therefore, ammonia consumption is equal to the retort volume (~ 1 m3).

The first nitriding stage (at 490ºC for 2 h) was performed using an ammonia flow rate of 30 l/min, which con-

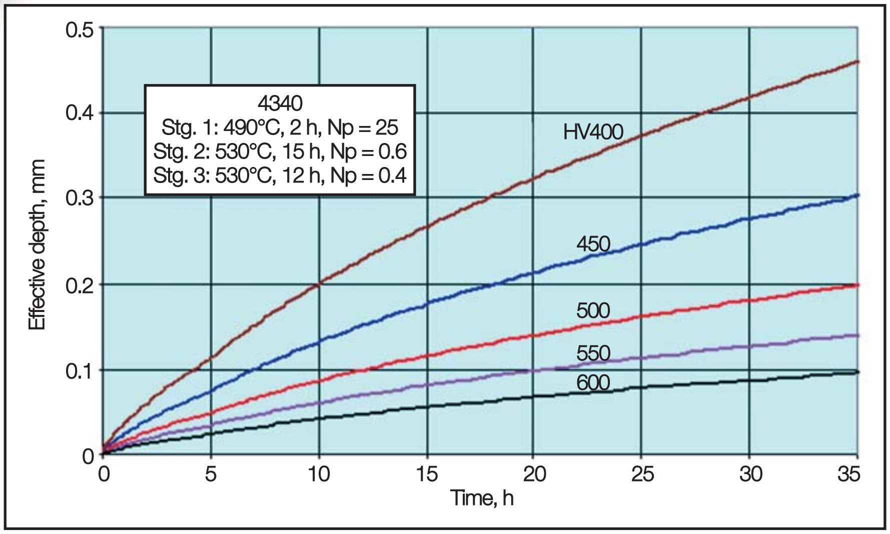

Fig. 3 — Growth of effective depth case on 4340 steel.

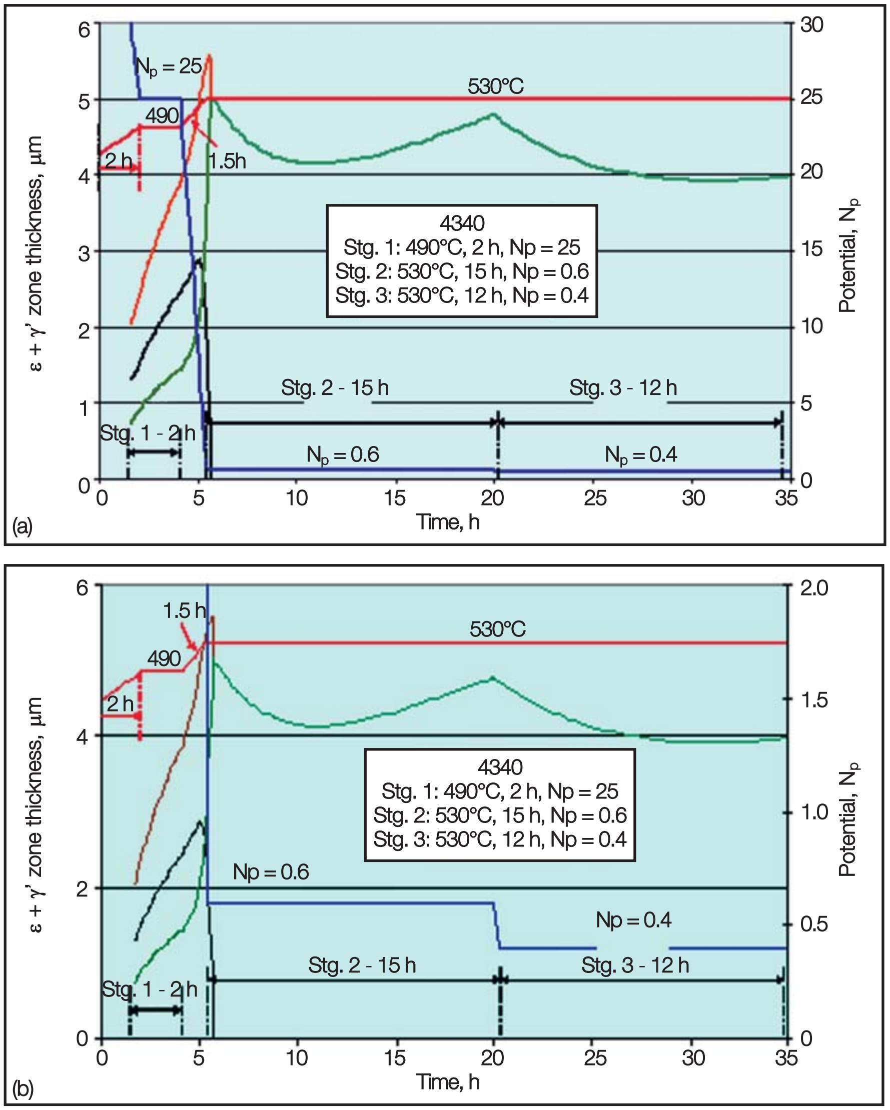

Fig. 4 — Growth of white layer on 4340 (a) and reduced range of K_N on the right axis (b).

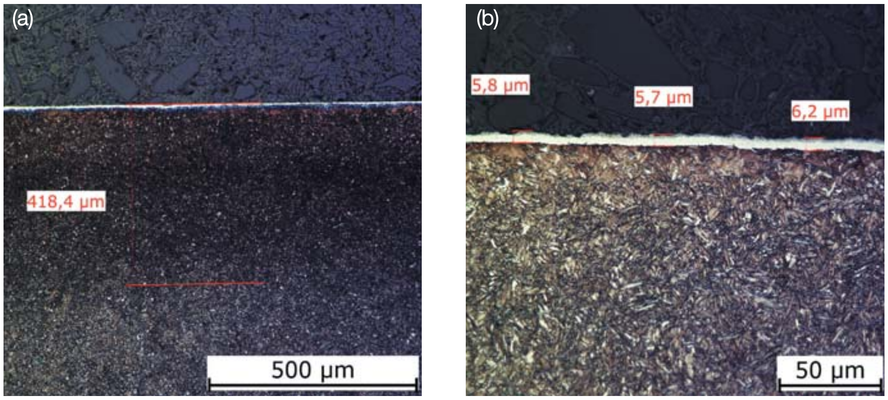

Fig. 5 — Photomicrographs of the nitrided layer (a) and iron nitrides (b) on 4340 steel nitrided using ZeroFlow process at: stage I (490°C, K_N = 22 atm$^{-1/2}$, 2 h) and stage II (530°C, K_N = 0.8 atm$^{-1/2}$, 15 h and K_N = 0.7 atm$^{-1/2}$, 12 h).

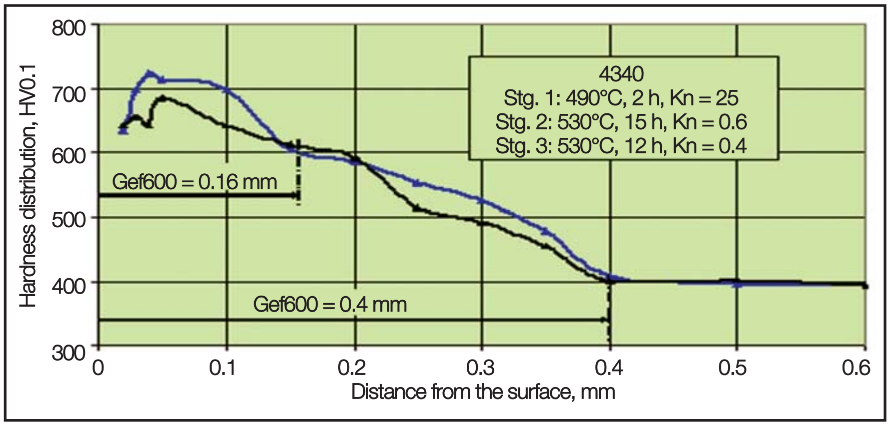

Fig. 6 — Hardness distribution of nitrided layer on 4340 steel.

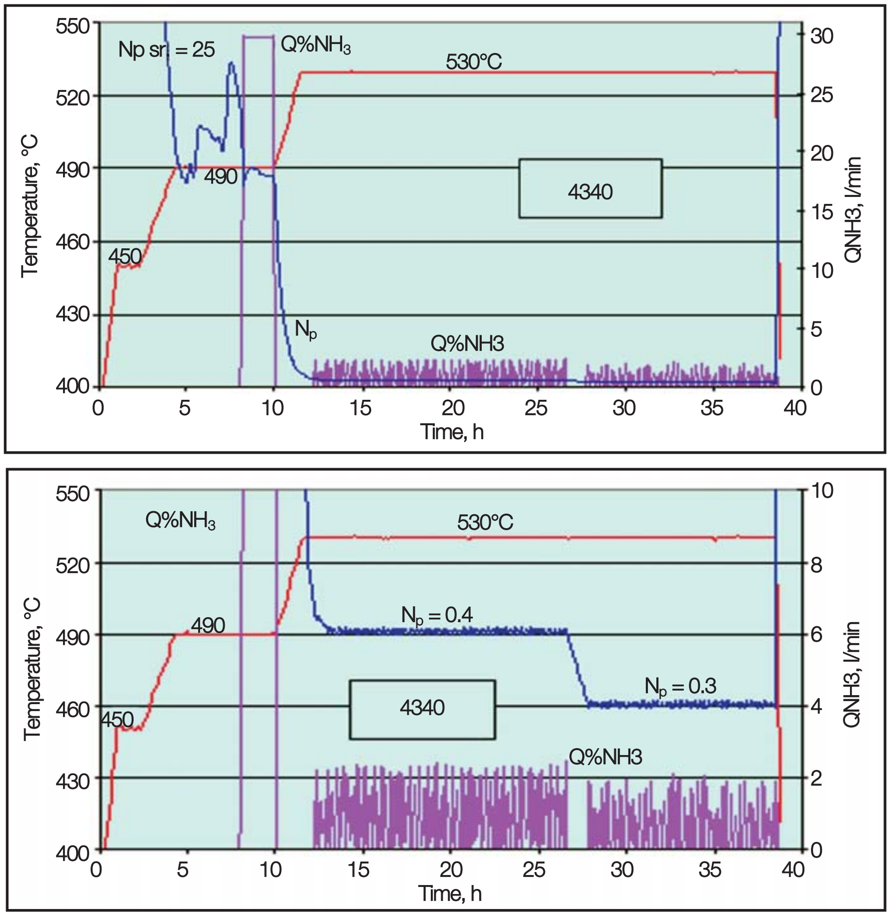

Fig. 7 — Top view shows variations of temperature, inflow rate of NH_3 into the retort and NH_3 content in a retort during nitriding of crankshaft; (bottom) enlarged portion of the top graph.

sumed 1.8 m^3. In the second and third stages, the average flow rate was 0.8 and 0.7 l/min, respectively, which consumed 1.2 m^3. Cooling was performed without any ammonia flow. When the temperature was close to ambient, atmosphere was removed via vacuum pump. Total ammonia consumption was 6.6 m^3.

NH_3 Consumption in Different Processes

The consumption of ammonia was compared in the ZeroFlow process and in atmosphere processes using NH_3 + diss. NH_3 (Floe process) and NH_3 + N_2. In the process using NH_3 + diss. NH_3, atmosphere replacement (purging) and the first stage of nitriding are performed identically; i.e., using only NH_3. Therefore, the consumption of NH_3 is the same as in the ZeroFlow process. Differences in NH_3 consumption appear in the second and the third nitriding stages. The consumption is determined by the chemical composition of the atmosphere (NH_3 + diss. NH_3) fed into the furnace. The literature[2] shows that the more NH_3 is diluted with diss. NH_3, the higher will be the flow of that mixture to the furnace ensure the required nitriding potential K_n; therefore the higher NH_3 consumption will be.

For comparison, the typical atmosphere 30% NH_3 + 70% diss. NH_3 used in the industry was used. The consumption in the second and the third stage is 21.5 m^3, or 18 times higher than the consumption in the ZeroFlow process. Total consumption of NH_3 is 24 m^3, or four times higher than in the ZeroFlow process. The difference increases with process time.

The consumption of NH_3 + N_2 atmosphere was not measured. According to previous work[4], for the typical industrial atmosphere (25% NH_3 + 75% N_2), consumption of NH_3 in the second and third stage is two times higher than in the ZeroFlow process. For the entire nitriding process, that difference drops to approximately 1.5 times. However, in the process using NH_3 + N_2 atmosphere, the nitrogen used amounts to a few times higher than NH_3, or about 30 m^3 (no nitrogen is used in the ZeroFlow process).

Nitriding of plates for casting glass bulbs for lamps. The plates are made of N135M (41CrAlMo7-10) steel. In the center of the plate, there is a hole (Fig. 8) through which the glass bulb is cast.

The internal surface of the hole, especially its upper edge is subjected to wear due to erosion. The operational durability of 300 h is required. The nitrided layer should be 0.20 to 0.25 mm thick and should not have the white zone, which required double-stage nitriding (490ºC, 2 h and 550ºC, 24 h). Figure 9 shows the microstructure of transverse microsections of obtained layer. The consumption of NH$_3$ in the ZeroFlow method was 5.0 m^3. By comparison, the consumption of NH$_3$ would be about 15.5 m^3 using a 20% NH$_3$ + 80% diss. NH$_3$.

Therefore, the consumption of NH$_3$ in the described process is approximately 3 times lower than the consumption obtained in the Floe process (in case of crankshafts this ratio was 1:4). The lower difference than for the crankshafts results from slightly shorter nitriding time.

Nitriding of toothed wheels for wind power plants. Nitriding technology was developed using the ZeroFlow process to treat toothed wheels for a European company making wind power plants. The wheels are made of 42CrMo4, 31CrMoV9, and 34CrNiMo6 steels. Wheel diameters ranged from 30 to 180 cm (Fig. 10 shows the smaller wheel). Due to high surface pressure on the teeth, a thick nitrided layer (0.6 to 0.8 mm) without the white layer is required.

The same method was used as that for nitriding the crankshafts. To increase the kinetics of layer growth, the nitriding temperature was increased to 550ºC, nitriding time was increased to 60 to 80 h depending on the steel type, and the nitriding potential K_n of atmosphere was lowered in the second and third process stage (0.3 and 0.2 were used).

Atmosphere replacement and the first stage of nitriding were performed in the same way as for the crankshafts. It required increasing of NH$_3$ flow rate to the retort to 0.5 and 0.4 l/min. As the times of the second and the third stages were longer (~50 to 78 h), the consumption of NH$_3$ totaled 1.7 to 2.3 m^3, and 7.3 to 7.9 m^3 for the entire process.

In the Floe process (to compare the consumption), a typical 20% NH$_3$ + 80% diss. NH$_3$ atmosphere was used in the second and the third stages. NH$_3$ consumption was 52 to 70 m^3, and 58 to 72 m^3 for the entire process; i.e., 7.5 to 9 times higher than in the ZeroFlow process.

Fig. 8 — Plates for casting glass cans to light bulbs.

Nitriding of dies for pressure aluminum casting. Dies for pressure aluminum casting require thin nitrided layers (50 to 90 µm) and a few microns of compounds zone. Therefore, the process is performed at a temperature of 500ºC with a nitriding time of 0.5 to 1 h. Due to such a short time, the process should be treated as unbalanced and partially non-isothermal (actually a significant part of the nitriding layer part is created during heat up.

In the ZeroFlow method, the amount of used NH$_3$ was limited only to filling the retort (after extracting the air), amounting to about 1 m^3. In the traditional nitriding method, such short processes are performed similarly, with use of NH$_3$ alone, often with limited constant flow of the gas. Therefore, there is no significant difference between the two processes.

Summary

The use of ZeroFlow nitriding in industrial practice for many parts has proven to be as efficient process as previously used methods of controlled nitriding. It allows creating layers with phase structure required by the industry, with required thicknesses in the zones layer, and with required hardness distribution. In addition, the ZeroFlow process has proven to be much more economical in gas consumption than others. Experience shows that the hydrogen or ammonia analyzer and the feeding valve for NH$_3$ are sufficient for the adjustment of atmosphere chemical composition in the retort (nitriding potential).

By comparison, in the processes using binary NH$_3$ + diss. NH$_3$ and NH$_3$ + N$_2$ atmospheres, two gas flow meters are required. Furthermore, processing using NH$_3$ + diss. NH$_3$ atmosphere requires a dissociator, which also increases the nitriding costs. Processing using NH$_3$ + N$_2$ does not require a dissociator, but due to introduced nitrogen, it requires chemical analysis of the furnace input and output atmosphere, which complicates process control.

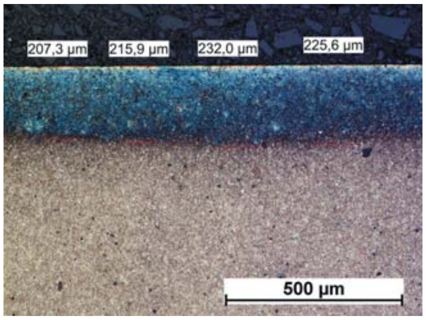

Fig. 9 — Photomicrograph of the nitrided layer on NIT 135M steel nitrided using ZeroFlow process at: stage I ((490°C, K_N = 22 atm$^{-1/2}$, 2 h) and stage II (550°C, K_N = 0.5 atm$^{-1/2}$, 12 h and K_N = 0.2 atm$^{-1/2}$, 12 h).

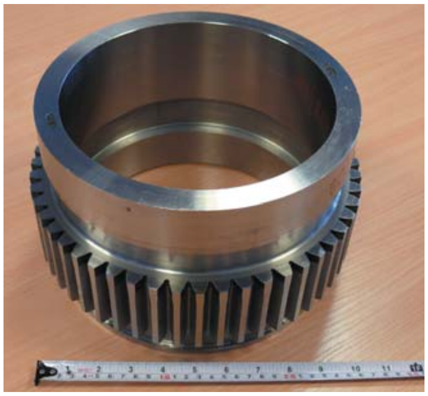

Fig. 10 — Gears for a wind-power station.

Fig. 11 — Nitrided aluminum extrusion dies.

Experience also shows that in the retort designed for operation in vacuum conditions (also at a temperature of 350ºC after the air surface activation), nitrogen may never be needed to replace the retort atmosphere, and is required only for emergency situations. HTP

References

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