Extrusion dies are usually regenerated several times (min 4 times). The phenomenon of extended life after each regenerative nitriding process has not been explained. In this work, the regeneration process of dies used in the extrusion of aluminum profiles has been presented. In the article, it was sought to explain the cause of increased die durability after the third or fourth nitriding. Also in this work is presented an analysis of the influence of the parameters of gas nitriding with the ZeroFlow method on hardness of dies. Results were verified under industrial conditions at extrusion company, comparing the durability of the dies nitrided with the ZeroFlow method with so-far-used dies nitrided in the commercial way. An increase of the dies durability was achieved after a single ZeroFlow nitriding.

Key words: aluminium, extrusion, nitriding, die, wear

INTRODUCTION

Extrusion of aluminum profiles is one of the most widely propagated methods of forming semi-finished products. This process is performed hot and is usually applied to obtain long products in the form of bars, pipes, and other profiles. The stock, heated to a high temperature, is extruded in horizontal presses with a press force from 4 to 100 MN through an open die. This process is usually continuous thanks to the application of automatic stock feeding and combining it with plastic working. As shown by data in the literature and the author’s own studies, the stock and die are typically heated to a temperature within the range of 450-550 °C, however, as a result of internal friction between the material and forming surfaces, it may rise even up to 620 °C [1]. Extrusion dies are usually made from AISI H11 hot work steel (EN X37CrMoV5-1) or AISI H13 (EN X40CrMoV5-1) thermally treated by quenching and high-temperature tempering. Manufacturing experience shows that nitrided layers produced on dies do not provide an appropriately long operating life. They also do not ensure repeatability of results. Wear phenomena of such dies are described in various publications, including in [2], where different wear types are indicated, from adhesive wear through spalling to abrasive wear [3, 4]. Considerations of such high temperatures on the working surface has led to attempts made for several years now to implement more expensive technology for modification of the working surface of dies by applying PVC/CVD methods and analyzing their wear resistance.

The authors of study [5] proved that the shape of a die has a significant impact on the uniformity, depth, and quality of the nitried layer, as well as on the hardness, both surface hardness and its distribution from the surface into the die. They observed that exterior corners exhibit a broad nitried layer due to simultaneous diffusion from two convergent directions. However, in the case of interior corners, diffusion is hindered and a more shallow nitried layer is formed. This effect causes a reduction of surface hardness, and thus, a reduction of strength. The results of their experiments emphasize the necessity of accounting for the shape of an individual die profile for nitriding in order to optimally design new die types for aluminum extrusion, with a greater operating life [5]. This effect can be eliminated by applying e.g. the controlled gas nitriding process, which allows for such selection of the parameters of process that prevents the formation of a layer rich in iron carbonitrides, particularly on corners of the working surface.

Regeneration of dies for extrusion of aluminum profiles is a difficult process based on preparing a bearing surface, removing aluminum residue (etching in caustic soda with a concentration of approx. 25 % at 80 °C and subjecting them to repeated gas nitriding. This safe and clean process can only be performed on specialized machinery imported from German, Italian, or British companies. After their surfaces are prepared and their profiles and shape are corrected, dies may be subjected to gas nitriding. This technology, as shown in the literature, is the most economical and is more advantageous compared to deposition technologies (PVD and CVD).

The importance of the problem of increasing die operating life is illustrated by the fact that the average operating life of an aluminum extrusion die amounts to 25 tons of gross processed aluminum. In the case of production at a level of 100 000 tons of extruded profiles in Poland, it is necessary to use approx. 4 000 dies at an estimated cost of 10 000 000 €. Reduction of purchases...
ing needs by at least 10% means that potential savings amount to 1-2 mln €, and on the scale of Europe, where consumption of aluminum profiles is at a level of 1.2 mln tons annually, this amount is much greater.

Considering the temperature of the nitriding process and working temperature, one can pose the hypothesis that growth of the diffusion layer may take place during exploitation as a result of long-term holding at a temperature similar to the nitriding temperature, and this will have an impact on increasing die operating life after the third regeneration nitriding.

IDENTIFICATION OF DIE WEAR

In the first stage, 5 die types for extrusion of hollow profiles (consisting of a core part and a plate part) and 8 die types for extrusion of flat profiles, including dies for extrusion of profiles with complex shapes, were subjected to study. Dies subjected to varying numbers of nitriding regeneration cycles, applied for dies multiple times during the production process for the purpose of improving their operating life, were studied (dies were nitrided in 1 to 8 cycles). Dies that underwent various degrees of exploitation were analyzed (new, after exploitation – suitable for further work, at different nitriding regeneration cycles, and worn out – withdrawn from further production).

Observations of the surface morphology of the bearing surface of dies, on which the greatest forces/stresses occur over the course of the extrusion process, were conducted by means of an Inspect S (FEI) scanning electron microscope. Next, metallographic specimens were prepared and subjected to examinations through an Eclipse L150 (Nikon) optical microscope as well as tests of hardness distribution on die cross-sections, from the nitrided layer in the direction of the core, by means of a Micromet 2104 hardness tester (Buehler). Based on analysis of results, the following causes of wear on the bearing surface of dies were identified:

− cracks and spalling caused by thermomechanical fatigue (Figure 1a - b),
− corrosion pits caused by cleaning/etching of the die in a sodium hydroxide solution. Cleaning is an inter-operational to reveal the condition of its surface, and it is always performed before the die regeneration process (Figure 1c),
− abrasive wear on the bearing surface, grooves in the extrusion direction, and spalling, which was initiated by flaking or cracks in the die’s surface layer (Figure 1d).

Based on analysis of data from the Albatros Aluminium company, it was observed that die operating life increases after successive nitriding processes. This data was compared to the results of metallographic examinations presenting the thickness and structure of the nitrided layer as well as hardness distribution profiles on the die’s cross-section in its calibrating part. Examples of structure images are shown in Figure 2. It was established that dies after the 2nd, 3rd, and 4th nitriding process exhibit the greatest operating life. Analysis of the structure and hardness of layers of the lowest durability made it possible to accept preliminary assumptions as to the structure and hardness of the prototype nitrided layers by ZeroFlow method.

Layers produced in the first conventional nitriding process are shallow, and their thickness is below or approx. 100 μm (Figure 2a), which quickly creates the necessity of their regeneration. In further nitriding processes, when dies are regenerated, the thickness of the nitrided layer increases to a value within the range of 150 to 550 μm (Figure 2b-c), depending on the contractor of nitriding services, the shape of the die, total number of nitriding cycles, and the die’s condition before regeneration. In subsequent nitriding processes, a mesh of nitride phases may appear on the die’s calibrating surface, which is unfavorable from the perspective of exploitation because that is where cracks and spalling initiate.
Based on obtained results of studies and consultations with the manufacturer of profiles, it was assumed that it would be a good solution to produce layers using the ZeroFlow method in just one nitriding process, which will provide an operating life greater than that yielded by conventional processes (with successive nitriding regeneration processes). Such a solution will reduce production costs by reducing the frequency at which die regeneration becomes necessary and by reducing the number of dies in the production of a given profile. Furthermore, it will eliminate the problem of the criterion for transferring dies for regeneration, which cannot be unequivocally determined under production conditions.

**DIE OPERATING LIFE AFTER ZEROFLOW NITRIDING**

Production tests were conducted for dies with prototype nitrided layers obtained in ZeroFlow processes and dies subjected to conventional nitriding (with successive nitriding regeneration processes). This method makes it possible to produce layers with a specific phase structure using simple yet precise regulation of the chemical composition of the atmosphere in the furnace retort by means of periodical opening and closing of ammonia supply to the retort. Such a solution reduces NH3 consumption several times as well as emissions of post-process gases into the environment, and as a result it also reduces nitriding costs [6, 7]. Extrusion processes were conducted at Albatros Aluminium on their hot aluminium extrusion process line in a standard production cycle. Dies with prototype layers nitrided using the ZeroFlow method and conventional dies were used interchangeably at the same extrusion parameters (process temperature and rate). Before installation on the extrusion press, dies were subjected to soaking at a temperature of 470 °C for approx. 4 - 5 h. After approx. 2 - 2.5 tons of profile were extruded through every die opening, dies were uninstalled and soaked once more.

Over a dozen dies were subjected to nitriding using the ZeroFlow method at various process parameters, including dies for extruding standard hollow and flat profiles with a regular shape and wall thickness > 2 mm, dies for extruding complex profiles with diverse shapes and wall thickness from 1.5 to 2 mm, as well as dies for extruding special profiles with complex shapes and wall thickness from 0.8 to 1 mm. The shapes of the selected dies are shown in Figure 3. Parameters of die nitriding using the ZeroFlow method are compiled in Table 1.

<table>
<thead>
<tr>
<th>Process No.</th>
<th>Parameters of the nitriding process</th>
<th>Effective depth /μm</th>
<th>Near-surface hardness / HV 0,1</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>550 °C, Np = 0,4, t = 24 h</td>
<td>160</td>
<td>988</td>
</tr>
<tr>
<td>2</td>
<td>550 °C, Np = 0,4, t = 55 h</td>
<td>325</td>
<td>924</td>
</tr>
<tr>
<td>3</td>
<td>550 °C, Np = 1,0, t = 4,5 h</td>
<td>145</td>
<td>1072</td>
</tr>
<tr>
<td>4</td>
<td>550 °C, Np = 1,0, t = 55 h</td>
<td>465</td>
<td>988</td>
</tr>
<tr>
<td>5</td>
<td>550 °C, Np = 1,0, t = 100 h</td>
<td>545</td>
<td>876</td>
</tr>
</tbody>
</table>

*Criterion of effective depth: core HV+50 HV 0,1

After nitriding, specimens were subjected to metallographic examinations, hardness measurements for the purpose of determining hardness distribution profiles, and effective depth. Selected results of hardness tests are presented in Figure 4.

Specimens were nitried using the ZeroFlow method together with dies in order to determine the structure and hardness profiles, and in order to relate this data to die operating life depending on applied nitriding parameters.

Figure 5 presents selected results of analysis of the operating life of conventional dies and dies nitrided using the ZeroFlow method, expressed as the amount of aluminium extruded through every die opening, in the case of a dual-opening die for extrusion of hollow profiles with wall thickness of 1.2 mm (Figure 5a) and six-opening die for extrusion of flat profiles with a wall thickness of 0.9 mm (Figure 5b).

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**Table 1 Die nitriding parameters in the ZeroFlow method (with 1st step: T = 490 °C, Np = 15 atm⁻¹, t = 1 h), effective depth and near-surface hardness of nitrided layers**

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**Figure 3 Shapes of dies for extruding aluminium profiles and wall thicknesses of the profiles extruded through them**

**Figure 4 Hardness distribution in die segments nitrided using the ZeroFlow method at various parameters of the 2nd process step (1st step parameters: T = 490 °C, Np = 15 atm⁻¹, t = 1 h)**
ANALYSIS OF RESULTS AND SUMMARY

Analysis of the results of production tests conducted on dies allowed for the following conclusions to be made:
− an increase in operating life was achieved for dies nitrided one time using the ZeroFlow method at the specified parameters in comparison to conventionally nitrided dies. Single nitriding using the ZeroFlow method made it possible to replace from 3 to 7 processes of conventional nitriding regeneration,
− the longest operating life of dies nitrided using the ZeroFlow method, regardless of their shape, was achieved at the following nitriding parameters: 1st step $T = 490 \, ^\circ C$, $N_p = 15 \, \text{atm}^{-\frac{1}{2}}$; $t = 1 \, \text{h}$, 2nd step $T = 550 \, ^\circ C$, $N_p = 0.4 \, \text{atm}^{-\frac{1}{2}}$; $t = 55 \, \text{h}$. These parameters made it possible to obtain a nitrided layer with an effective depth of $325 \, \text{mm}$ and near-surface hardness of approx. $924 \, \text{HV 0.1}$, and the produced nitrided layer was free of nitrate mesh and the so-called corner effect.
− based on a comparison of the results of metallographic examinations of die segments and actual dies after exploitation, an increase in the thickness of the nitried layer was observed in all of the analyzed cases under the influence of work. This is explained by the effect of nitrogen diffusion into the die under the influence of profile extrusion conditions (temperature approx. $500 \, ^\circ C$).

REFERENCES


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