LOW TEMPERATURE GAS NITRIDING OF Fe-Cu-C SINTERED COMPONENTS

P. Zlateva, T. Pieczonka, M. Stoytchev

Abstract

Low temperature gas nitriding was used for iron based sintered compacts to increase their surface hardness without causing significant dimensional changes. Sintered compacts were produced by conventional pressing/sintering technique using as a starting material the elemental powders mixture containing Höganäs SC 100.26 iron powder grade as a base and fine electrolytic copper powder and C-UF graphite powder, as alloying elements. Sintering was carried out in an industrial belt furnace in a protective atmosphere of dissociated ammonia NH₃. Sintered compacts were nitrided at 570°C for three different saturation periods of 300, 600 and 900 seconds in ammonia atmosphere. The influence of nitriding time on nitrided microstructure, type and amount of nitride phases and hardness and microhardness of the nitrided layer was established.

Keywords: sintered structural iron based parts, nitriding, surface hardening, iron nitrides

INTRODUCTION

The new powder metallurgy products have gained a widespread application in the development of new technologies. The products based on ferrous powder SC 100.26 are of great practical interest to industry. Examples of widely used iron powders are the reduced NC 100.24 and ASC 100.29 which have lower physical and mechanical properties than SC 100.26. Scientific research aims at production and investigation of materials satisfying the technological needs and requirements, i.e. materials with specific properties.

Powder metallurgy is among the most prestigious and promising industries marked by a rapid development last decades of the 20th century [2,3,4]. Approximately 800 thousand tons of iron powder are consumed annually to produce sintered parts. These technological processes are among the most effective methods offering extensive opportunities to save material, labor and energy. The processes in question are comparable with the other conventional processes in terms of the properties of the products obtained, and in many cases, even surpass them.

A growing part of the products of powder metallurgy are designed to work in conditions of stress and shock loading, at elevated temperatures and corrosive environments and they have to meet the requirements for high strength, durability, corrosion resistance and good presentation. Practice shows that over 50% of the construction products based on powder metallurgy should be subjected to thermal or chemical heat treatment to increase the reliability and durability of the porous details [3, 5].

Penka Zlateva, Department of Heat Transfer, Technical University of Varna, 9010 Varna, Bulgaria
Tadeusz Pieczonka, AGH-University of Science & Technology, Cracow, Poland
Marin Stoytchev, PM Laboratory, Institute of Metal Science, Bulgarian Academy of Sciences, Sofia, Bulgaria
Key factors affecting the results of nitriding are the saturation temperature, nitriding time, density and type of the sintered alloy metal matrix and the input size of the pressed powder particles.

Essential for nitrided sintered alloys is not only the total quantity of pores, but rather the nature of porosity. The change in the input powder particle sizes, brought to the same absolute density (porosity) of the sintered alloys to different particle sizes, dramatically modify the degree of saturation. The effect is significant – a reduction of the powder particle size by 2 times causes an increased degree of saturation by 1.5 times, and the modification of particle size by 3 times results in a saturation level increased by 2-3 times [1, 6].

The tendency towards complete saturation of iron sintered products leads to a peculiar phenomenon - a sharp change in their size. It is determined by the degree of saturation, namely the duration of the process and the initial density and sizes of the input particles (pores) of the sintered alloy. At the same density of sintered alloys, the volume change depends on the concentration of nitrogen.

The present research includes examining three main questions in the process of nitration. The first is connected with the study of factors affecting the performance of PM products. For this purpose, it is necessary to carry out microstructural analysis, hardness and microhardness measurements and quantification of phases.

EXPERIMENTAL

A powder mixture of iron powder SC 100.26, 2.0% electrolytic Cu powder (lower than 63 μm) and 0.2% graphite powder (C-UF, Höganäs AB, Sweden) was pressed under 630 MPa. Sintering was carried out in an industrial belt furnace in a protective environment dissociated NH₃, heated to 1120°C, 60 min isothermal holding and cooling to room temperature. Specimens were nitrided in the laboratory at 570°C in retention times of 300 s (5 min), 600 s (10 min) and 900 s (15 min) in NH₃ atmosphere.

RESULTS AND DISCUSSIONS

Studies were carried out on sintered PM articles, nitrided with a stay in the protective gas environment for 300, 600 and 900 s. The results of microstructural analysis of iron samples for duration of 300 and 900s are given in Fig.1a and 1b.

In general saturation of iron with nitrogen phase formation occurs in sequence of alternating single-phase areas, according to the state diagram of Fe-N. The type and composition of phases depends on the saturation temperature. At temperatures lower than the eutectic temperature α-, γ₁-, ε-phases are successively formed. In the transition from one phase to another, the concentration varies unevenly. Nitriding structure analysis shows (Fig.1a and Fig.1b) the following characteristic properties.

The structure of the sample at nitriding temperature 570°C, 300 s, shows (Fig.1a) the following phase composition: αₙ - main quantity; γ₁ - all in the volume of ferrite grains along the entire section; ε - only surface traces. Notably, Fig.1a also shows the presence of αₙ-phase, which is significant, and the initial stages of formation of γ₁-and ε-phases the relative change of the mass is experimentally determined at saturation ($\Delta m / m_o$%) – 0.22%.

Nitriding 900 s sample shows (Fig.1b) the following phase composition: αₙ; γ₁-and ε - phases of the order of 2-3 microns. Saturation along the whole section of the sample is uniform. Penetration of nitrogen into the workpiece is 2.5 mm (Fig.1b) and relative mass change of saturation is 0.75%.
From the data, it can be concluded that the increase in the nitriding time of the sample raises the nitrogen concentration of the surface. Therefore the gas environment stay of the material to its full saturation results in improved output characteristics necessary for operating conditions after the nitriding (durability, corrosion resistance, etc.). Complete saturation of the iron porous material in nitrogen is obtained after 15 min, and the nitrogen saturation of iron rich material in is obtained for 36 hours. The faster saturation of the model is due to the porous nature of the environment. Therefore, the use of PM iron products is desirable as it reduces their manufacturing time and cost.

Figure 2 presents the microhardness figures of the investigated nitriding alloys made of iron powder marks SC 100.26 for 900 s in nitrogen environment.
Figure 3 represents data from X-ray phase analysis of SC 100.26 iron powder after chemi-thermal treatment in a medium of NH$_3$ at 570°C.

Figure 3 indicates that for 300 s in gas atmosphere, $\alpha_N$-phase prevails, there is $\gamma^I$-phase and $\varepsilon$-phase is negligible. For nitrogen retention time of 600s, there is a significant drop in $\alpha_N$-phase and an increase in $\gamma^I$- and $\varepsilon$-phases. According to the radiograph of the iron PM samples remaining in nitrogen atmosphere for 900s, $\alpha_N$-phase decreases, $\gamma^I$-phase is almost the same, and $\varepsilon$-phase increases.

Quantitative assessment of the volume ratios between the phases established after nitriding has been carried out and displayed in Fig.3 [7]. The following numerical values for the $\alpha_N$, $\gamma^I$- and $\varepsilon$ - phase have been obtained and are presented in Table. 1.
Fig.3. Radiograph following the phase analysis of SC 100.26 iron powder after chemical heat treatment in NH$_3$ atmosphere at 570°C.

Tab.1. Phase amount (%).

<table>
<thead>
<tr>
<th>Material</th>
<th>Prevalent Nitride Phases [%]</th>
<th>Nitriding Time [s]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\alpha_N$</td>
<td>$\gamma'$</td>
</tr>
<tr>
<td>SC 100.26</td>
<td>88</td>
<td>11</td>
</tr>
<tr>
<td>SC 100.26</td>
<td>55</td>
<td>35</td>
</tr>
<tr>
<td>SC 100.26 + 0.2% C</td>
<td>19</td>
<td>38</td>
</tr>
<tr>
<td>SC 100.26 + 0.2% C + 2% Cu</td>
<td>11</td>
<td>40</td>
</tr>
</tbody>
</table>

From the results it follows that for iron nitriding samples PM 900s, the degree of saturation is greatest. Consequently, the percentage of nitrogen in these structures is the
highest. It can be claimed that with increasing time of nitration, an increase in $\gamma$- and $\varepsilon$-phases and reduction in $\alpha_N$-phase are observed.

CONCLUSIONS

Taking into consideration the experimental studies, the following conclusions should be noted:

1. The nitrogen containing phases, i.e. $\alpha_N$-, $\gamma$- and $\varepsilon$-phases, were recognised within the surface layer whose thickness increases with nitriding time.
2. Atomic nitrogen, available during nitriding, intensively diffuses into the iron metal matrix.
3. Nitriding of the compacts with 10-15% porosity is stopped after about 900 seconds when the saturation state is achieved.

An iron PM product was obtained with a particular structure of the diffusion layer, which determines the efficiency of the product at the appropriate operating conditions. Thus, the research has defined the PM product’s physical and mechanical properties, depending mainly on the structure and phase composition of the surface layer determined by the parameters of HTO in the process - temperature, length of process, etc., and furthermore the type of treated material. A diffusion layer was obtained, with a structure suitable for the operation of the iron PM product in a suitable (corrosion resistance and durable) environment.

REFERENCES