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Nitriding of multilayer steel materials

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Abstract. The paper presents results of the metallographic study aimed to determine the impact of the structurization degree of multilayer metal materials on the parameters of diffused layers in the course of thermochemical treatment by nitriding. It is demonstrated that reduction of the laminar layer thickness from 100 to 2 μm results in more than twofold increase of the diffused layer in comparison with the reference polycrystalline sample. It is determined that more significant structure refinement for multilayer materials particularly to the estimated layer thickness of 0.3 μm massively reduces the depth of diffusant penetration into the material along the laminar boundaries.

1. Introduction

The essential scientific and technological objective of the present-day machinery engineering is to reduce the dimensions and weight of the parts and structural components. Solution of this task will enable enhancement of performance characteristics of the entire product significantly. Experience has proven that application of the materials characterized by the presence of a gradient macro- or microstructure remains quite efficient in the development of advanced state-of-the-art equipment items. They may include the new class of structural metal materials produced by synthesis of multilayer composite blanks. Application of such materials considerably extends the operational life of parts and structures operating under high temperature and force loads with a simultaneous saving of expensive alloying elements. In this case implementation of the set of very high-performance characteristics in the equipment parts is a significant advantage as these characteristics are impossible to achieve by application of monometallic materials.

Along with well-known production methods for composite multilayer materials [1-3], homogeneous materials consisting of hundreds and thousands of layers separated by large-scale angular boundaries may prove to be advantageous.

The undertaken studies demonstrated that it was possible to obtain such a structure particularly when same-metal-based alloys with different crystalline structure (BCC and FCC lattices) were used in the initial composition. The range of technically significant alloys is considerably extended if hot rolling is used as the basic type of processing in contrast to well-known process flowcharts [4]. In this case, deforming can be performed within the temperature range where the initial components of the composite blank retain different types of crystalline lattices. Application of the developed experimental process flow enables to produce strip blanks with a width of 100 mm and a thickness of 2 to 10 mm. The microstructure of the material is laminar which may be characterized as alternating same-metal-based layers with an identical or similar chemical composition separated by large-scale angular boundaries with the layer thickness of 100 to 0.3 μm (according to the calculation).

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Theory and practice of thermochemical treatment (TCT) show that irregularities of the crystalline structure have a significant impact on the alloying element diffusion processes. It is known that the grain-boundary diffusion coefficient value in metal materials exceeds the volume diffusion coefficient values by an order of magnitude. Thus the idea to form a peculiar type of regular microstructure with the configuration enabling to increase the rate and depth of alloying element penetration in surface impregnation of the equipment parts may prove to be quite advantageous in spite of any potential difficulties.

This configuration may be illustrated through the example of a cog-wheel made of a multilayer strip blank (Figure 1).

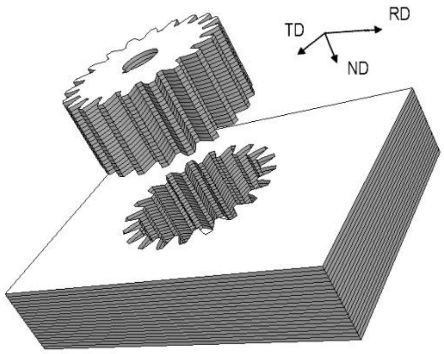
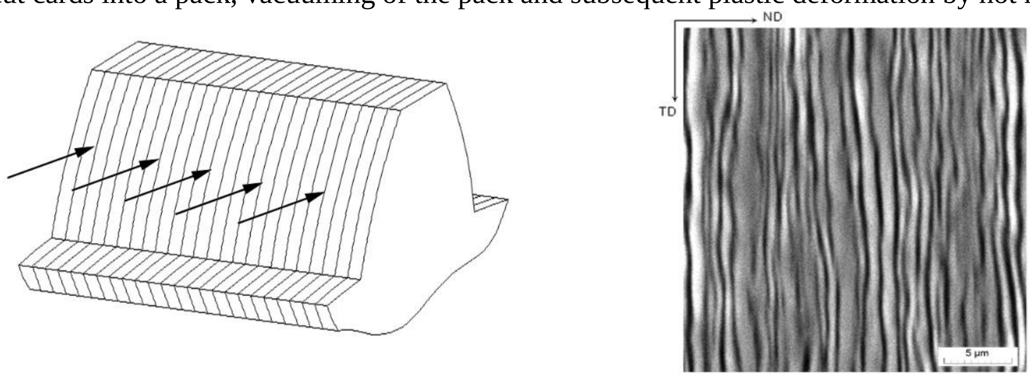


Figure 1. A scheme of cog-wheel cutting out of a multilayer steel blank.

It can be seen that the working surface hardened by diffusion alloying has multilayer configuration (Figure 2); taking into account the position of large-scale angular (interlayer) boundaries predominant accelerated penetration of alloying elements exactly through these additional channels may be expected. However, it is evident that the efficiency of thermochemical treatment, in this case, will depend on many factors and primarily on the number and thickness of laminar layers on the working surface of the part.

2. Materials and methods

Multilayer composite blanks consisting of 100 alternating cards made of steels AISI 430 and AISI 304 with the thickness of 0.5 mm, the length of 200 and the width of 50 mm (50 pieces of each steel grade) at the beginning of the manufacturing cycle were used to conduct the studies. The above-mentioned blanks were subjected to three complete manufacturing cycles in accordance with the experimental process flow including cutting of the cards to length out of sheets, their surface treatment, assembly of the cut cards into a pack, vacuuming of the pack and subsequent plastic deformation by hot rolling [5].



(a) (b)

Figure 2. A scheme of the cog-wheel tooth working surface (a) and the electronic image of the impregnated surface structure of the multilayer material (b). Nitrogen diffusion direction is shown with arrows.

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Samples obtained after the first manufacturing cycle with the layer thickness of 100 and 20 μm respectively, samples obtained after the second manufacturing cycle with the layer thickness of ~2 μm and also samples after the third manufacturing cycle with the estimated layer thickness of 0.3 μm were selected as the study subjects.

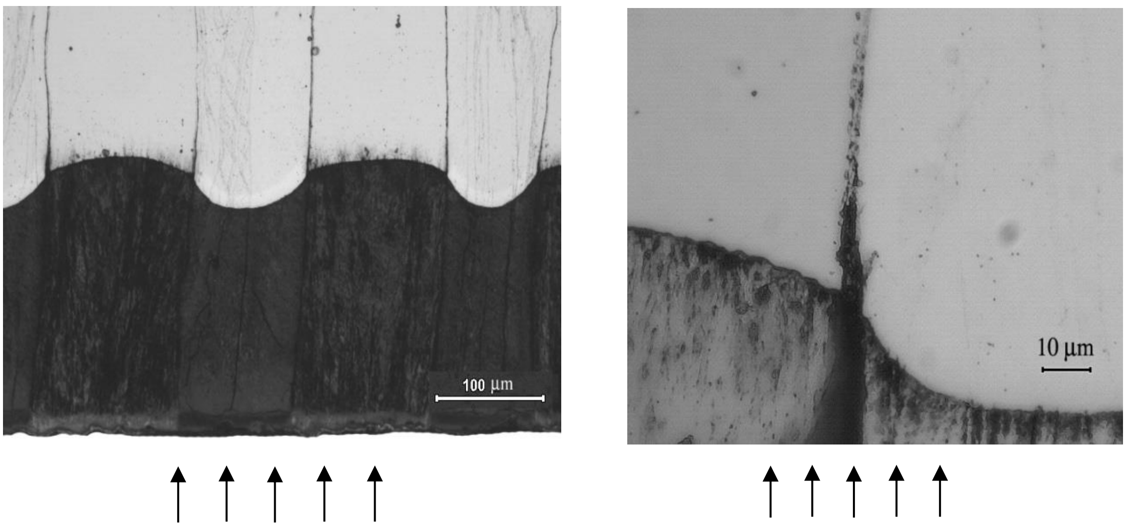
Nitriding was carried out in a gas environment at the temperature of 540 °C with the ammonia dissociation degree of 20 to 40% for 45 hours. All studied samples were processed in a single batch, and in this case, a sample of AISI 304 steel with ordinary polycrystalline structure was used as a reference.

Microstructure of the material after nitriding was studied through the use of an optical microscope on the basis of the samples cut out along the diffusion flow direction and perpendicular to the assumed working surface (Figure 2) and also through the use of a scanning electronic microscope VEGA TS5130 in the secondary electron mode at the acceleration voltage of 5 to 20 kV.

3. Results and discussion

Microstructure studies for the reference sample of steel AISI 304 showed that the thickness of the obtained nitrated layer was approximately 130 μm corresponding to well-known reference data and literature reports.

Microstructure studies for multilayer samples showed that the diffusant penetration depth in the course of nitriding depended on the thickness of laminar material layers. It was determined that the nitrogen penetration depth in multilayer samples with the laminar layer thickness of 100 μm was approximately 150 μm on an average (Figure 3a). Diffusion front of nitrogen penetrating in advance along the boundaries of laminar layers is manifested rather clearly in case of chemical etching (Figure 3b). In this case, it can be seen that the front has various curvature - concave meniscus in AISI 304 steel layers and convex meniscus in AISI 430 steel. Based on the Fisher model one may suggest that the convex meniscus in AISI 430 steel is formed due to the combination of two diffusion flows – the flow of nitrogen moving within the layer volume and the flow diffusing from the laminar boundary [6].



(a) (b)

Figure 3. The microstructure of the nitrated layer in multilayer material (a). Predominant nitrogen penetration along the interlayer boundary (b). The nitrogen diffusion direction is shown with arrows.

In this case, the meniscus curvature sign is determined by the difference in the nitrogen volume diffusion coefficients in steels, that is why the layers of AISI 430 steel more permeable for nitrogen than AISI 304 steel are subjected to concentration "pressure" from the direction of the laminar boundary bending the diffusion front accordingly. The same pattern, in general, is retained in the multilayer

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samples with the laminar layer thickness of 20 μm (Figure 4). But the nitrogen penetration depth is increased up to approximately 200 μm conforming the proposed mechanism of the interlayer boundary impact when the nitrogen flow from this boundary impregnates the thinner laminar layer at a higher rate thus promoting increased concentration gradient in the layer within the shorter period of time.

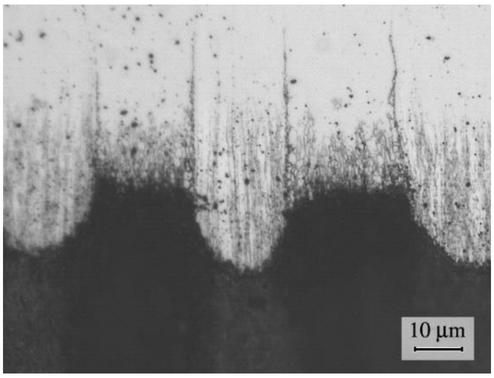

Figure 4. The microstructure of the nitrated layer in multilayer material (laminar layer thickness of 20 μm).

It also comes under notice that the depth of nitrogen penetration into the laminar layer along the structural defects is comparable to the depth of penetration along the interlayer boundaries; in this case, it is greater in AISI 304 steel which is more resistant to nitriding than AISI 430 steel.

Nitriding of the samples with the laminar layer thickness of 2 μm results in even more significant increase of the nitrated layer thickness up to 280 μm on an average; in this case, predominant diffusion along the boundaries of laminar layers remains (Figure 5).

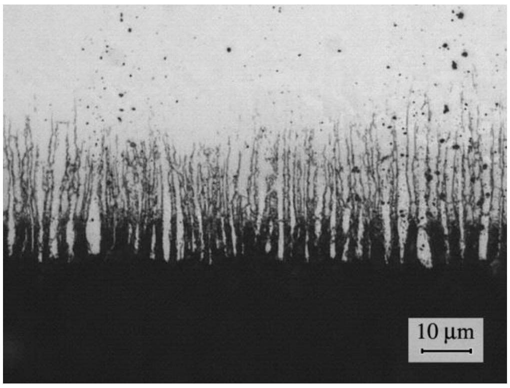

Figure 5. The microstructure of the nitrated layer in multilayer material (laminar layer thickness of 2 μm).

The pattern changes drastically with impregnation of the samples after three manufacturing cycles having the estimated layer thickness of 0.3 μm. In this case, the depth of nitrogen penetration into the material is sharply reduced up to the values not exceeding 30 μm that is considerably below even the thickness of the nitrated layer in the reference sample of AISI 304 steel. It may be supposed that processes related to rearrangement of the crystalline lattice can have such strong inhibiting effect on the diffusing substance flow. In this case recrystallization under these conditions is evidently due not only to change of the phase composition but also to low-temperature stability of the multilayer material with a high degree of structurization (Figure 6).

In view of the foregoing, one may suggest that there is a certain critical thickness of a laminar layer in multilayer materials (h_{cr}) overranging of which results in reduction of efficiency for thermochemical treatment by nitriding. For the given composition of the multilayer material, this thickness is within the range of 2 to 0.3 μm.

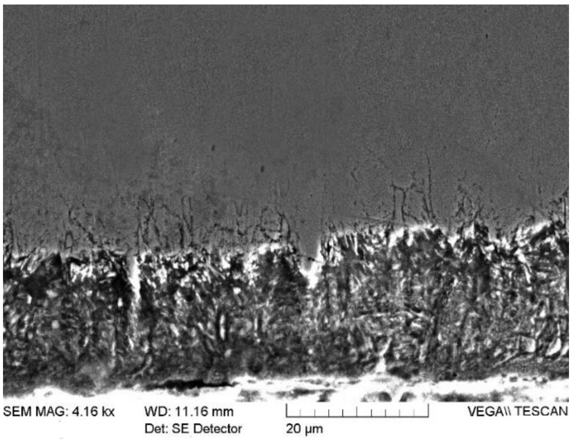

Figure 6. The microstructure of the nitrated layer in multilayer material (laminar layer thickness of 20 m).

4. Conclusion

The undertaken studies showed that the diffusant penetration depth in the course of nitriding depended on the thickness of laminar material layers. It was determined that reduction of laminar layer thickness from 100 to 2 μm results in more than a twofold increase of the diffused layer in comparison with the reference polycrystalline sample. At the same time more significant structure refinement for multilayer materials particularly to the estimated layer thickness of 0.3 μm massively reduces the depth of diffusant penetration into the material along the laminar boundaries. On the basis of the foregoing, existence of certain critical laminar layer thickness may be supposed, and overranging of this thickness makes thermochemical treatment by nitriding inefficient.

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