

HS 6-5-2 STEEL SURFACE LAYER DEVELOPMENT IN CARBONITRIDING WITH ZEROFLOW METHOD

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Original scientific paper

The paper reviews the process of surface layer development with the use of carbonitriding and carbonitriding with oxidation. Employed for that purpose was a modern and ecological method called ZeroFlow. Research was carried out using HS 6-5-2 steel. Single-component nitrifying atmosphere of ammonia NH_3 was applied. The temperature of the processes was 570 °C. Diffusion layers were obtained on the steel. The thickness of the layers achieved during the microstructural research equalled from about 125 μm after carbonitriding with oxidation to about 187 μm after carbonitriding. Surface hardness HV_1 was the highest for the carbonitrided layer - 1027 HV_1 , whereas in the case of carbonitriding with oxidation it amounted to 1018 HV_1 . As far as surface hardness HV_{30} is concerned, it was at the level of 813 HV_{30} for the carbonitrided layer and 795 HV_{30} for carbonitriding with oxidation. Surface hardness test results indicate surface layer hardening.

Keywords: carbonitriding; microhardness; nitriding; oxidation; surface layer

Dobivanje površinskog sloja HS 6-5-2 čelika karbonitriranjem uz primjenu metode ZeroFlow

Izvorni znanstveni članak

U radu se opisuje postupak dobivanja površinskog sloja primjenom karbonitriranja i karbonitriranja s oksidacijom uz pomoć suvremene i ekološke metode poznate kao ZeroFlow. Istraživanje je provedeno uporabom čelika HS 6-5-2. Primijenjena je jednokomponentna nitrificirajuća atmosfera amonijaka NH_3 . Postupak se vodio na temperaturi 570 °C. Na čeliku su nastali difuzijski slojevi. Debljina slojeva dobivenih tijekom mikrostrukturnog istraživanja bila je od oko 125 μm nakon karbonitriranja s oksidacijom do oko 187 μm nakon karbonitriranja. Površinska hrapavost HV_1 bila je najviša za karbonitrirani sloj - 1027 HV_1 , dok je u slučaju karbonitriranja s oksidacijom iznosila 1018 HV_1 . Kad se radi o površinskoj tvrdoći HV_{30} , ona je za karbonitrirani sloj bila na nivou od 813 HV_{30} , a kod karbonitriranja s oksidacijom iznosila je 795 HV_{30} . Rezultati ispitivanja površinske tvrdoće ukazuju na otvrdnjavanje površinskog sloja.

Ključne riječi: površinski sloj; nitiranje; karbonitriranje; oksidacija; mikrotvrdoća

1 Introduction

One of the principal methods of surface integrity hardening encountered in industrial applications is thermochemical treatment [1 ÷ 3]. Obtaining the desired surface quality is a very important parameter for the functional maintenance of a part [4]. The most hardened parts in industrial conditions are: drills, screw taps and dies [5]. What seems to be a common problem in industrial facility is how to increase the durability of the tools in use. Cutting tools and industrial blades can serve as an example [6]. From the production point of view, one of the fundamental criteria for tool selection is its operational durability, whose high level can be established by choosing suitable ways of its enhancement. The popularity of thermo-chemical treatment of steel through nitriding provoked a remarkable development of nitriding methods. Currently, there are various nitriding methods differing mainly in the ways of obtaining nitriding atmosphere and process parameters [7]. Another significant factor worth mentioning is the possibility to conduct nitriding with a simultaneous saturation with other chemical elements e.g. carbon [8]. Producing surface layers through a controlled gas carbonitriding with the use of a ZeroFlow method [9] is one of such methods. Development of new materials and their properties is important for the diagnostics of all machines [10].

ZeroFlow is a modern and ecological as well as less expensive method compared to other conventional nitriding processes [11]. What has been used most frequently in the existing methods of gas nitriding is a process based on controlled two-component atmospheres

– dissociated $\text{NH}_3 + \text{NH}_3$ and $\text{NH}_3 + \text{N}_2$. ZeroFlow is based on conducting a controlled nitriding process with the use of a single-component atmosphere in the form of ammonia NH_3 which results in a reduction of process costs. First time this method was described by Małdziński et al. [12]. The processes of controlled gas nitriding are predominantly run in a two-step manner. The first one can be characterized by an intensive nitriding atmosphere, meaning a high nitriding potential, and is carried out at a lower temperature. In this stage a surface layer is saturated with nitrogen and a several-micrometer-thick layer of iron nitride is developed. The second one takes place at a higher temperature, in a nitriding atmosphere of a lower intensity of nitriding. In this stage, the area of internal nitriding expands and a layer of iron nitride as well as a nitriding atmosphere serve as the source of nitrogen. At the same time, a slight increase of an iron nitride layer occurs at the surface [13, 14]. In the ZeroFlow method, the regulation of an atmosphere chemical composition in a retort (regulation of a nitrogen potential) undergoes exclusively through the regulation of the intensity of ammonia flow through the retort. There are several advantages of the ZeroFlow method, which are minimum consumption of work gas and a simplified gas installation which have a positive influence on reducing the costs of its operation. During the nitriding process it is possible to saturate the surface with other chemical elements too. Carbonitriding with the use of the ZeroFlow method is based on simultaneous surface saturation with nitrogen and carbon. Applying ZeroFlow carbonitriding method in order to harden surface layers employed in a tool industry can have a positive influence on their costs and operational durability.

ZeroFlow method of controlled nitriding and gas carbonitriding as well as carbonitriding with oxidation can be adopted in both laboratory and industrial environments. What is striven for in the case of tool steels and High Speed Steel (HSS) is obtaining a diffusion layer with the help of a nitriding method without or with a minimum thickness of an iron nitride surface layer [15]. The thickness of a surface layer usually amounts up to 0.3 mm [16].

The paper raises issues regarding hardening of a High Speed Steel HS 6-5-2 through the use of a controlled gas carbonitriding of the ZeroFlow method.

2 Material and methods

HS 6-5-2 steel was adopted as a research material. It is HSS applied for tools and machine parts with a dynamic load and operating in an elevated temperature. The chemical composition of HS 6-5-2 steel is shown in Tab. 1.

Table 1 Chemical composition of HS 6-5-2 steel / %wt.

C	Si	Mn	Cr	Mo	Ni	V	W	S	P
0,82 to 0,92	Max 0,5	Max 0,4	3,5 to 4,5	4,5 to 5,5	Max 0,4	1,7 to 2,1	6,0 to 7,0	Max 0,03	Max 0,03

Applied processes of thermo-chemical treatment were carbonitriding and carbonitriding with oxidation. Research samples were cleaned in acetone. Carbonitriding and carbonitriding with oxidation were carried out with the use of a Seco/Warwick industrial oven for controlled ZeroFlow method gas nitriding with a possibility to control the parameters of the process such as its time, atmosphere, temperature and nitriding potential. They were monitored and recorded at every stage of the process. Control of a chemical composition of a nitriding atmosphere was carried out by means of periodic closing and opening inflows of ammonia NH₃ and CO₂ into a retort. The processes were led in a two-step manner. The maximum temperature was 570 °C, the total time of the I and II stage equalled 360 min., whereas nitriding potential amounted to 12. Fig. 1 presents a multi-task and professional industrial station for nitriding with the use of the ZeroFlow method. Nitriding was conducted at The Center of Engineering Studies of The Institute of Technical Sciences and Aviation in The State School of Higher Education in Chelm.

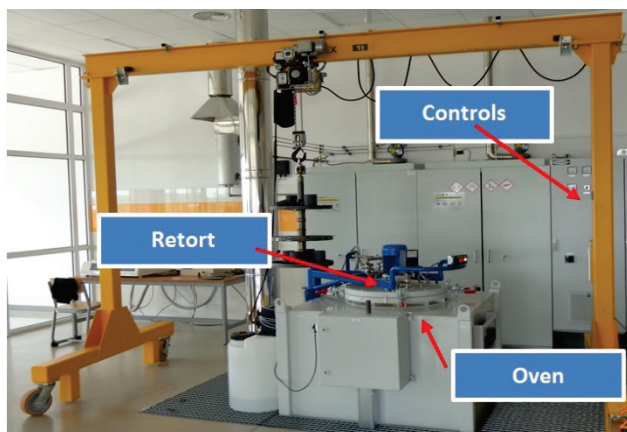


Figure 1 ZeroFlow method nitriding station

Obtained layers were characterised through analysing the microstructure, surface hardness and a hardness traverse of a surface layer. A device used for hardness study was a Wolpert-Wilson Tukon 2500 microhardness tester. Vickers method was applied. Surface hardness was determined for a load equalling 1 kg (HV₁) and 30 kg (HV₃₀). Measurements were performed in various areas of the analysed layers. Measurement results represent average values obtained from 30 measurements carried out on a surface of an analysed sample after a suitable process of thermo-chemical treatment. Hardness traverse of a surface layer was measured for a load at the level of 0,5 kg (HV_{0,5}). The samples were prepared using standard metallographic techniques [17] by grinding, polishing, and etching. A device adopted for the purpose of a microstructural research was Nikon Eclipse MA 200. The focus of the observation was the thickness of the layers in cross section of the samples. The samples are presented in Fig. 2.

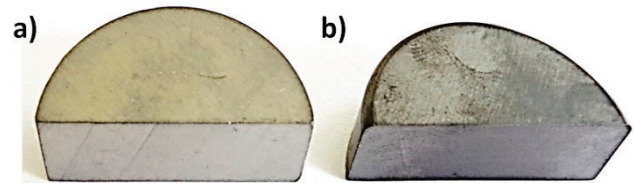


Figure 2 The view of the samples: a) after carbonitriding, b) after carbonitriding with oxidation

3 Discussion

Regulating growth kinetics of the layers was conducted by controlling the parameters of the process. HS 6-5-2 steel was subject to carbonitriding and carbonitriding with oxidation according to the parameters presented in Tab. 2. For every procedure, the temperature, time and a nitriding potential were provided. Programmed nitriding time was 8,35 hours after carbonitriding and 9,47 hours after carbonitriding with oxidation. The overall duration of the processes (heating, (reaching), warming (in the air, in nitrogen), nitriding, nitrogen purging and cooling) was higher and equalled 18 hours after carbonitriding and 15 hours after carbonitriding with oxidation. The temperature of the first stage was 490 °C in the time of 60 minutes. Its nitriding potential was 12. In the case of the second stage, the temperature reached 570 °C, the time of the process was 300 min. and its nitriding potential 1.

After carbonitriding of HS 6-5-2 steel, the properties of the obtained layers were analysed. What happens as a result of carbonitriding and carbonitriding with oxidation is a considerable hardening and a beneficial change in mechanical properties of HS 6-5-2 steel. The evaluation of HS 6-5-2 surface hardness was carried out with the help of two loads - 1 kg (HV₁) and 30 kg (HV₃₀). Surface hardness HV₁ was the highest for a carbonitrided layer and it equalled 1027 HV₁. The layer which was subjected to carbonitriding with oxidation had a slightly lower surface hardness amounting to 1018 HV₁. A similar situation could be observed in the case of surface hardness HV₃₀. After carbonitriding, it was 813 HV₃₀, whereas after carbonitriding with oxidation - 795 HV₃₀. Having determined surface hardness, the thickness of the

obtained layers was examined. For that purpose, hardness traverse of a surface layer was studied and microstructural tests were carried out. Hardness examination was performed with the use of the Vickers method under the

load of 0,5 kg ($HV_{0,5}$). Hardness traverse of proper layers was examined five times. Results are presented in Fig. 3. Hardness profiles constitute average values obtained for every sample.

Table 2 The parameters of the process of controlled carbonitriding and carbonitriding with oxidation with the use of the ZeroFlow method

Item number	Procedure	Carbonitriding			Carbonitriding with oxidation		
		Temp.	Duration	Order no.	Temp.	Duration	Order no.
		°C	Min.	-	°C	Min.	-
1	ONE-GAS NITRIDING	490	60	12	490	60	12
2	TWO-GAS NITRIDING	570	300	1	570	300	1
3	NITROGEN PURGING WITH WARMING*	-	-	-	520	30	-
4	WATER OXIDATION*	-	-	-	520	2	-
5	WATER OXIDATION*	-	-	-	520	40	-

* procedures for carbonitriding with oxidation

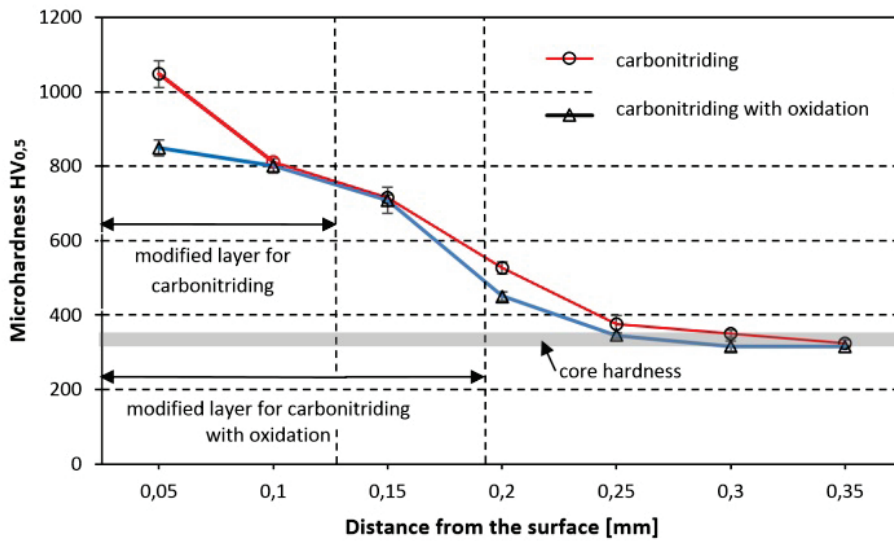


Figure 3 Hardness profiles for HS 6-5-2 steel

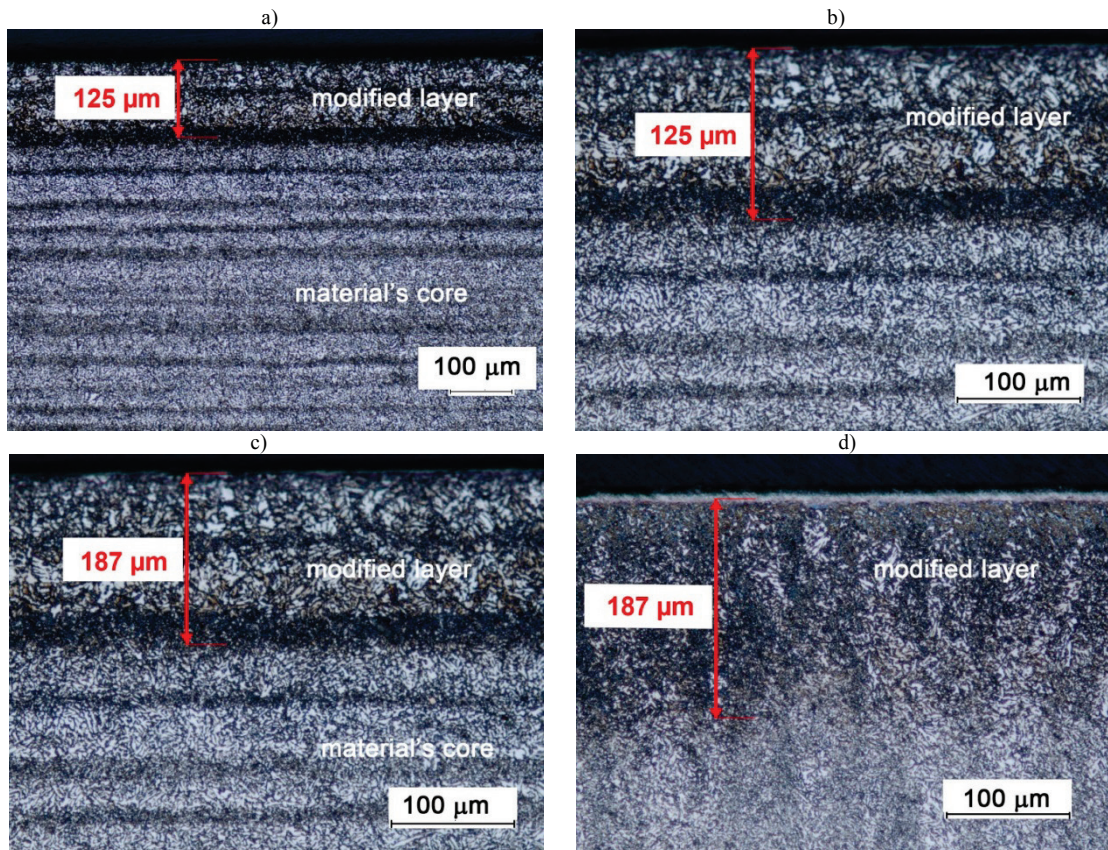


Figure 4 HS 6-5-2 steel diffusion layer thickness values: a, b) after carbonitriding, c, d) after carbonitriding with oxidation (magn. $\times 200$)

Core hardness of the HS 6-5-2 steel samples under examination was 350 HV_{0,5}. Applying layer thickness criterion, which was core hardness + 50 HV_{0,5}, the thickness of all the obtained diffusion layers equalled about 0,25 mm. The highest thickness amounting to 1048 HV_{0,5} was at the depth of 0,05 mm. It was lower for the layer subjected to carbonitriding with oxidation - 838 HV_{0,5}. What served as a verification of the achieved results was a microstructural research. Diffusion layer thickness values after carbonitriding and carbonitriding with oxidation are presented in Fig. 4.

What was observed while analysing research results is that the thickness of a layer after carbonitriding was about 125 µm (Fig. 4a), whereas the thickness of a diffusion layer after carbonitriding with oxidation was approximately 187 µm (Fig. 4c). As a result of the undertaken research, relatively even hardened surface layers were obtained. A thin surface layer of iron nitride was observed in the pictures, constituting so-called "white layer" whose thickness was ranging from a few to several µm (Fig. 4b, d). The white layer was more distinctive in the case of carbonitriding with oxidation (Fig. 4d)

4 Conclusions

The results obtained show that there is the possibility to apply carbonitriding and carbonitriding with oxidation with the use of the ZeroFlow method in order to develop properties of a technological surface layer on a HS 6-5-2 High Speed Steel. As a result of the conducted research the following final conclusions can be drawn:

- 1) Relatively even and repetitive surface layer hardness values were obtained for the following processes: carbonitriding and carbonitriding with oxidation.
- 2) Thickness values of the layers obtained during the microstructural research varied from about 125 µm after carbonitriding with oxidation to about 187 µm after carbonitriding.
- 3) As to the layer thickness criterion which is core hardness + 50 HV_{0,5}, the thickness of the obtained diffusion layers equalled about 0,25 mm.
- 4) Surface hardness HV₁ was the highest for a carbonitrided layer (1027 HV₁), it was 1018 HV₁ as far as carbonitriding with oxidation is concerned. As for surface hardness HV₃₀, it was at the level of 813 HV₃₀ for a carbonitrided layer and 795 HV₃₀ for a layer after carbonitriding with oxidation.
- 5) Having analysed research results, it can be stated that the hardening of a surface layer of HS 6-5-2 High Speed Steel with the use of controlled gas carbonitriding as well as carbonitriding with oxidation where ZeroFlow method is applied is highly advantageous. Layers obtained in such a way have a considerable potential in a wide range of applications. The main benefit resulting from their use is the improvement of mechanical properties of machine and tool parts made of HS 6-5-2 steel.

This project and its results also established the basis for further studies of surface integrity after thermochemical treatment and the relation of technological parameters to surface properties.

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