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Vacuum carburizing – process optimization

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Abstract: Recognition of physical and chemical processes during low-pressure carburizing enables a precise process control with the aid of computer simulation. The results of the investigations aiming in better recognition of the phenomena during the process of carburization as well as their effect on the possibility of prediction of carbon concentration after the process are presented in the work. This in turn allowed selection of optimal parameters of the low-pressure process and creation of a model for process simulation. A comparison of the experimental carbon concentration profiles with the simulated ones for plain carbon and alloyed steels are given in the work as well.

Keywords: Vacuum carburizing; Computer simulation; Surface phenomena.

1. INTRODUCTION

The vacuum carburizing is characterized by quick saturation in carbon atoms of the steel surface layer. The hydrocarbon decomposition proceeds in avalanche by means of different chemical reactions [2,4,5]. Due to the intrinsic unequilibrium of these processes it is impossible to apply the conventional methods of process control. That is the reason that the computer simulations are being used for the process control. A detailed analysis of the flux of carbon atoms delivered to the surface under carburization has led to an important conclusion. It has been found that an effective use of carbon atoms process delivered to the surface is possible only if the whole process is divided into two stages: (1) a boost stage, in which an important amount of carbon atoms from the carburizing atmosphere is being delivered to the surface and (2) a diffusion stage consisting in holding the charge at the same process temperature without further supply of the gas components of the atmosphere [6]. Such a partitioning of the carburizing process is more advantageous because the solubility of carbon atoms in austenite is limited and in excess of carbon atoms a solid carbonaceous layer is being created on the surface of the steel [1,3]. The influence of the carbonaceous deposit onto the carbon concentration profile in the surface layer is not taken into account in the assumptions of the computer simulation, therefore it is crucial that the the maximum solubility of carbon in the austenite is not overstepped.

2. EXPERIMENTAL

The samples for carburization made of two different steel grades - C20 mild steel and the alloyed one 20CrMnTi5-4-1 - were of cylindrical shape of the dimensions $\text{Ø}20 \times 100$ mm, and, additionally, thin foils of the thickness 0,2 mm made of Armco iron. The samples vacuum carburized at 920°C and 950°C.

During the first experiments the carburizing consisted of single boost within the time intervals of 5, 15 and 120 minutes with subsequent direct gas quenching from the carburizing temperature. In the next experiments the substrates $\text{Ø}20 \times 100$ mm were carburized with alternating boost and diffusion stages in order to compare the obtained carbon concentration profiles with the simulated ones. The time intervals of the boost/diffusion stages were: 20/10, 5/15, 5/20, 5/25, 5/35 or 5/15 minutes. The carburizing atmosphere in all the experiments consisted of ethylene and acetylene mixtures diluted in hydrogen, optimally selected on the basis of the experimental data [4].

After carburizing process the carbon content in thin foils was measured with use of LECO CS-200 carbon and sulphur analyzer and the metallographic inspection has been made on cylindrical specimens. The carbonaceous deposits found in a number of processes have been examined by means of TOF-SIMS method (secondary ions mass spectroscopy with time of flight analysis). Metallographic analysis of the carburized layers have been performed as well.

3. RESULTS

The carbon concentration in Armco foils achieves the maximum solubility in austenite at a given temperature in conformity with iron-graphite line E'S' of the binary Fe-C diagram after a time as short as few minutes (Figure 1). If the carbon potential of the atmosphere exceeds the maximum solubility in the austenite, the surface of the specimens has gray a gray tint characteristic for the carbonaceous deposit.

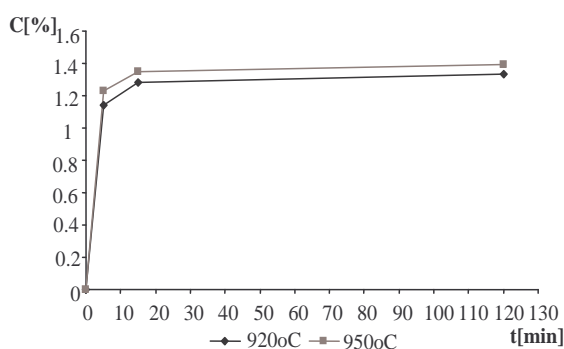


Figure 1. Carbon concentration in the Armco foil as a function of the dwell time in in the carburizing atmosphere

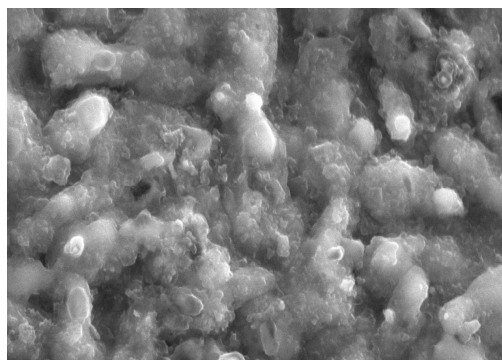


Figure 2. BSE image of the Armco specimen after 120 min low pressure carburizing at 950°C with carbonaceous deposit on the specimen surface

The deposit consists mainly of: hydrocarbon groups CH_n , represented by methyl group CH_3 (of the reduced mass of the group $m/z=15$, where m corresponds to the mass of the group and z – the ionisation level), methylene CH_2 ($m/z=14$) and CH ($m/z=13$), single C atoms ($m/z=12$), hydrogen atoms ($m/z=1$) and the groups of atoms Fe-H ($m/z=57$). The complexes with two carbon atoms are represented by C_2H_3 ($m/z=27$) and C_2H_5 ($m/z=29$) groups (Figure 5). The hydrocarbon groups

containing three carbon atoms are represented by complexes C_3H_5 ($m/z=41$) and C_3H_7 ($m/z=43$). Both the C_1 and C_3 hydrocarbons in the deposits are the proofs that the double $C=C$ bonds of the primary ethylene molecules adsorbed on the iron surface have been broken. The C_2 complexes can represent the dimerization or aromatization reactions. The concentration of hydrocarbon complexes containing four and more carbon atoms on the sample surface is very low. Ions of the reduced mass m/z 13÷97 detected among the positive ions correspond to the fragments $CH_x\div C_7H_x$ (where x is a number from the interval 1÷13) is a proof that hydrocarbons of different degree of saturation are present on the carburized surface.

After the maximum carbon solubility in alloyed austenite has been overstepped the surface layer of steels containing alloying elements of high chemical affinity to carbon is probably covered with thin carbide layer (Figure 3) and the carbonaceous deposit probably is being created only later on. At a very short carburizing time (less than 5 minutes) the carbide layer was not observed. For the mild steel, regardless of the time interval of the boost stage, the carbides do not appear on the surface and the excess of carbon precipitates in form of carbonaceous deposit.

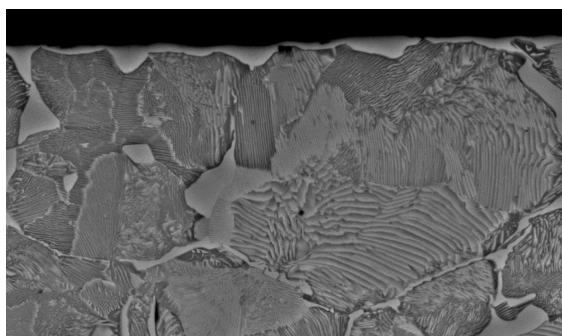
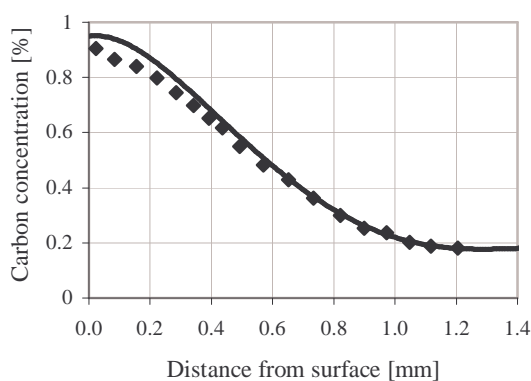


Figure 3. SEI image of the micro-structure of the layer in the vicinity of the surface of 20CrMnTi 5-4-1 steel after 15 minutes vacuum carburizing at 950°C. Magn. x 3500



◆ - real carbon concentration profile
 - - simulation carbon concentration profile

Figure 4. The real and simulated carbon concentration profiles for 20CrMnTi 5-4-1 steel vacuum carburized at 920°C with use of the time intervals of saturation/diffusion: 20/10, 5/15, 5/20, 5/25, 5/35 and 5/15 (in minutes).

The comparison of carbon profiles obtained in alloyed and mild steels after vacuum carburizing with the ones obtained from computer simulation confirms the earlier observations concerning different surface phenomena taking place during carburizing.

In comparison with the results of simulation the real carbon concentration profile for the C20 steel shows a small differences coming from the carbonaceous deposit on the steel surface, whereas they are considerably greater for the 20CrMnTi 5-4-1 steel (Figure 4). The different behaviour of the alloyed steel in comparison with the non-alloyed one during carburization results from the joint influence of carbides nucleation of the alloying elements and of the carbonaceous deposit on the surface which, in consequence, change the carbon distribution in the vicinity of the surface layer of alloyed steel.

4. CONCLUSIONS

The different phenomena which occur on the surface of the steel during carburizing influence the process to a great extent and, at the same time, make difficult the process control. The selection of an optimum time of the boost stage is still more difficult in case of carburization of alloyed steels. The time interval of the boost stage should be not too long in order that the carbon concentration on the surface does not exceed the maximum solubility of carbon in the austenite. If however it exceeds this limit, the carbides of the alloying elements and carbon deposit appear on the surface. This in turn changes rapidly the mechanisms of carbon diffusion, decreases the delivery of "fresh" carbon atoms to the surface and in consequence makes difficult obtaining the layers of pre-determined properties.

The precipitation of only carbon deposit on the surface is not as disadvantageous phenomenon and does not influence the carbon profile significantly, because it is itself a source of carbon atoms due to temperature activated hydrocarbon decomposition in the deposit. Nevertheless it is of importance for the process control because it has to be taken into account in that if it does exist on the specimens surface then the first minutes of the boost stage would look similarly to the diffusion stage until the carbon deposit was used up.

These are the reasons that it is difficult to predict the carbon profile in the steel after carburizing at the conditions at which the maximum solubility of carbon in austenite is exceeded. The computer programs for simulation of carburizing processes currently available at the market are based on the non-reactive diffusion assumptions, which do not consider the nucleation of carbides on the steel surface. The reason is, first of all, the high complexity of relevant mathematical models of carburizing and, as a result, a decrease of the rate of calculation. Therefore it is important to consider in the simulation model the maximum solubility of carbon in austenite. In that case the time interval of the boost stage should be short enough in order to not exceed the maximum solubility of carbon and, at the same time, as long as possible to minimize the time consumption for the whole process of carburizing.

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