RECENT J. (2022), 68:112-117
https://doi.org/10.31926/RECENT.2022.68.112

Pack Carburizing Effect on Microstructure and Hardness of 1.7131 Steel

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Abstract
Pack carburizing technology was used for case hardening of 1.7131 steel at 930 °C in different condition. Effects of holding time (2-, 4-, 6- hours), quenching environment (air, water) and annealing temperature (170 °C) on microstructures, case and core hardness were investigated. All samples were characterized by the appearance of four zones with different morphology (intermetallic compound, hypereutectoid, eutectoid and hypoeutectoid), caused by carbon diffusion and quenching parameters from the carburizing temperature. An adapted setup of pack carburizing holder was performed to apply direct water quenching. Carbon distribution was calculated using Fick's law and effective case depths were identified, agreeing with the measured hardness values. The most significant hardness values were measured at approximatively 0.5 mm depth 55-65 HRC (590-850 HV). Depending on holding time, high hardness values were recorded also after 1 mm case depth. The advantages of using pack carburizing for low-carbon or low-alloy steels are the low-cost processing and the combination of high surface hardness and high core toughness needed in the parts durability.

Keywords
pack carburizing, 1.7131 steel (16MnCr5), diffusion, carbon distribution

1. Introduction

Considering the final production cost, choosing the right processing technology of a specific product can assure the durability and high performance in use. The continuous study of both metallic materials and processing technologies ensures the evolution of new application needed in all engineering fields. There are several ferrous and non-ferrous metallic alloys which associated with specific thermal and, or thermochemical treatments will allow to achieve adequate properties for the final product [1, 2].

Guiding elements, cores, machine parts as gears which are the most important power transmission elements are mandatory [3] subjected to various heating treatments that also can involve placing them in surface enrichment environments with different alloying elements [2, 5]. The benefit of the thermochemical treatments, especially in the low-carbon and low-alloy steels is the combination of high surface hardness and high core toughness that give lifetime endurance. Case-hardening (surface hardening) of aforementioned types of steels can be accomplished by carburizing process.

According to Fe-Fe_3C phase diagram, diffusion of carbon atoms is possible in austenitic range at the upper temperature of the C solubility in Fe-γ, when a specific part is heated up to 850°C in a surrounding environmental capable to release C atoms [1, 3-5]. Dual properties (hardened surface layer vs. soft and tough core) of the same steel part is reached according to the martensitic microstructure of the outer place of the part and ferritic-pearlitic microstructure in the core section [1, 6].

Steels usually used for carburizing must not exceed 0.3 wt.%C while the target for carburizing layer being between 0.8-1.0 wt.%C. The carburizing deepness of the parts must meet the requirements that usually are between 0.5-2.5mm [1, 4].

Carburizing methods are based on various technologies where the carbon can be released by using an atmosphere (gas, plasma, vacuum), liquids (salt bath) or solid compounds (pack) [4]. Most commonly used technology is gas carburizing followed by pack carburizing, on the last place being plasma carburizing due to highest costs (of equipment and monitoring installation) from all the methods described above. The advantages of pack carburizing consist in process simplicity and low-cost materials and installation used compared with all other methods. The messy of the solid powder compounds, longer processing time, difficulty in part elimination from the holder to apply direct quenching and difficulty to control the layer deepness are the process limitation. Although the pack carburizing is the oldest methods from all mentioned above, it has the advantage of a safety method for students practice in university laboratories, compared with other technologies.

16MnCr5 steel was investigated in the present paper using pack carburizing at a temperature of 930 ºC and different holding times. Adjustments in the working steps and materials were performed for shorting the processing time and to allow reutilization of the part holder. Since the study of case deepness and hardenability are the most important parameters of any carburizing, those will be followed along the microstructure evaluation of each obtained sample.

2. Materials and Methods

2.1. Materials

In this study, samples of 1.7131 steel (16MnCr5) with chemical composition shown in (Table 1) were provided by Meusburger steel supplier with dimensions of 10x20x10 mm.

Table 1. Chemical composition of 1.7131 steel (wt.%)

C	Si	Mn	Cr	S	Fe
0.16	0.30	1.20	1.00	0.035	Bal.

Samples surfaces were cleaned using a water base solution of 20% NaOH before building the setup. After 1 hour immersing, samples were dried in oven at 70ºC.

Solid component used for pack thermochemical treatment is a combination between charcoal powder (80 wt.%) with 0.5 - 2 mm particles size and Na_2CO_3 (20 wt.%) as an energizer which facilitate the reduction of carbon dioxide to form the carbon monoxide gas needed in the setup holder.

For microstructure evaluation, embedded samples in cold-hardening acrylic resin were metallographic prepared using water-grinding paper and diamond grinding paste of 0.1 μm particle size at the final step. Furthermore, samples etching was done in a 10% nitric acid solution (HNO_3) for 5 s followed by 30 s holding in 10% hydrochloric acid (HCl) to reveal carburized surface layer. Microstructures analyses were performed using Nikon metallographic microscope (Nikon Eclipse MA100, Japan).

Hardness measurements were carried out using a Vickers microhardness tester (Future-Tech FM700, Japan) using 1000 gf load and 15 s dwell time as a testing condition. Even the Vickers method is used, the values was converted and displayed in HRC units. All samples were measured in cross section from the edge to 2.5 mm in depth, each point being an average value of three measurements. A scale of 0.5 mm was chosen as a measuring step until the maximum depth was reached.

2.2. Pack carburizing

To perform pack carburizing six samples of 16MnCr5 alloy were independently placed and sealed in the pack holder as indicated in Figure 1. Each sample (identified as "S" in Figure 1) was placed in the centre of an alumina crucible (3) surrounded by the solid high-carbon component (1), over which refractory cement sealer (2) was blocked. Also, in Figure 1 are indicated the dimensions of each layer component.

Pack carburizing parameters are indicated in Table 2. To compare the hardness evolution of the core and steel surface, three of the samples were subjected to water quenching and annealing after thermochemical treatment. Temperature choice is in accordance with the values range of 1.7131 steel from SR EN ISO 683-3:2018 standard [7].

Water quenching was done directly from the oven, each sample being quickly release in the cold-water container due to adapted sealer on holder inner diameter.

3. Results and Discussion

Cross section microstructures of each pack carburized samples can be found in Figures 2 and 3. Areas from the edge, core, and overview of 16MnCr5 microstructures are grouped together for a better evaluation. All the samples can be characterised by the presence of four zones with different morphology caused by carbon diffusion and quenching parameters from the carburizing temperature.

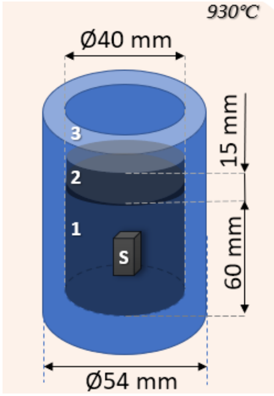
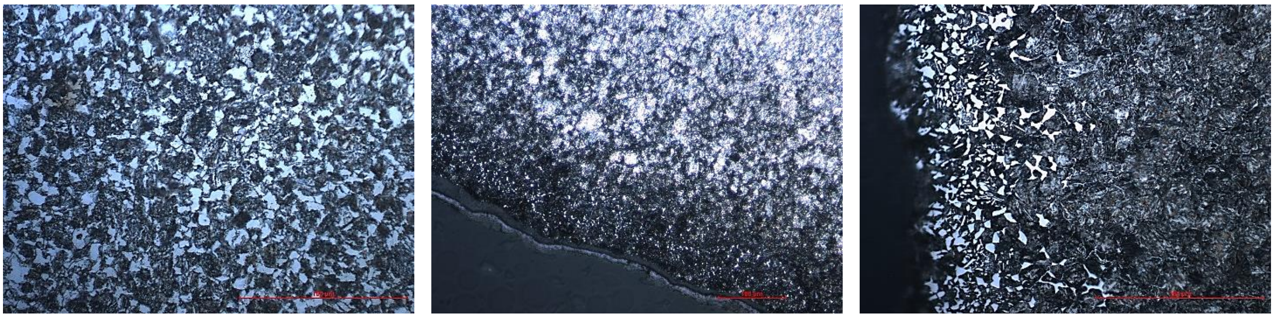


Fig. 1. Pack carburizing setup

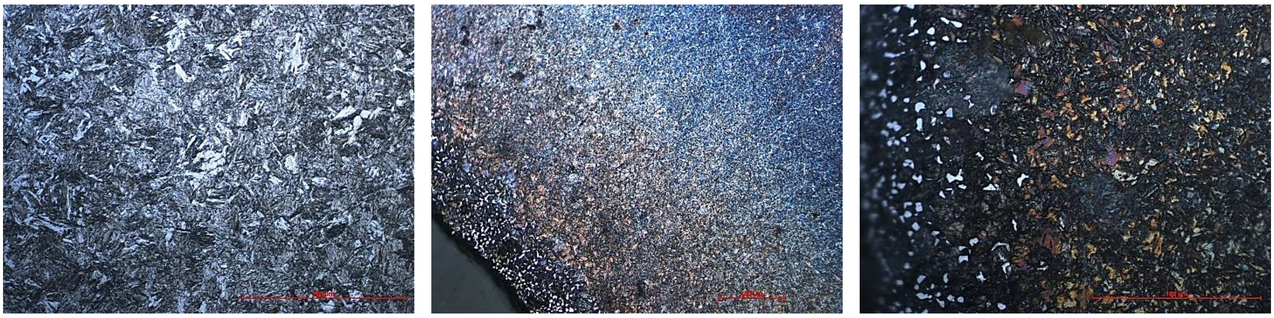
Table 2. Pack carburizing parameters

	Thermochemical treatment		**Thermal treatment**	
	Temperature [°C]	Holding time [min]	Quenching environment	Annealing [°C]
1	930	120	air	-
2		240		-
3		360		-
4		120	water	170 60 min
5		240		
6		360		



a) b) c)

Fig. 2. Microstructures of case-hardening 1.7131 steel after air quenching, for different holding time and magnification: a) 2h, core area (×500); b) 4h, edge area (×200); c) 6h, edge area (×500)



a) b) c)

Fig. 3. Microstructures of case-hardening 1.7131 steel after water quenching + annealing: a) 2h, core area (×500); b) 4h, edge area (×200); c) 6h, edge area (×500)

The core microstructure of the steel that was hold for 2h at 930 °C and air quenched it can be seen in Figure 2a, showing a ferrite-pearlite mixture, ferrite area being whiter, and the darker area consisted by pearlite constituent. For the same pack carburizing condition but with a much higher cooling rate (water quenching) and annealing for 1h at 170 °C, the core structure is a martensitic one as can be seen in Figure 3a. Martensitic structure can be a disadvantage for high toughness (tenacious) requested for core area and thermal treatment must be chosen carefully not to have the residual austenite in the microstructure or the microcracks occurrences [3, 4, 9]. These results are in good correlation with the measured hardness values, up to 1.5 mm distance from the edge.

Overview microstructures can be observed in Figure 2b and Figure 3b for 4h holding time at carburized temperature and then subjected at different quenching environment. According to carbon diffusion from the surface to the core, each zone can be approximatively appreciated and also correlated with hardness values. All four zones can be identified from the case to the core for all samples and contain the following: intermetallic compounds zone (as Fe, Cr carbides), hypereutectoid zone (>0.77 %C), eutectoid zone (0.77 %C) and hypoeutectoid zone (down to 0.16 %C). Core area is estimated at 0.16 %C as the chemical composition and is not being part of the previous classification.

Figures 2c and 3c reveal the case microstructures for pack carburized samples at 6h holding time. It can be clearly distinguished one edge zone with brighter particles that can be identified with carbides of Fe or Cr followed by a hypereutectoid zone with perlite and cementite (Figure 2c), respectively martensite (Figure 3c). It was taken into consideration also a decarburized of the surface, but according to references [1, 4] the appearance of the surface is a vast brighter area for 3h, 5h and 8h holding time of pack carburizing and not in clusters as we can see in (c) figures. Also, hardness values recorded for the case isn't indicate a decarburized phenomenon, Figure 4.

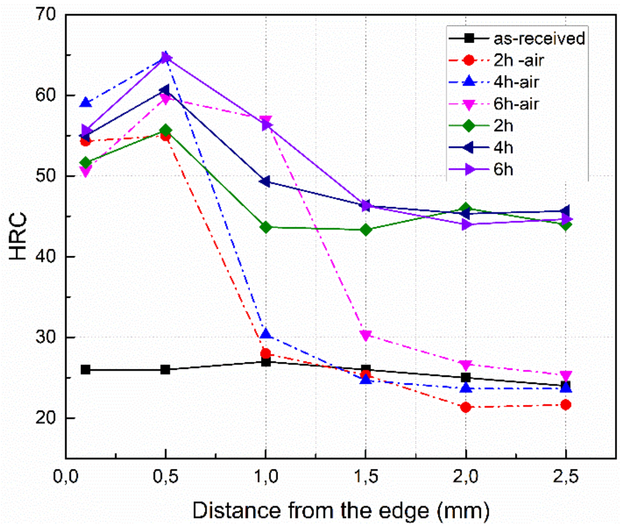


Fig. 4. Hardness values of all sample's conditions

To evaluate the carbon diffusion process first and second Fick's laws must be implied [2, 4, 8]. Calculated distribution of carbon at pack carburizing is indicated in Figure 5.

Case depth was calculated with the relation (1) from 0.1 mm to 1.2 mm, 1.6 mm, 2 mm, in other words until the value of 0.16 %C was reached.

$$C(x,t) = C_0 + (C_s - C_0) \cdot \left[1 - erf\frac{x}{2\sqrt{D \cdot t}}\right] \tag{1}$$

In (1) $C(x,t)$ is the carbon content at x [cm] case depth and t [s] holding time at carburized temperature; C_0 is initial C wt.% in 16MnCr5 steel; C_s is carbon saturated concentration in austenite at 930 °C (1.34wt% C according to Fe-C phase diagram); D is diffusion coefficient [cm^2/s].

To be able to calculate relation (1) diffusion coefficient must be also calculate with relation (2):

$$D = (0.07 + 0.06 \cdot \%\mathrm{C}) \cdot e^{\frac{-Q}{R \cdot T}} \tag{2}$$

Calculated value of diffusion coefficient was 1.76×10^{-7} cm^2/s, for this study data.

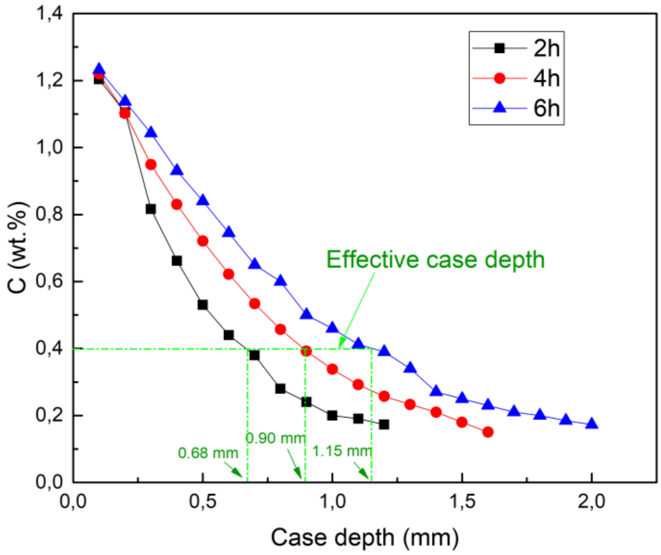


Fig. 5. Calculated carbon distribution at carburizing based on Fick's laws

Effective case depths were highlighted on Figure 5 as the carburizing is considering to be needed one for steel applications and is considered to be around 0.4 wt.% C [2, 4]. Therefore, after 2h of pack carburizing the effective case depth has a value of 0.68 mm, after 4h 0.9 mm, and after 6h 1.15 mm respectively.

In Figure 4 hardness of all samples is plotted depend on the distance from the carburized edge. As received steel (cold rolled condition) was measured for comparison, having almost constant value of 26 HRC. For all pack carburizing samples the carbon diffusion is no more available after 1.5 mm case depth because hardness values have reached the core values. Measured hardness value between 55-65 HRC was plotted for pack carburizing samples followed by water quenching and annealing. Due to water quenching the core hardness is increased also, to 45 HRC. These values can be in detriment of toughness needed for steel part core. Comparable values of 60 HRC were recorded in [1, 10] for the same 16MnCr5 steel or low-alloyed steel with low carbon content.

The most significant difference between hardness value of the case and core for the same sample is for 4h holding time for pack carburizing followed by air quenching. In this case the hardness case value is 65 HRC at 0.5 mm from the edge and decrease around 30 HRC at 1 mm from the edge.

4. Conclusions

The metallographic analysis confirmed the formation of a martensitic or ferrite-perlite microstructure in the carburized layer for the six tested conditions, observing the transition regions of carbon diffusion from the case to core. To the best of my knowledge, there are no pack carburizing studies on 1.7131 steel that indicate clusters formation on the edge specimens also with hardness value between 50-60 HRC. Furthermore, estimation of cluster formation of Fe or Cr carbides must be confirmed by an X-ray diffractometer and energy dispersion spectroscopy technique.

Water quenching directly from the pack carburizing was done due to adapted holder.

The highest hardness (65 HRC) value for pack carburized 1.7131 steel was obtained after 4- and 6-hours holding time at 930 °C at 0.5 mm case depth.

The combination of high surface hardness and high core toughness needed in the parts durability can be achieved with a ferrite-perlite microstructure in the specimen core, where no martensitic appear. This situation can be obtained after air quenching without other post-heat treatments, where hardness values of the core is similar with the initial sample (as-received).

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