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Surface analysis of SAE 3310 carburized steel in environment saturated with carbon nanofibers

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Abstract. The carburizing process is described by the reaction between environment atmosphere and metal surface followed by carbon transfer in a metal matrix. The main issue of heat treatment consist in ensuring the carburizing controlled atmosphere with a superior carbon potential level than metal surface of steel, in the carburizing furnace at a temperature that ensures process development. Obtaining carbon nanofibers relatively easily led to a new carburizing treatment, in the atmosphere full of carbon nanofibers. The use of nanomaterials for carburizing requires less time and best diffusion. The most common area of carbon concentration and residual austenite presence, the edges and corners are sensible reduced, the hardness is higher than the hardness obtained in regular carburizing treatment. Experiments made on samples of steel SAE 3310 have demonstrated the beneficial role of the presence of carbon nanofibers in carburizing atmosphere. It has developed the conclusion that the carburized layer properties depend on the amount of carbon nanofibers, during carburizing.

1. Introduction

The use of nanomaterials in carburizing heat treatment is a less researched option, presenting a high potential for innovation. The possibilities presented by carbon nanotubes to be captured and embedded in the steel matrix were investigated in heat treatment furnace. The morphology of single walled nanotubes is characterized by the properties of the chirality vector of graphene, which limited the configuration of nanotubes to armchair or zig-zag forms. The researches confirmed the thermal stability of SWCNT (single walled carbon nanotubes) in vacuum up to 2800⁰ C and in the air up to750°C [1].





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In the carburizing process, multi walled carbon nanotubes from commerce were used, with pronounced variability (length, diameter of fibers, number of walls and chirality), mixed in emulsion. Nanotubes are hollow fibers with 2 dimensions at nanoscale, with cylindrical nanostructure of carbon allotrope (figure 1). The interaction between metal matrix and CNT (carbon nanotubes) is possible to achieve by several methods: laser surface melting in presence of carbon nanotubes [2], electrophoretic deposition [3], powder metallurgy [4]. These methods are synthesized in figure 2.



Figure 2. Interaction between CNT (carbon nanotubes) and metal matrix and its results.

The results of the interaction are composites, coated metals and heat treated metals. The carburizing process consists in mass transfer on the surface of the metallic products of carbon atoms, which can be adsorbed by the solid. The process is the same as a solid carburization in graphite, with the difference that the graphite is replaced by an emulsion impregnated with MWCNT (multi walled carbon nanotubes).

2. Theoretical consideration

Mass transfer from the carburizing environment on the metallic surface occurs due to the difference between the average concentration of carbon in the environment and the metal surface [5, 6]. Diffusion occurs in metallic material due to its appearance in the superficial layer, a surplus of carbon through mechanisms of adsorption [7, 8]. Nanotubes from the metal surface will diffuse into the surface layer which results in the formation of a carburized layer. Some atoms of metal will be absorbed in a carbon nanotubes structure. The carburized layer formation mechanism is presented in figure 3.



Figure 3. Carburized layer mechanism a) at starting time b) absorption of carbon atoms (1-steel structure; 2-ferrite; 3-pearlite; 4-carbon nanotubes; 5-emulsion; 6-cementite; 7- martensite; 8-austenite; 9-carbon nanotubes).

3. Experimental work

For the production of samples, the study material will be steel used for bearings components, SAE 3310. The samples will be coated with a sodium silicate emulsion. The samples were heated at 980°C, the holding time is 2 hours. Carbon diffusion is made by cooling at 600°C. A different quantity of carbon nanotubes and graphite are mixed in an emulsion with sodium silicate (Na₂O₃Si) -20%, isopropyl alcohol (C₃H₈O) -30%, phenol formaldehyde resin -30% (multi walled carbon nanotubes powder acquired in commerce with the following parameters: purity > 97%, average outside diameter >55 nm, average inside diameter 8 nm, length 10-30 μ m, color black, density ~2.1 g/cm³). The carburizing process was made according to the diagram presented in figure 4.



Figure 4. Diagram of carburizing process [6].

In order to establish an optimum process, time and temperature, some restrictions are made. The restrictions consist in control carbides formation and retained austenite formation. These drawbacks can be avoided by an optimum carbon potential. The microscopic investigation showed the presence of retained austenite on the surface of carburized samples, the presence of martensite and carbides in the layer and the presence of sorbite in the core (figure 5).



Figure 5. Microstructures of carburized samples a) core structure, b) Carburized layer structure (0.5 mm), c) Carburized layer structure (0.2 mm), d) Carburized layer structure (at surface).

Figure 5 reveals the microstructure obtained after 2 hours of treatment, depending on the depth of carburizing: in core - fine sorbate with hardness 36 HRC (attack: nital 2%, magnification 100x); at 0.5 mm - hardening martensite plus retained austenite plus fine carbides with hardness 58 HRC (attack: nital 2%, magnification 400x); at 0.2 mm, - retained austenite plus fine carbides plus traces of hardening martensite with hardness 52 HRC (attack: nital 2%, magnification 800x); at surface, carburizing: 2 hours plus diffusion: 30 min - hardening martensite plus retained austenite plus fine carbides with hardness 61 HRC (attack: nital 2%, magnification 800x). The formation of a quantity of residual austenite is almost inevitable in microstructures of carburized layers containing high levels of carbon, which, in quantities of more than 50%, cause a significant decrease in the level of hardness and reduced fatigue. The main cause of excess residual austenite is the existence of higher carbon content in the surface layer. The most common areas of the surface carbon concentration are edges and corners of the piece. Another consequence of the high carbon content is revealed by the formation of massive carbides, which are formed at the limit of austenite grains and may have different morphologies in interdependence with the degree of alloying of the carburized steel. In table 1, table 2 and table 3 the hardness of the samples is shown after 2 hours of carburizing and 30 min for diffusion treatment, with different emulsions.

		Hardness [HRC]										
Samples / Type of	In controlled	Emulsion 10%	Emulsion 5% graphite	Emulsion	Emulsion							
carburization	atmosphere	graphite	+ 5% CNT	5% CNT	10%CNT							
1	60	57	59	60	61							
2	59	54	60	59	62							
3	59	56	61	59	61							
4	58	59	60	58	62							
5	60	58	60	59	60							
Average	59.2	56.8	60.0	59.0	61.2							
1-graphite 10%, 2- controlled atmosphere, 3- 5% CNT, 4- 5% CNT+5 % graphite, 5- 10% CNT												
Table 2. Value of hardness at 0.5 mm of samples.												
	Hardness [HRC]											
Samples / Type	In controlled	Emulsion 10%	Emulsion 5% graphite	Emulsion	Emulsion							
of carburization	atmosphere	graphite	+5% CNT	5% CNT	10% CNT							
1	49	44	49	49	51							
2	48	41	51	49	52							
3	48	46	51	48	51							
4	47	45	49	48	50							
5	49	47	49	47	50							
Average	48.2	48.2	51.8									
1-graphite 10%	6, 2- controlled a	atmosphere, 3- 5%	O CNT, 4- 5% CNT+5 %	graphite, 5-1	10% CNT							
Table 3. Value of hardness at 1 mm of samples.												
	Hardness [HRC]											
Samples / Type	In controlled	Emulsion 10%	Emulsion 5% graphite	Emulsion	Emulsion							
of carburization	atmosphere	graphite	+5% CNT	5% CNT	10% CNT							
1	39	36	39	40	41							
2	38	37	41	41	43							
3	37	35	41	40	41							
4	38	36	42	39	40							

Table 1. Value of hardness at surface of samples.

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39

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5

Average

39

38.2

4

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4. Conclusions

The experiments made it possible to plot the graphic in figure 6.



Figure 6. Influence of carburizing environment to the hardness and depth.

The curves of figure 6 represent the results of the experiments shown in table 1, table 2 and table 3: 1- pack carburizing with graphite 10%; 2 – carburizing in controlled atmosphere; 3 – pack carburizing with 5%CNT; 4 – pack carburizing with 5% graphite+5% CNT; 5 – pack carburizing with 10%CNT. Hardness increase with amount of carbon nanotubes. In the proposed treatment with carbon nanotubes a decrease of residual austenite due to a better diffusion of carbon in the layer is expected, which realizes redistribution of carbon atoms in the layer. The consumed energy will decrease because of low time carburizing and due to simplicity of carburizing installations towards carburizing in controlled gaseous environment.

5. References

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