U.P.B. Sci. Bull., Series B, Vol. 83, Iss. 1, 2021 ISSN 1454-2331

RESEARCH ON PARTS SURFACES CHEMICAL COMPOSITION CHANGES IN LIQUID AND SOLID STATE

Anişoara CORĂBIERU[1], ȘTEFAN VELICU[2], Petrică CORĂBIERU[3], Mirela Gabriela SOHACIU[4], Ioana Arina GHERGHESCU[5*]

The paper presents the research results on a new manufacturing process of metal parts, consisting in chemical composition changes of the parts surfaces in liquid and solid state followed by superficial hardening. This new process is used in Romania and abroad, with comparable results with the conventional technological processes. The novelty of this technology is highlighted by the following features: the alloying paste is used directly in the liquid phase; the hardening mixture is used in the solid phase prior to the induction heat treating; the possibility of performing some of the experimental technological flow stages simultaneously.

Keywords: durability, superficial liquid state alloying, hardening

1. Introduction

The development of superficially alloyed steels, especially alloyed tool steels, has underwent a qualitative leap in improving the performances of these materials. This may be done by several methods, such as:

- Production of new alloys;
- Optimizing the cutting tools geometry;
- Application of thermochemical treatments and non-conventional technologies (vacuum heat treatments, thermomechanical treatments, etc.).

In this regard, a new process of manufacturing high durability and low-wear metal parts is proposed. It consists in the production of a vacuum casting hypoeutectoid steel submitted to a superficial alloying in the liquid phase, followed by a surface deposition of a hardening mixture, an induction heat treatment and a quenching followed by tempering final heat treatment. The

[1] Technical University ”Gheorghe Asachi”, Iasi, Romania
[2] Department of Machines and Production Systems, University POLITEHNICA of Bucharest, Romania, e-mail: velstefan@hotmail.com
[3] S.C. PROCOMIMPEX, Iasi, Romania
[4] Department of Metallic Materials Processing and Eco - Metallurgy, University POLITEHNICA of Bucharest, Romania, e-mail: msohaciu@yahoo.com
[5*] Metallic Materials Science and Physical Metallurgy Department, University POLITEHNICA of Bucharest, Romania, e-mail: ioanaarinagherghescu@gmail.com

obtained results are comparable to the existing technologies both in the country and abroad [1 -3].

Due to the simultaneity of the technological flow stages, i.e. superficial alloying in the liquid phase during casting, followed by a hardening mixture deposition on parts surfaces and induction field processing, one may conclude that this process is viable, coherent and adaptable to an industrial technological flow.

2. Experimental part

The manufacturing of the high quality metal parts through the process described above comprises 12 technological phases, among which the most important are [4, 5]:

- Production of a C35 hypoeutectoid steel in vacuum (chemical composition: C = 0.32 – 0.39 %, Si, Cr, Ni = max. 0,4%, Mn = 0.5 – 0.8%, Mo = max. 0,1%, S = max. 0.045 %) and superficial alloying through the interaction of the melt with the layers deposited on the mold walls. The superficial alloying elements were less than 0.5% of the whole metallic volume, the elemental composition of this alloying paste denoted PM is shown in Figure 1 [6, 7].

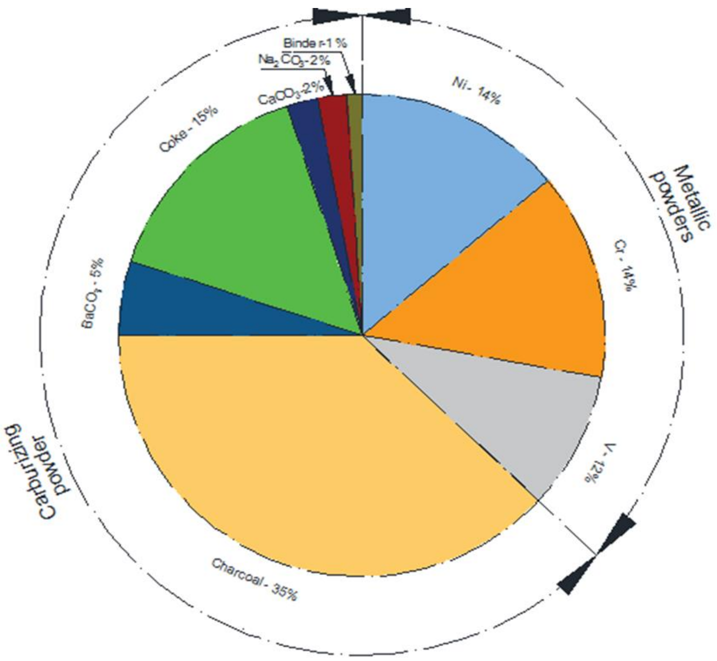

Fig.1. Chemical composition of the PM paste

Table 1.

Chemical composition of the PM paste

Paste symbol	PM paste chemical composition, %								
	Metallic powders, 40%			Carburizing powder, 60%					
	Ni %	Cr %	V %	Charcoal %	$BaCO_3$ %	Coke %	$CaCO_3$ %	Na_2CO_3 %	Binder %
PM	14	14	12	35	5	15	2	2	1

- Deposition on the parts surfaces of the hardening mixture denoted AD, whose chemical composition is shown in Figure 2 [8]:

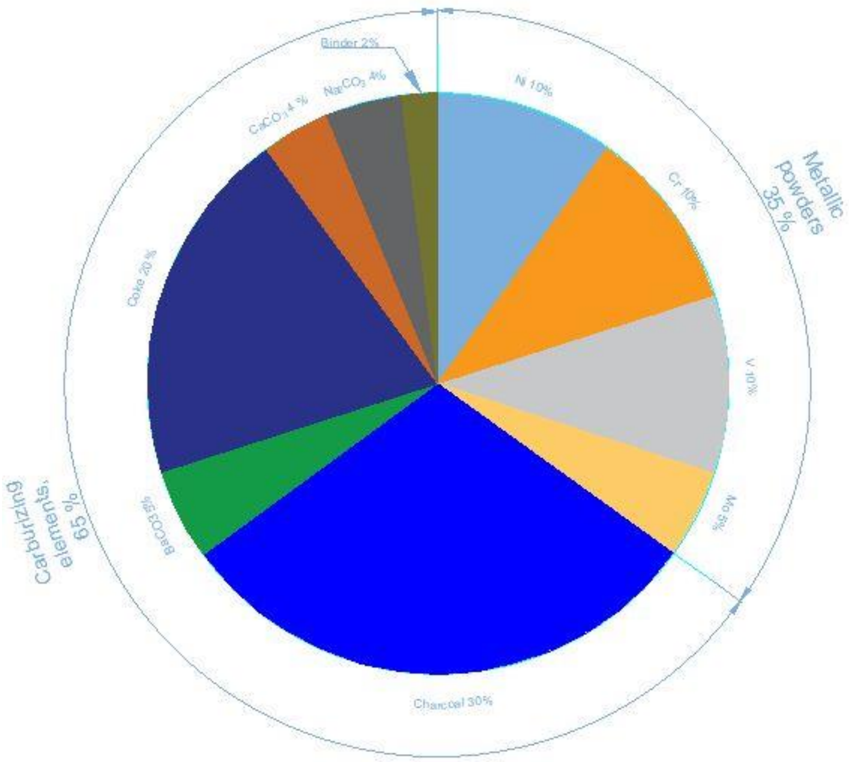

Fig.2 Chemical composition of the hardening mixture AD

Table 2

Chemical content of the hardening mixture AD

Hardening mixture symbol	Hardening mixture composition, %									
	Metallic powders, 35%				Carburizing elements, 65%					
	Ni %	Cr %	V %	Mo %	Charcoal %	$BaCO_3$ %	Coke %	$CaCO_3$ %	Na_2CO_3 %	Binder %
AD	10	10	10	5	30	5	20	4	4	2

- Induction heat treatment, with the following parameters: T=1000 - 1050°C; $t_{heating}$ up to the induction heating temperature = 2 ÷ 5 s; holding time at this temperature = 2 ÷ 5 min; inductor specific power: P_{sp} = 1 kW/cm^2; current intensity, I = 700 A; electric tension, U = 20 ÷ 30 V [9, 10].

- Final heat treatment: rapid cooling in air after induction heat treatment + subcritical intermediate annealing (650°C/60min/air) + quenching of the outer layer (830°C/30min/water) + low temperature tempering (180°C/60min/air) [9, 11].

The manufacturing process follows the experimental technological flow shown in Figure 3. One may observe the possibility of performing some working steps simultaneously:

- superficial liquid alloy during casting: phases 1, 2, 3 and 4;
- hardening of the solid phase by induction field processing: phases 7, 8 and 9;
- the possibility of the technological flow stages simultaneity that influences directly the labor productivity and the industrial competitiveness of the technological process [8].

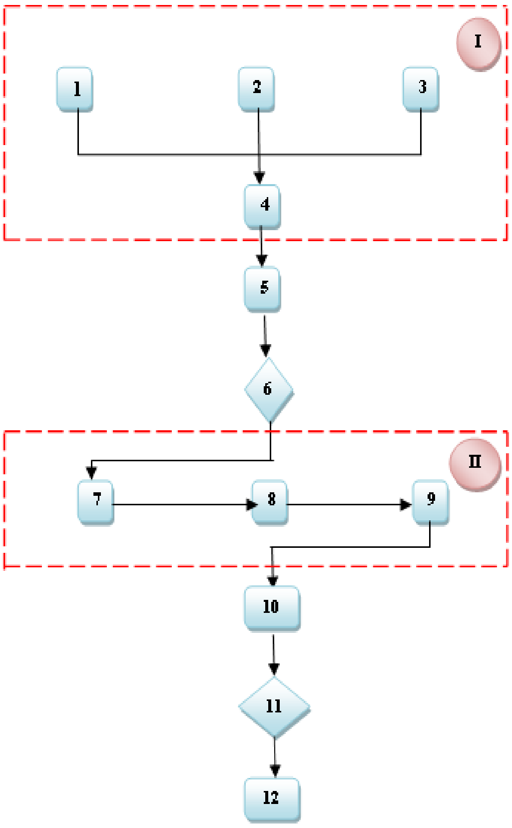

Fig. 3. The experimental technological flow of the manufacturing process of the mandrel metal parts.
Flow stages: 1 – preparation of the alloying paste; 2 – preparation of the casting mold; 3 – production of steel in vaccum conditions (hypoeutectoid steel); 4 – casting and alloying in the liquid phase; 5 – preliminary machining; 7 – preparation of hardening mixture; 8 – depositing the hardening mixture onto the metal part surface; 9 – induction heat treatment; 10 – final heat treatment; 12 – delivery of the finished product.
Control stages: 6 – control of the superficial layer; 11 – final control of the layer.
Simultaneity of the flow stages: I – simultaneity of the liquid phase alloying stages; II - simultaneity of solid state hardening stages.

The parts manufactured by this experimental procedure were of the mandrel type (Fig. 4). Samples were taken for optical microscopy analysis on an Leika optical microscope and scanning electron microscopy investigations on a QUANTA INSPECT electron microscope.

Fig. 4. A part (mandrel) manufactured by the hardening process in liquid and solid phase

3. Results and Discussion

Optical microscopy structural investigations

The optical micrographs are shown in Figure 5 (microstructure highlighting a transition from a martensitic zone to a carbide – free ferritic and pearlitic one).

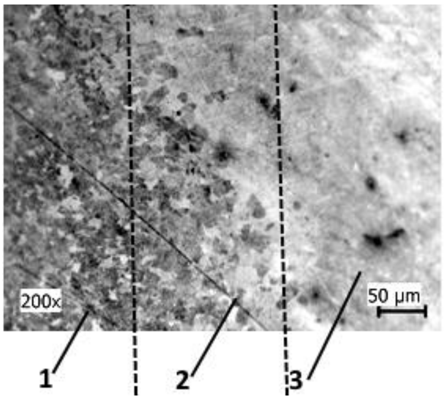

Fig. 5. Optical micrograph of the transition from a mainly martensitic zone to a carbide – free ferritic and pearlitic one. 1 – martensitic structure; carbides and scarce retained austenite areas are also present. 2 – transition zone; 3 - ferritic and pearlitic structure. Reagent: Nital 2%. Magnification 200x.

Optical microscopy analysis reveals the carbides distribution (small, bright particles) and shows an average diffusion of the alloying elements (components of the alloying paste and hardening mixture) in the superficial layer. The layer structure is mainly martensitic with complex carbides. The structure of the intermediate zone exhibit a transition from a martensitic structure to a ferrite + pearlite structure. The core structure consists in ferrite + pearlite.

The structure of the sample is in accordance with the results of the distribution of the alloying elements which are components of the alloying paste used in the liquid phase and of the hardening mixture used in the solid phase.

Structural Investigations by Scanning Electron Microscopy (SEM)

Results of scanning electron microscopy investigations performed on a QUANTA INSPECT F (FEI -PHILIPS) microscope are given in Figures 6 and 7.

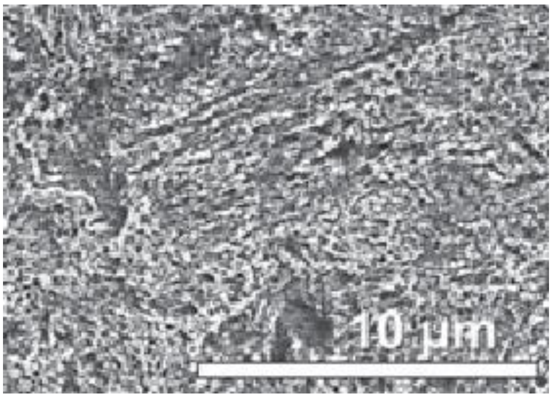

Fig. 6. SEM microstructure consisting of martensite, complex carbides and retained austenite.

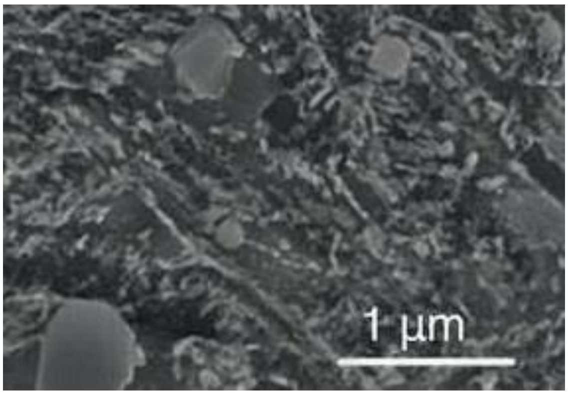

Fig. 7. Another SEM microstructure exhibiting martensite, complex carbides and retained austenite.

The variation of the alloying elements concentration along the outer layer depth is shown in Figure 9.

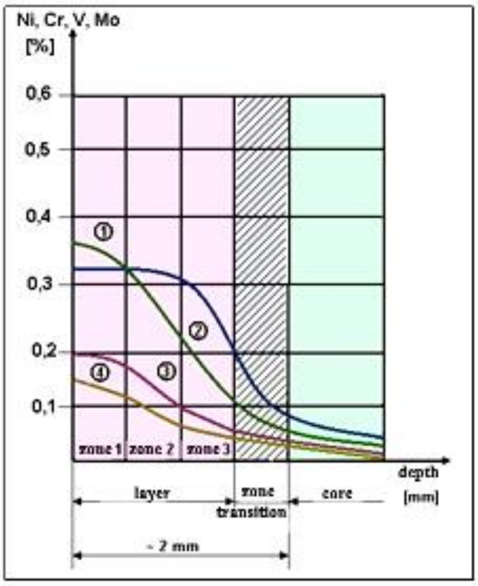

Fig. 9. Distribution of microalloying elements in the superficial layer: 1 – Cr variation; 2 – Ni variation; 3 – V variation; 4 – Mo variation.

The quantification of the superficial layer chemical composition of the mandrel part was carried out as follows: strips were taken from the processed parts having an adequate hardness of the surface layer. The surface layer was mechanically machined by cutting in steps with a depth of 0.5 mm. On each step surface the chemical composition was determined by spectral analysis.

According to this graph one may assume the following:

- A medium diffusion intensity of Cr in the outer layer (rapidly decreasing distribution in microzones 2 and 3);
- A medium diffusion intensity of Ni in the outer layer (constant distribution in microzones 1 and 2);
- A lower diffusion intensity of V in the outer layer as against Cr and Ni (constant distribution in microzone 1, medium decreasing distribution in microzones 2 and 3);
- A weak diffusion of Mo in the outer layer.

The distribution of the alloying elements in the layer, the transition zone and the core are in correlation with the results obtained by optical microscopy performed on the same areas.

The final heat treatment used in the experiments for the realization of the mandrel part consisted in :

- Rapid cooling after induction heat treatment;
- Subcritical intermediate annealing (T = 650ºC, t = 60 min.);
- Quenching (T = 830ºC; t = 30 min.);
- Low temperature tempering (T= 180ºC; t = 60 min.).

The chemical composition of the PM alloying paste provides an appropriate superficial alloying which is achieved by the interaction between the melt and the layers deposited on the mold walls.

The mandrel parts have a mainly martensitic structure with complex carbides, the carbon diffusion in the layer being highlighted by the presence of the alloying elements carbides.

Optical microscopy investigations reveal an medium diffusion intensity of the PM and AD alloying elements in the superficial layer of the mandrels.

Their microstructure consists in the following zones:

- Outer layer: martensite with dispersed carbides of the alloying elements
- Transition zone: intermediate structure between a martensitic zone and a carbide – free ferritic and pearlitic one
- Core: ferrite and pearlite

The distribution of the alloying elements in the superficial layer reflects the medium diffusion intensity of the alloying elements in the technological phase 4 - casting and alloying in the liquid phase, as well as along the technological phase 9 - induction heating processing.

4. Conclusions

The research on the manufacture of metal parts by the hardening in liquid and solid state phase procedure led to the conclusions presented below:

The distribution of the superficial alloying elements and the chemical composition of the mandrel superficial layer confirms an moderate-to-high diffusion of Ni and Cr and a moderate diffusion of V. During the induction heat treatment, the diffusion is carried out with an intensity that depends on the technological parameters (heat treatment temperature, temperature reaching time and holding time).

The microstructural analysis of the outer layer reveals a homogeneous, martensitic structure with the superficial alloying elements found as dispersed carbides. The carbon level exceeds the concentration of 0.50 % in the layer, which confirms that the percentage of 60 % carburation powders in the total composition of the alloy paste covers the needs for achieving the stated objective.

The final heat treatment may be applied on charges containing a large number of parts. It is the optimal variant in terms of economic efficiency and quality level.

The results obtained by optical microscopy for the layer, the transition zone and the core are in accordance with the results of the alloying element distribution. The process is adaptable to mandrel parts because the surface layer benefits of a moderate diffusion of Ni with a constant distribution, a moderate diffusion of Cr with a rapidly decreasing distribution and a moderate diffusion of V with a relatively constant distribution.

For the stressing complex to which a mandrel is submitted in intermittent operating regimes (contact pressure, shock, compression, vibration), the chemical elements diffusion and the carbide content of the superficial layer provide adequate wear resistance.

Therefore we are entitled to conclude that the application of this process at industrial scale will result in the manufacture of improved quality metal parts (tough core, an uniform and homogeneous martensitic structure of the superficial layer, presence of the superficial alloying elements in the outer layer) thanks to the superficial alloying in liquid and solid phase.

The experimental process proposed by the authors is adaptable for the manufacture of several mandrel types (fixing bolt, support mandrel, locking mandrel, forging mandrel, main tool holder).

The manufacturing process requires adaptations to be implemented on an already existing industrial technology flow. Research and investigations will continue in order to improve the adaptation to requirements of an industrial technological flow with the emphasis on studying the influence on the competitiveness of the company.

R E F E R E N C E S

1. *D. Dragomir, M. O. Cojocaru, N. Dumitru,* "The Effect of Change of Carburizing Media Nature on Growth Kinetics of Layer", UPB Sci Bull, Series B, vol. 75, no.1, 2013, pp. 181 – 192.
2. *M. Cojocaru, S. Iorga*, "Processing conditions influence on the carbochromized layers on sintered iron matrices", UPB Sci Bull, Series B, vol. 74, no.3, 2012, pp. 115 – 124.
3. *M. Cojocaru, L. Druga, D. Dragomir*, "The Influence of Sub-Critical Thermochemical Processing on Fatigue Resistance of 42CrMo4 Steel", UPB Sci Bull, Series B, vol. 77, no. 2, 2015, pp. 169 – 180.
4. *D. D. Vasilescu, P. Corabieru, A. Corabieru, M. Boicu, D. Mihaileanu, M. J. Agop*, „On a Constitutive Material Law at Nanoscale", COMPUT. THEOR. NANOS., vol. 13, no. 1, 2016, pp. 265 - 269.
5. *P. Corabieru, St. Velicu, D. Zait, A. Corabieru, D. D. Vasilescu*, „Tendences and perspectives regarding the recycling of automotive waste", ENVIRON. ENG. MANAG. J., vol. 8, no. 5, 2009, pp. 1151-1154.
6. *A. Corabieru, D. D. Vasilescu, P. Corabieru, V. Vasilescu,* Procedeu şi amestec de durificare superficială a unei piese din oţel, Brevet de invenţie nr. 129648/30.03.2016 (Superficial hardening technique and mixture of a steel part, Patent)
7. *P. Corabieru, D. D. Vasilescu, A. Corabieru,* Procedeu de depunere a unui metal pe o piesă din oţel, prin centrifugare şi pastă de aderenţă utilizată de aceasta, Brevet de invenţie nr. 129840/30.08.2017 (Deposition procedure of a metal on a steel part by centrifugation and adhesive paste used, Patent).
8. *A. Corabieru, St. Velicu, P. Corabieru, M. G. Sohaciu*, „Research on metal parts hardening by superficial alloying", REV. CHIM. (BUCHAREST), vol. 70, no. 2, 2019, pp. 470-474.
9. *A. Corabieru, St. Velicu, P. Corabieru, D. D. Vasilescu, I. Paunescu*, „Research on the structure of metal parts manufactured by the innovative procedure of combined processing operations", Proceedings in Manufacturing Systems, vol. 12, no. 3, 2017, pp. 101-106.
10. *G. Enciu, I. Paunescu,* "Research on Improving the Reliability of Copper Alloys Parts by Polyester Coating", MATER. PLAST., vol. 52, no. 4, 2015, pp. 564-567.
11. *R. Paunescu, I. A. Gherghescu*, „Structural Relevant Aspects through Scanning Electron Microscopy (SEM) of Some Thermal Barrier Ceramic Coatings Deposited on Copper Support", METAL. INT., vol. XV, no. 7, 2010, pp. 12 -18.