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RESEARCH ON THE OPTIMIZATION OF THE MANUFACTURING TECHNOLOGY OF THE METAL PARTS HEAT TREATED IN LIQUID AND SOLID PHASE

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Abstract: *This paper analyzes the quality of pinion type parts manufactured through the technological procedure of hardening in liquid and solid state phase. The investigations are made by electronic microscopy and optical microscopy, highlighting the structure and composition of the superficial layer and the micro-hardness determinations. Depending on the results obtained, the paper suggests measures for optimization, aiming especially at the values of the most important technological parameters for the technological phase of microalloying in liquid state and induction heat treatment in solid state phase. The hardening of the superficial layer of the pinion type parts is revealed by the high values of the micro-hardness determinations. The hardening and structure of the superficial layer are supporting the implementation of the technological procedure at industrial level.*

Key words: *hardening, micro-hardness, liquid state phase, solid state phase.*

1. INTRODUCTION

The technological procedure of hardening in liquid and solid state phase includes a number of 12 technological phases. The pinion type parts made by heat treatment in liquid and solid state phase have the following features [1, 6]:

- high level of micro-hardness values;
- high durability during utilization;
- high values of the mechanical features;
- the investigations conducted by electronic microscopy and optical microscopy highlighted the following issues: martensitic structure with micro-alloying elements finely dispersed within the superficial layer, presence of complex carbides and absence of the agglomerations of elements such as Si and Mn;
- the structural non-homogeneities are isolated and do not influence the quality of the superficial layer [4, 6].

The distribution of the microalloying elements present in the superficial layer is uniform on the entire surface. The optimization of the values of the main technological parameters contributes to the improvement of the final product quality, namely the pinion type part. [3, 7] The quality of the finished product will enforce and support the implementation of the technological procedure at industrial level [2, 5, 8].

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2. EXPERIMENTAL TECHNOLOGICAL FLOW

The experimental technological flow is formed of 12 technological phases; the most important are the technological phase no 4 (casting and microalloying in liquid state phase) and the technological phase no 9 (induction heat treatment) [9, 11].

Technological phase 1 – preparation of microalloying paste PM:

- preparation of microalloying paste PM: metal dust 40% + carburizing powder 60%;
- chemical composition of microalloying paste PM: Ni 14%, Cr 14%, V 12%, charcoal 35%, BNaCO3 5%, coke 15%, CaCO3 2% Na2Co32%, binder 1%;
- particle average size: 40 μm;
- particle shape: granular [6, 10].

Technological phase 2 – preparation of casting mold:

- chemical and mechanical cleaning: chemical solvent-based cleaning solution + mechanical brushing;
- ventilated air drying: air flow from 0.5 to 1 m^3/min;
- applying of microalloying paste PM: Applying type: manual paint brushing, Number of layers: 3 [6].

Technological phase 3: *Elaboration of basic material*

- furnace type: induction furnace; capacity: max.100 kg; Chemical composition of the basic material: C < 0.3%; S < 0.02%; Si = max.1%; Mn = max.0.45; alloying elements under 0.5%;
- $T_{elaboration}$ = 1600ºC; $t_{elaboration}$ = 2-3 hours; $t_{evacuation}$ = max. 1 min.

Technological phase 4 – casting and microalloying from liquid phase:

- $T_{casting}$= 1550–1600°C; $V_{casting}$ = 0.2–2 kg/s; $t_{casting}$ = 2–60 s.

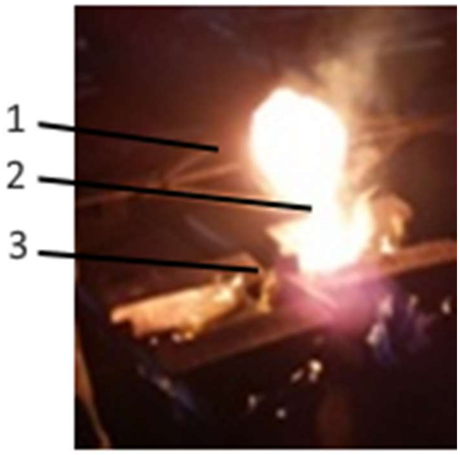


Fig. 1. Casting and microalloying in liquid state phase: 1 – casting dipper; 2 – basic material in liquid state; 3 – casting mould.

- Microalloying is performed by interaction of liquid steel with layers applied on the casting mold walls;
- Successive layers are applied as microalloying paste PM.

Casting total period < 20 min for avoiding metal bath oxidation [6].

Technological phase 5 – preliminary machining operations – finishing turning of superficial layer:

- cutting depth: ap = 0.05–0.40 mm;
- feed rate: f = 0.05–0.10 mm/rev.;
- average chemical composition at surface of superficial layer after preliminary machining: C 0.50%; Ni 0.40%; Cr 0.40%; V 0.05%.

Technological phase 6 – superficial layer control:

- macroscopic control; metallographic structure control; control of superficial layer chemical composition: highlighting of energy dispersive X-ray spectrum (EDAX);
- scanning electron microscopy SEM;
- optical microscopy.

Technological phase 7 – preparation of hardening mixture AD:

- preparation of hardening mixture AD: metal dusts 35% + carburizing elements 65%;
- chemical composition of hardening mixture AD: Ni 10%, Cr 10%, V 10%, Mo 5%, charcoal 30%, BNaCO3 5%, coke 20%, CaCO3 4% Na2Co3 4%, binder 2%; particle shape: granular.

Technological phase 8 – application of hardening mixture on the surface of the part:

- cleaning of surface: chemical solvent based cleaning solution + mechanical brushing;
- applying of AD mixture: Number of layers: 3;
- ventilated air drying: air flow 0.5–1 m^3/min;
- average thickness of coating mixture: 0.1 mm.

Technological phase 9 – induction heat treatment:

- T = 1000–1050°C; $T_{heating}$ = 2–5 s; hold time: 2–5 min; part diameter: max. 35 mm;
- optimal frequency, $f_{optimal}$ = 10 kHz;
- specific power at surface of the part: Psp = 1 kW/cm^2; current intensity: 700 A; voltage: 20–30 V; depth of heated layer: 2–4 mm. (Fig.2) [6].

Fig. 2. Induction heat treatment of pinion type parts: 1 – helix inductor; 2 – pinion type part; 3 – hardening mixture AD applied to the active surface of the pinion type part.

Technological phase 10 – final heat treatment:

- variant A – direct hardening CD + low tempering RJ (applicable to parts of minor importance which must have particularly high values of hardness);
- variant B – increased cooling + simple hardening layer CS + low tempering RJ (to be applied for parts with deformations, dropping the direct hardening);
- variant C – increased cooling + sub-critical intermediate annealing RcI + simple hardening layer CS+ low tempering RJ (applicable to parts and components that require machining after treatment).

Technological phase 11 – control of layer:

- Control of hardness: average hardness of superficial layer: 55 HRC;
- Control of structure: transition zone structure: from a predominantly martensitic structure to a predominantly ferrite+ carbides-free pearlite structure;
- Layer structure: finished, predominantly martensitic, presence of alloying elements as dispersed carbides (variant C of final heat treatment) (Fig. 3).

Technological phase 12 – delivery of finished part:

- checking the competitiveness elements: the estimated production costs fall within quality-cost matrix required by the market;
- verification of compliance for: green product →green supplier [5, 8].

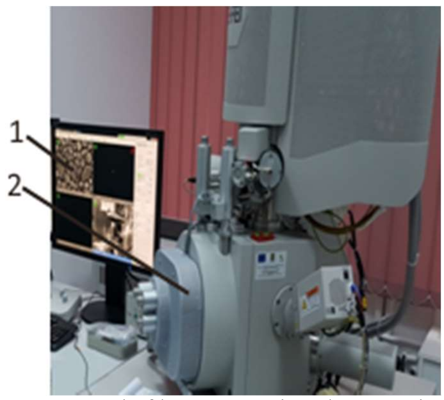


Fig. 3. Control of layer– scanning electron microscopy SEM; 1-view of scanning electron microscopy image; 2 – quanta Inspect electron microscope.

3. EXPERIMENTS. CHECKING THE VALUES OF THE MAIN TECHNOLOGICAL PARAMETERS

The experiments consisted of pinion type parts manufacturing by heat treatment in liquid and solid state (Fig. 4).

The values of the main technological parameters obtained during experiments are listed below:

PM: metal powders 40% + carburizing powders 60%;

- flow rate – $d_a = 1$ m^3/min;
- temperature of pre-heating: $T_{pre\text{-}heating} = 100$ºC;

Basic material: C = 0.26%; S = 0.01%; Si = 0.80%; Mn = 0.45%; alloying elements = 0.5%;

- temperature of elaboration: $T_{elaboration} = 1600$ºC;
- time of elaboration: $t_{elaboration} = 2.5$ hours;
- time of evacuation: $t_{evacuation} = 1$ min;
- temperature of casting: $T_{casting}$= 1570°C;
- speed of casting: $V_{casting} = 1.2$ kg/s;
- time of casting: $t_{casting} = 50$ s;
- duration of casting $D_{casting} = 20$ min;

AD: metal powders 35% + carburizing elements 65%;

- time of processing: $T_{processing}$ =1050°C;
- time of heating: $t_{heating} = 5$ s;
- time of holding: $t_{holding} = 4$ min;
- frequency of current: $f_{optimal} = 10$ kHz;
- power density: $Psp = 1$ kW/cm^2;
- useful power: $P_u = 20$ kW;
- current intensity: I =700 A;
- voltage: $U = 20$ V.

Variant C consist of intensified cooling + subcritical intermediate re-annealing RcI + layer simple hardening CS+ low tempering RJ.

The analysis performed by electron microscopy is shown in Fig. 5.

Variant C was chosen for final heat treatment because it is suitable for the parts submitted to wear and tear in normal operation conditions (Fig. 6).

The analysis of the superficial layer micro-composition made by means of the energy dispersive X-ray spectroscopy (EDAX) revealed the presence of the microalloying elements of PM microallying paste and of AD hardening mixture in the layer (Fig. 7).

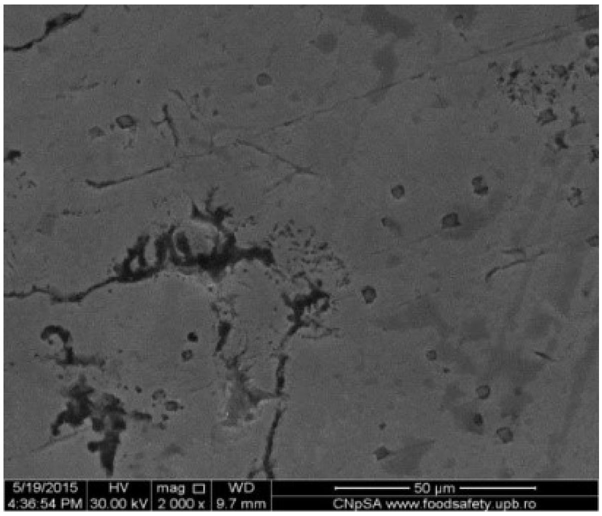
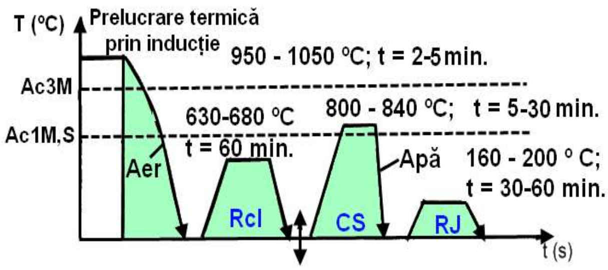


Fig. 5. Image of scanning electron microscopy (SEM), magnification × 2000.
Condition: not attacked; Micro-zone of interest: layer Structure: finished, predominantly martensitic, presence of alloying elements in form of dispersed carbides (after final heat treatment, variant C).



Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	R	A	F
Si K	1.13	2.22	29.59	8.49	0.0037	1.1462	0.9153	0.2842	1.0098
V K	0.45	0.49	32.26	7.63	0.006	0.9808	0.9824	0.959	1.4114
Cr K	0.54	0.58	21.69	9.69	0.0083	0.9981	0.9888	0.9768	1.5699
Fe K	97.31	96.18	1.61	947.12	0.9914	0.9982	1.001	0.9958	1.025
Ni K	0.57	0.53	61.8	3.66	0.0046	1.0148	1.0123	0.7735	1.0372

Fig. 6. Final treatment: variant C.

Fig. 4. Part type: pinion.

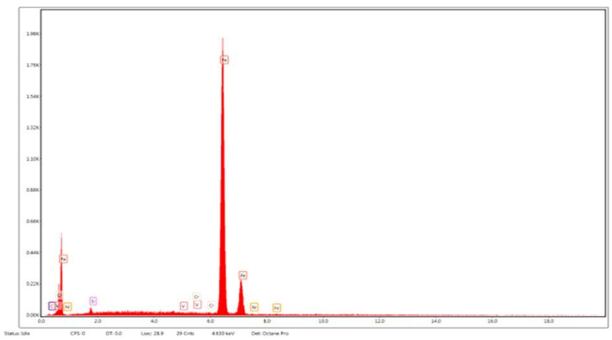


Fig. 7. Micro-composition: Energy dispersive X-ray spectroscopy (EDAX). Condition: not attacked Micro-zone of interest: layer. Presence of Ni, Cr and V in the superficial layer under the form of complex carbides (proper diffusion of Ni, Cr and V, poor diffusion of Mo).

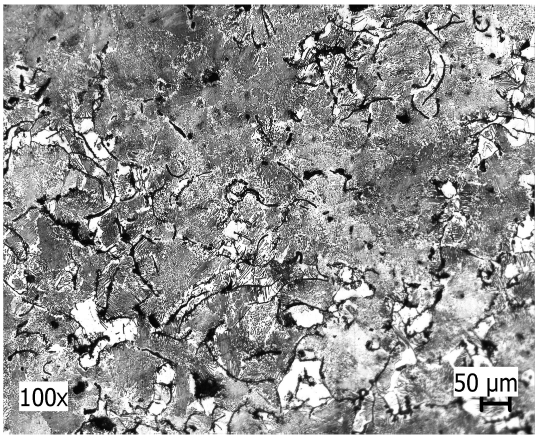


Fig. 8. Image of optical microscopy (Leika –Germany), magnification × 100
Condition: nital attack 2% ; Micro-zone of interest: layer.
Structure: finished, predominantly martensite + ferrite, presence of alloying elements in form of carbides (after the final heat treatment, variant C).

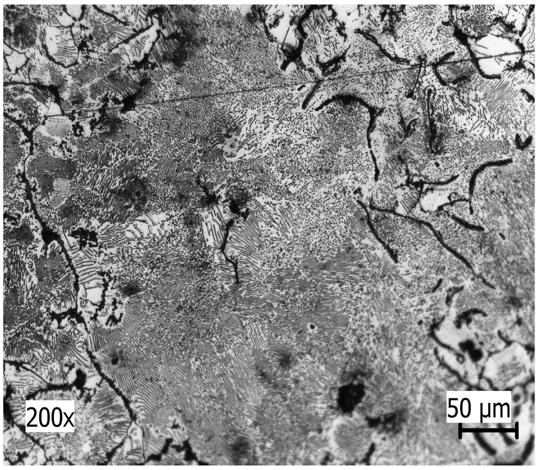


Fig.9. Image of optical microscopy (Leika –Germany), magnification x 200
Condition: nital attack 2%; Micro-zone of interest: layer
Structure: finished, predominantly martensite + ferrite, presence of alloying elements in form of carbides (after the final heat treatment, variant C).

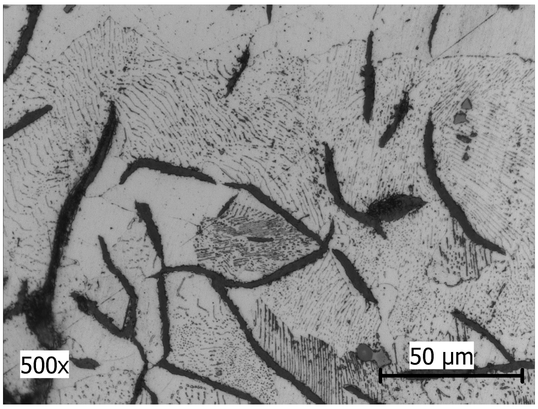


Fig. 10. Image of optical microscopy (Leika –Germany), magnification × 500
Condition: nital attack 2%; Micro-zone of interest: transition area;
Structure: transition zone from a predominant martensitic structure to a structure, mainly ferrite + perlite.

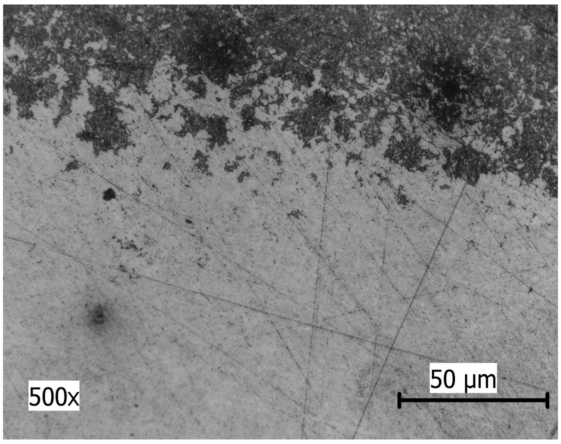


Fig. 11. Image of optical microscopy (Leika –Germany), magnification × 500
Condition: nital attack 2%; Micro-zone of interest: transition area
Structure: transition zone from a predominant martensitic structure to a structure, mainly ferrite + perlite.

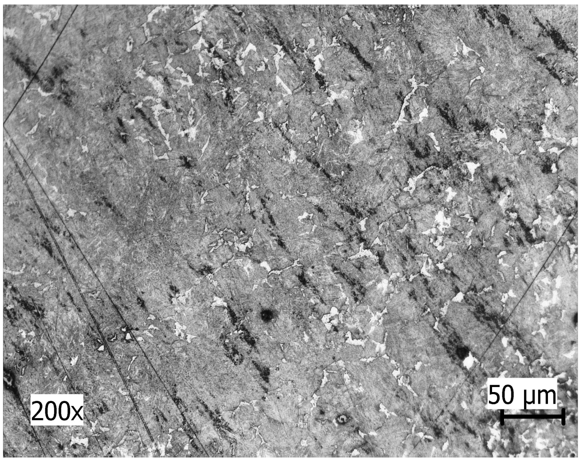


Fig. 12. Image of optical microscopy (Leika –Germany), magnification x 200
Condition: nital attack 2%; Micro-zone of interest: core
Structure: finite media, ferro-perlite, uncomplicated Si and Mn rare islands.

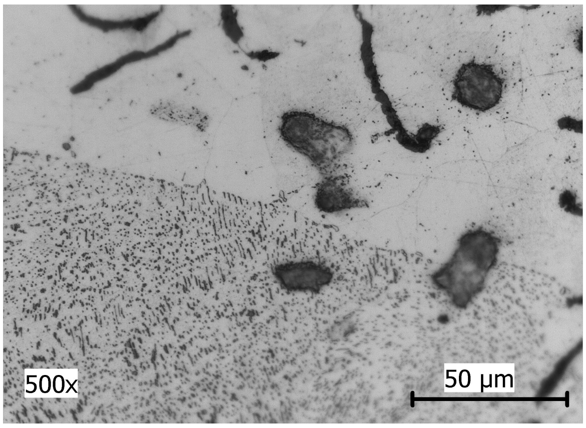


Fig. 13. Image of optical microscopy (Leika –Germany), magnification × 500
Condition: nital attack 2%; Micro-zone of interest: core
Structure: finite media, ferro-perlite, uncomplicated Si and Mn rare islands.

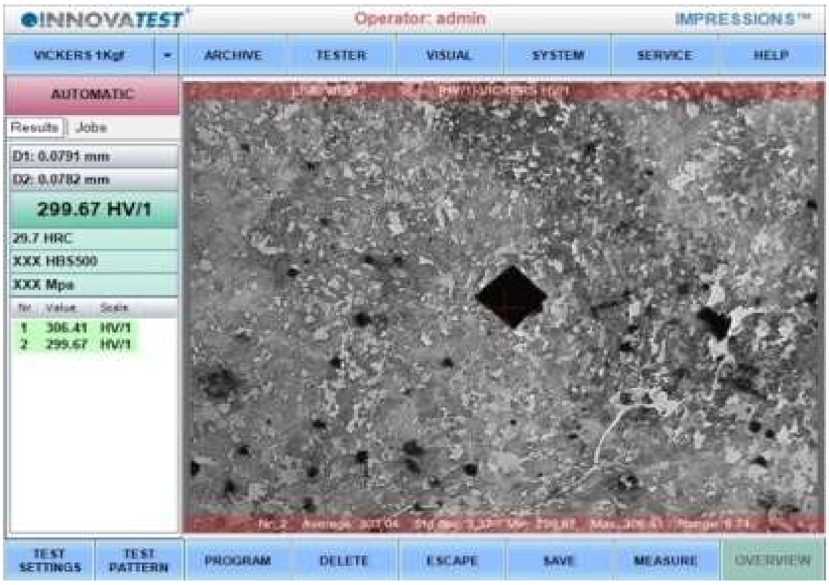


Fig. 14. Determinations of micro-hardness made on the samples taken from pinion type parts.

The analysis by optical microscopy highlights the predominantly martensitic finished structure of the superficial layer, according to Figs.8 and 9. The analysis of the transition area is shown in Figs. 10 and 11. The optical microscopy analysis of the core of the piece is shown in Figs 12 and 13.

The determinations of micro-hardness confirm the hardening of the superficial layer, with high average values of the micro-hardness (Fig.14).

The values of the micro-hardness determinations in the superficial layer 740 HV (62 HRC) are consistent with the results of the alloying elements distribution in the superficial layer (proper diffusion of Ni, Cr and V simultaneously with a low diffusion of Mo).

4. MEASURES TO OPTIMIZE THE PROCEDURE

The results of the investigations conducted during the experiments highlight the following aspects:

- the values of the superficial layer micro-hardness determinations are high and prove the proper distribution of the microalloying elements in the superficial layer;
- proper diffusion of Ni, Cr and V in the superficial layer under the form of complex carbides;
- low diffusion of Mo in the superficial layer.

In order to improve the quality and evenness of the superficial layer, in conformity with the results obtained through electronic microscopy, optical microscopy and micro-hardness determinations, the technological phase no. 4 requires the increase of the casting temperature $T_{casting}$ from 1570°C to 1590°C and the increase of the casting speed $V_{casting}$ from 1.2 to 1.6 Kg/s.

Thus the optimum values of the main technological parameters for the pinion type part were obtained with the following composition of the basic material C = 0.26%; S = 0.01%; Si = 0.80%; Mn = 0.45%; alloying elements = 0.5%:

- $T_{elaboration}$ = 1600 ºC;
- $t_{elaboration}$ = 2.5 ore;
- $t_{evacuation}$ = 1 min;
- $T_{casting}$= 1590 °C;
- $V_{casting}$ = 1.6 Kg/s;
- $t_{casting}$ = 50 s;
- $D_{casting}$= 20 min;
- $T_{processing}$= 1050 °C;
- $t_{heating}$= 5 s;
- $t_{holding}$= 4 min;
- $f_{optimal}$ = 10 kHz;
- Psp = 1 kW/cm^2;
- P_u = 20 kW;
- I =700 A;
- U = 20 V;
- final heat treatment: variant C.

5. CONCLUSIONS

The research conducted during the experiments and the results obtained on this occasion lead to the following conclusions:

1. The carbon diffused in the layer properly, exceeding the concentration of 0.5% at the surface. The distribution of the nickel and chrome is uniform while the vanadium is distributed in a non-uniform way, with more reduced intensity. The molybdenum has a minor diffusion from the microalloying paste PM in the superficial layer.

2. The composition of the superficial layer reveals a proper diffusion of the Ni, Cr and partially V, which attests a medium intensity of the microalloying reactions directly from liquid state phase.

3. The diffusion of the elements of the hardening mixture AD into the layer during the induction heat treatment took place at a relatively high intensity.

4. The agglomerations of Si and Mn in the form of strips and grids diminish the durability. The non-uniform dispersion of the carbon and micro alloying elements as well as the level of inclusions decrease the durability. The main causes are related to the inadequate values of the temperatures and durations of heat treatment.

5. The results of the structural analysis of the superficial layer indicate a structure mostly homogenous, dominantly martensitic, with the presence of microalloying elements in the form of dispersed carbides. The agglomerations of Si and Mn in form of strips or grids are very rare. The complete elimination of the agglomerations involves the improvement of the control of the technological parameters values, of the compositional dosing and of the PM pastes and AD hardening mixtures drying.

6. The final heat treatment has a direct influence on the finishing of the superficial layer. Variant C is recommended for the pinion type part. A final treatment that does not meet the specific requirements of the work piece leads to the decrease of the features, entailing the diminution of the results achieved during the previous technological phases.

7. The results of the research prove the viability of the technological procedure of heat treatment in liquid and solid state phase and they support the implementation of the technological procedure at industrial stage.

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