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PRELIMINARY EXPERIMENTS ON PROCESSING TECHNOLOGY COMBINED LIQUID AND SOLID PHASE

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Abstract: *The combined processing technology used to manufacture the parts of the surface layer hardened steel. The novelty technology lies in the fact that the hardening of the surface layers is carried out both in liquid phase and in the solid state. Technology comprises three main stages with 12 technological phases encompassing 12 technological operations and 17 operations control, analysis and compliance.*
Stage I: Micro-alloying directly from the liquid surface, by interaction with the liquid steel layer deposited on the walls of the mold. Successive layers are deposited on the walls of the mold in the form of paste made from 40% PM micro-alloyed metal powders (nickel, chromium, vanadium) and 60% carburizing powder (charcoal, barium carbonate, coke, calcium carbonate, sodium carbonate, binder).
Stage II: Thermal treatment by induction of parts coated with hardening mixture AD: 35% metal powder (nickel, chromium, vanadium, molybdenum) and 65% carburizing powder (charcoal, barium carbonate, coke, calcium carbonate, sodium carbonate, binder).
Stage III: Final heat treatment by: the chemical composition of the base material; surface layers composition; application and operating conditions of the parts.
The paper analyzes the results obtained in stage I (micro-alloying directly from liquid surface). The results obtained in the stage I influence on the quality of the final product.

Key words: *combined processing, micro-alloying, surface hardening, induction treatment.*

1. INTRODUCTION

The combined processing technology achieves the face hardening of steel parts in both states: liquid and solid [4,5].
The experimental process flow consists of:
- 12 process stages;
- 12 process operations;
- 17 control, analysis and conformity operations.

During the preliminary experiments there were aimed the development and the identification of solutions of manufacturing technology through combined processing in liquid and solid state, according to Table 1.

2. EXPERIMENTAL TECHNOLOGICAL FLOW

Technological flows used in the preliminary experiments include the route from raw material to finished parts delivery (Fig. 1).

Table 1

Identification of solutions for combined processing technology

Phase techn.	Solutions related to:
1	Chemical content of micro-alloyage paste PM
2	Chemical and mechanical cleaning; deposit of micro-alloyage paste PM; ventilated air drying; preheating.
3	Chemical content of basic material, temperature and development time.
4	Casting process; casting temperature, speed and time.
5	Process parameters of preliminary mechanical processing.
6	Chemical content of outer micro area of samples after micro-alloyage directly from liquid state; chemical content at a depth of 0.20 mm in relation to the active surface
7	Chemical content of the hardening mix AD.
8	Deposit shape; number of successive layers;
9	Heating temperature; diameter of the part; depth of the heated layer; penetration depth of the electromagnetic field; quenching depth; frequency, specific power on the surface of the part; heating duration.
10	Process parameters (temperature, maintenance time, cooling method) of the final thermal treatment versions A, B, C, D.
11	Layer features, hardness, granulation, inclusions, specific adhesion, metallographic structure, defects.
12	Competition elements, green product, environment profile of the product, implementation possibilities of the overall quality principle on the process flow, recycling throughout the entire lifetime of the product.

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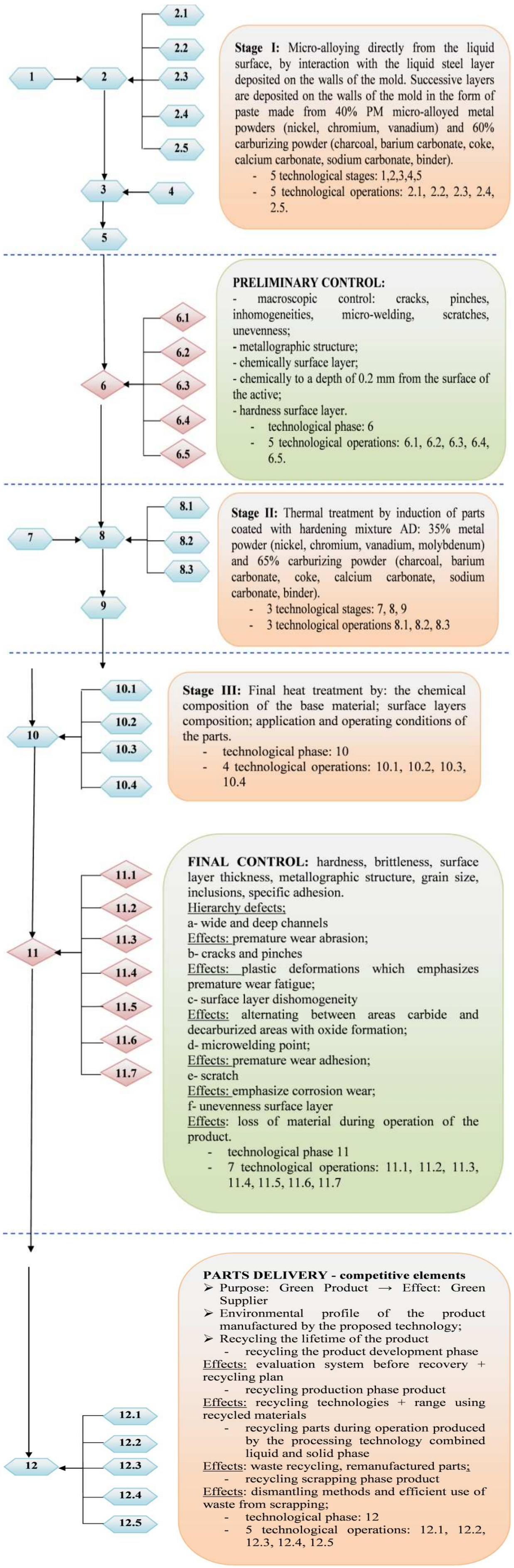

Fig. 1. Experimental technological flows.

3. PROCESS STAGES. PARAMETERS

The technology is aimed at hardening the surface. Surface hardening starts in the liquid phase. Hardening solid phase is completed [9].

Process stage 1: Preparation of the micro-alloyage paste PM: metallic powder 40% + carburizing powder 60% (Table 1).

Process stage 2: Preparation of the casting mould [2].
*Technological operation 2.1.*Chemical and mechanical cleaning: cleaning solution based on chemical solvents + mechanical brushing
*Technological operation 2.2.*Ventilated air drying: airflow 0.5 – 1 m^3/min
*Technological operation 2.3.*Deposit of the micro-alloyage paste PM

- type of deposit: manual brush;
- number of layers: 3.

*Technological operation 2.4.*Ventilated air drying: airflow 0.5 – 1 m^3/min
*Technological operation 2.5.*Preheating: T = 100ºC, $T_{maintenance}$ =60 min.

Process stage 3: Development of the basic material

- type of furnace: electric induction furnace;
- capacity: max.100 Kg;
- chemical content of the basic material.

C < 0.3%, S < 0.02%, Si = max.1%, Mn = max. 0.45%.

Process stage 4: Casting and micro-alloyage in liquid state [3, 10].
$T_{casting}$= 1550–1600°C; $V_{casting}$ = 0.2 – 2Kg/s; $t_{casting}$ = 2-60 s.

The micro-alloyage takes place by interaction between liquid steel and the layers on the walls of the casting mould. The deposited successive layers are under micro-alloyage paste PM (Table 2).

Process stage 5: Preliminary mechanical processing: finishing turning of the face layer.

- cutting depth: a_p = 0.05–0.40 mm;
- advance rate: f = 0.05 – 0.10 mm/rot.

Process stage 6: Face layer control
Technological operation 6.1. Macroscopic control;
Technological operation 6.2. Metallographic structure control;
Technological operation 6.3. Face layer chemical content control;
Technological operation 6.4. Chemical content control at the depth of 0.20 mm in relation to the active surface
Technological operation 6.5. Face layer hardness control

Process stage 7: Preparation of the hardening mix (Table 3).

Table 2

Chemical content of the micro-alloyage paste PM

Symbol paste micro alloyed	Composition paste microalloyed, %								
	Metal powders 40%			Carburizing powder 60%					
	Ni %	Cr %	V %	Charcoal %	$BaCO_3$ %	Cocs %	$CaCO_3$ %	Na_2CO_3 %	Binder %
PM	14	14	12	35	5	15	2	2	1

Table 3

Chemical content of the hardening mix AD

Symbol mixture hardening	The composition of hardening, %									
	Metal powders 35%				Elements of carburizing 65%					
	Ni %	Cr %	V %	Mo %	Charcoal %	$BaCO_3$ %	Cocs %	$CaCO_3$ %	Na_2CO_3 %	Binder %
AD	10	10	10	5	30	5	20	4	4	2

Process stage 8: Deposit of the hardening mix on the surface of the part.

*Technological operation 8.1.*Surface cleaning: cleaning solution based on chemical solvents + mechanical brushing

Technological operation 8.2 AD deposit

- type of deposit: manual brush
- number of layers: 3

Technological operation 8.3. Ventilated air drying: airflow 0.5 – 1 m^3/min.

Process stage 9: Induction thermal processing

- T =1 000–1050°C;
- $t_{heating}$ = 2–5 s;
- maintenance duration = 2–5 min;
- diameter of the part: max. 35 mm;
- optimal frequency, $f_{optimal}$ = 10 kHz;
- specific power on the part surface: Psp = 1 kW/cm^2;
- generator effective capacity: 20 kW;
- current intensity: 700 A;
- tension: 20 – 30V.

Process stage 10: Thermal treatment [1, 7]

Technological operation 10.1. Version A - direct quenching CD + low recovery RJ (it applies to low importance parts that need to present especially high values of hardening) ;

Technological operation 10.2. Version B - stressed cooling + simple layer quenching CS + low recovery RJ (it applies to deformed parts, putting aside direct quenching. The parts are accelerated under ventilated air and reheated at 800–840 °C, in order to ensure simple layer quenching);

Technological operation 10.3. Version C – accelerated cooling + subcritical inter annealing RcI + simple layer quenching CS + low recovery RJ (it applies in the case of parts that require post-treatment machine cutting. Between carburizing and simple layer quenching a subcritical inter annealing is introduced at 630–680°C);

Technological operation 10.4. Version D – double quenching with inter annealing (it applies to parts of medium alloyed steels superficially hardened).

Process stage 11: Layer control

Technological operation 11.1. Hardness control.

- Average of the face layer hardness: 55 HRC;

Technological operation 11.2. Shortness control.

- Absence of cracks near the trace obtained from pushing the head of the diamond penetrator with a strength of 100 daN;

Technological operation 11.3. Face layer thickness control.

- ε_u – useful depth of the layer whose carbon concentration is higher than 0.40%; the values of ε_u depend on the values of the carbon concentration determined in depth, in layers in accordance with the variation graphics of the carbon concentration on the layer depth, ε_u = 2.00 mm;
- ε_d – de depth up to which the minimum hardness is 600HV (~ 54 HRC), ε_d = min.0.50 mm; the ε_d values depend on the averages of the hardness determinations on the layer depth, step-up and in accordance with the variation graphics of the hardness on the depth of the carburized and micro-alloyed layer;
- ε_a – distance from the active surface to the point where the temperature at the end of heating has T_a = 800ºC; ε_a is determined through simulations, obtaining for T = 950 ºC→ ε_a = 2.111 mm, and for T = 1050 ºC → ε_a = 2.614 mm;
- ε – quenching depth determined by the empirical relation ε = (0.05–0.10)d (mm); for the diameter of the part of 35 mm it is considered ε = 0.40–3.00 mm.

Technological operation 11.4. Metallographic structure control

- Mainly martensitic structure + complex carbides without carbon separations ;

Technological operation 11.5. Granulation control

- Smooth granulation in the face layer - small grain index on the surface of the part: 9, 10. 11

Technological operation: 11.6. Control inclusions

- Reduced number of inclusions in the superficial layer: p=max.2;

A – sulphate = 0 ÷1; B – oxides linear = 1 (L_{max} = 5,5 μm) ÷ 2 (L_{max} = 11 μm, No. inclusion = 4, total length = 44 μm), C – silicates = 0–1; D – globular oxides = 1–2 (D = 11 μm, standard image area = 95 μm^2)

Technological operation: 11.7. Control specific adhesion $q = F/A$; F = pressing force, N; A = surface area, mm^2.

- soft metallurgical bonding: q < 80 N/mm^2;
- average metallurgical bonding: q = 80 – 170 N/mm^2;
- strong metallurgical bonding: q > 170 N/mm^2.

Process stage 12: Delivery of the finished part

Technological operation 12.1. Verification of the competitiveness elements: [6]

- the estimated production costs fall under the cost-quality matrix required on the EU market;
- similar features with parts manufactured in the EU;
- post-sale services: 12 month warranty;
- consolidation of the economic agent by creating new jobs;

Technological operation 12.2. Verification of the compliance requirements: Green product → Green supplier.

Technological operation 12.3. Development of the environment profile of the product manufactured by combined processing technology in liquid and solid state (Table 4).

Technological operation 12.4. Implementation of the total quality principle (Table 5)

Technological operation 12. 5. Recycling throughout the entire life cycle of the product (Table 6).

Table 4

Development of the environment profile of the product [6]

Technological operation 12.3.1	Identification of the environment impact generated by parts manufactured through combined processing in liquid and solid state.
Technological operation 12.3.2	Determine for each identified impact the critical stage of life cycles
Technological operation 12.3.3	Identify improvement margins.
Technological operation 12.3.4	Assess advantages and disadvantages of the improvement methods;
Technological operation 12.3.5	Replace quality steel with normal steel for parts by using combined processing in liquid and solid state and the design changes that ensure a resistance comparable to the initial materials.

Table 5

Implementation of the total quality principle

Technological operation 12.4.1	Regular staff certification.
Technological operation 12.4.2	Certification of analysis labs.
Technological operation 12.4.3	Technical level accepted for installations of combined processing in liquid and solid state.
Technological operation 12.4.4	High standards for: depollution, storage, collecting, recovery, reuse and recycling.

Table 6

Recycling throughout the entire life cycle of the product [8]

Technological operation 12.5.1	Recycling in the product development stage: Achieved effect: evaluation system before recovery + recycling plan.
Technological operation 12.5.2	Recycling in the product production stage: Achieved effect: recycling technologies + use of the recycled material range.
Technological operation 12.5.3	Recycling in the exploitation stage of parts manufactured through suggested technology: Achieved effect: waste recycling + refurbished parts.
Technological operation 12.5.4	Recycling in the product cassation: Achieved effect: dismantling methods + efficient use of waste resulted from cassation.

4. PRELIMINARY EXPERIMENTS

In preliminary experiments to check the stage I of technological concept; emphasis has been placed on micro-alloying directly from the liquid phase.

Preliminary experiments consist of:

- preparation of the micro-alloyage paste PM (40% metallic powder + 60% carburizing powder);
- micro-alloyage directly in liquid state.

The micro-alloyage was achieved directly from liquid state by interaction between liquid steel and the layers deposited on the walls of the casting mould.

Table 7 presents the values of the main technological parameters used in the preliminary experiments.

Table 7

Preliminary experiments - micro-alloyage directly in liquid state

No. sample	No. charge	Paste type	Type deposit	No. paste layer	Preheating temperat. [°C]	Hold time [min]	Sample mass [Kg]	Type casting sample	Casting temperature [°C]	Casting speed [kg/s]	Time casting [s]
P1	S1	PM	brush	3	100	40	0,20	direct	1560	0,10	2
P2	S1	PM	brush	3	100	60	0,30	direct	1580	0,10	3
P3	S1	PM	brush	3	100	50	0,40	direct	1560	0,20	2
P4	S2	PM	brush	3	100	50	0,60	direct	1600	0,20	3
P5	S2	PM	brush	3	100	60	0,60	direct	1560	0,20	3
P6	S2	PM	brush	3	100	40	0,20	direct	1580	0,10	2
P7	S3	PM	brush	3	100	40	0,40	direct	1560	0,10	4
P8	S3	PM	brush	3	100	50	0,60	direct	1600	0,20	4
P9	S3	PM	brush	3	100	60	0,40	direct	1580	0,10	4
P10	S3	PM	brush	3	100	60	0,60	direct	1560	0,20	3

The investigation methods and the main techniques used:

- Establish material hardness, with hardness micrometer MIC 20 Krautkramer;
- Microstructural and microcompositional analysis through scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX) on the scanning electron microscopy Quanta Inspect F.
- Phase analysis through X-ray diffraction on the Panalytical Diffractometer X'Pert Pro MPD

Methods of investigation determined that technological conditions micro-alloying occurred with maximum intensity.

5. RESULTS

The micro-alloyage directly from liquid state occurred with intensity in the case of sample P2, experimental batch S1.

Technological parameters for experimental sample P2, batch S1 are shown in Table 7. In Figs. 2 and 3 are shown test areas of interest micro P2.

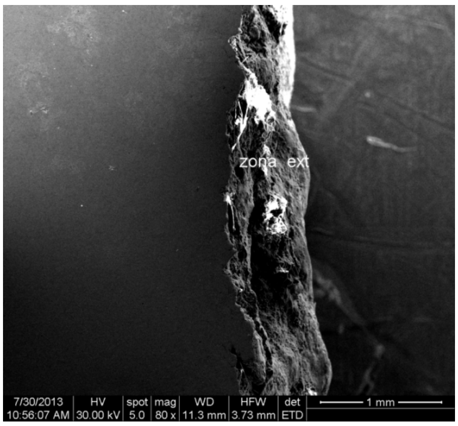

Fig. 2. Image of scanning electron microscopy (SEM) at a 80X zoom.

Sample P2

Interest microarea: outdoor

Depth of the outer area: 0.10 - 0.50 mm in relation to the active area of the sample. ETD determination: secondary electron detector.

Microarea closest to the outer active surface. A micro-alloying produced with maximum intensity in this area confirms the viability of the process.

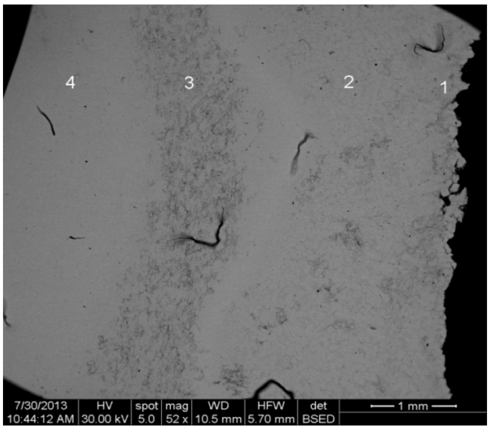

Fig. 3. Scanning electronic microscopy image (SEM) to a size of 52X with highlighting analysed interest microareas.
Sample P2
Outer microarea (0.10-0.50 mm from the active area)
Microarea 1 (0.75 mm from the active area) Microarea 2 (1.50 mm from the active area) Microarea 3 (2.50 mm from the active area) Microarea 4 (3.50 mm from the active area)
BSED determination: back-scattered electron detector.

In Figs. 4 and 5 distribution of element homogeneity is shown.

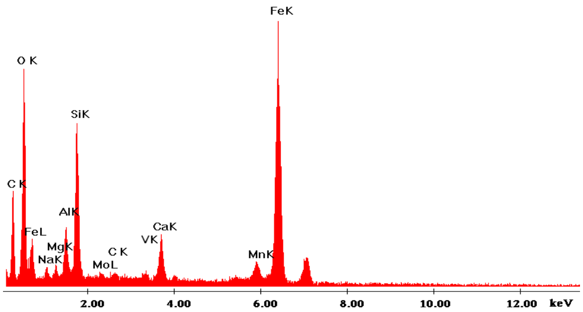

Fig. 4. Energy dispersive X-ray analysis (EDAX) obtained from outer microarea - sample P2.
Distribution of element homogeneity in the outer microarea - Sample P2 Composition: C: 0.52%; Ni:0.37%; Cr:0.38%; Mo:0.09%

The micro-alloyage took place intensely in the case of sample P2, experimental batch S1, through enrichment of the outer area in the micro-alloyage and carburising elements: C-0.52%; Ni-0.37%; Cr-0.38%; V- 0.11%; Mo-0.09% in relation to the active area.

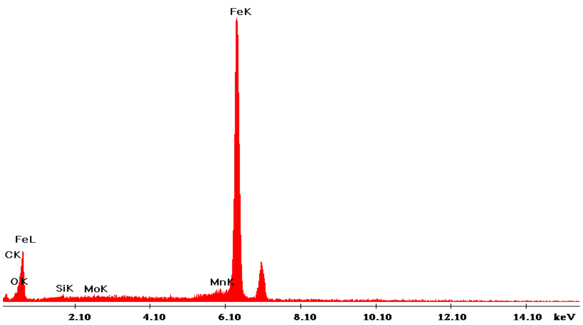

Fig. 5. Energy dispersive X-ray analysis (EDAX) obtained on the microarea 3 - sample P2.
Distribution of element homogeneity in the microarea 3 (2.50 mm from the active area) – Sample P2
Composition: C: 0.25%; Ni:0.05%; Cr:0.05%

Compositional analysis of face layers:
Experimental batch: 1
Sample: P2
Compositional analysis of the outer interest area (0.10 – 0.50 mm from the active area): C: 0.52%, Ni:0.37%, Cr:0.38%, V: 0.11%, Mo:0.09%
Compositional analysis of the interest area 1 (0.75 mm from the active area): C:0.37%, Ni:0.12%, Cr:0.11%, V: 0.05%
Compositional analysis of the interest area 2 (la 1.50 mm from the active area): C:0.32%, Ni:0.09%, Cr:0.07%
Compositional analysis of the interest area 3 (la 3.5 mm from the active area): C:0.25%, Ni:0.05%, Cr:0.05%.

Analysis on the structure and quality of face layers:

- raw structures casting the samples are analyzed by electron microscopy;
- micro-alloying directly from liquid superficial layers determines the presence of alloying elements;
- alloying elements are embedded in the phase nano-structured components.

In Figs. 6, 7, 8 and 9 Scanning electron microscopy images for sample P2 (different orders of magnification) are presented.

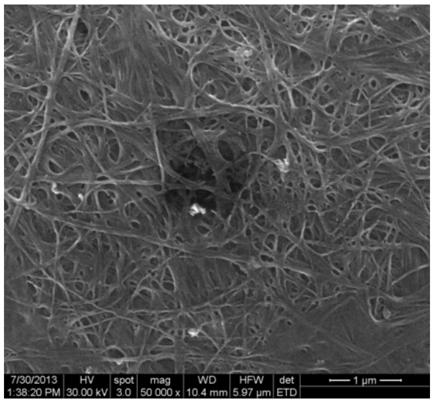

Fig. 6. Image of scanning electron microscopy (SEM).
Sample P2
Interest area: outdoor
Zoom: 50.000x
Unfinished raw casting structure
Presence of alloyage elements in the outer layer

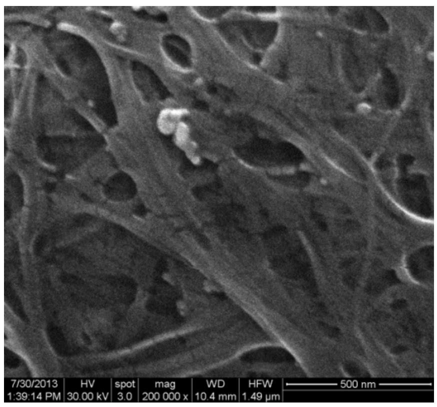

Fig. 7. Image of scanning electron microscopy (SEM).
Sample P2
Interest area: outdoor
Zoom: 200.000x
Raw casting structure unprocessed thermally
Presence of nano-structured phase parts in the basic materials

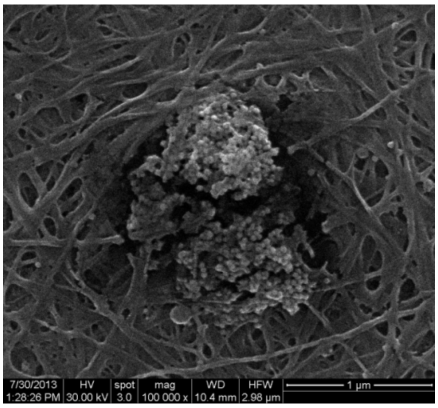

Fig. 8. Image of scanning electron microscopy (SEM).
Sample P2
Interest area: outdoor
Zoom: 100.000x
Unfinished raw casting structure
Alloyage phase parts nano-structured in the basic material

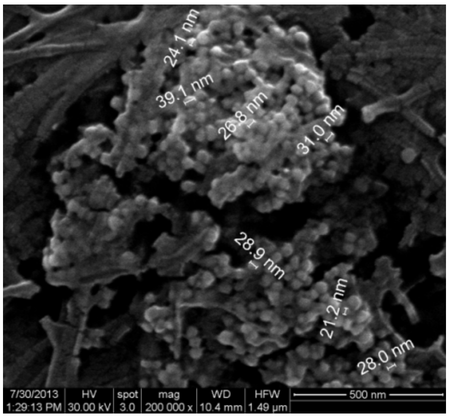

Fig. 9. Image of scanning electron microscopy (SEM).
Sample P2
Interest area: outdoor
Zoom: 200.000x
Unfinished raw casting structure
View of alloyage phase parts in the basic material
Dimensional size of nano-structured parts

The measurements for determining the hardness were done on 2 areas of each sample: the basic material and the face area, by using the hardness micro-meter MIC20 Krautkramer (Table 8).

The structural analysis confirms the present of alloyage elements in the face layers. The view of nanostructured alloy phasic components reveals that micro-alloying directly from the liquid phase occurred with intensity.

Table 8

Hardness determinations

No. sample	**Area of interest**	**Measured values [HV10]**	**Average**
P1	Basic material	470; 450; 500	473
	Superficial layer	703; 710; 720	711
P2	Basic material	358; 356; 360	358
	Superficial layer	710; 709; 712	710

6. CONCLUSIONS

The structural analysis highlights the presence of elements from the micro-alloyage paste in the outer area of the samples by creating a face layer as a highly-adhesive outer cover.

The hardness average of the face layer is high considering that the casted samples were subject to simple thermal processing, which highlights the direct micro-alloyage in the liquid phase. The high values of the hardness average and the presence of alloyage elements in the face layer support the durability of the process of combined processing in liquid and solid state.

Preliminary experiments will continue with stage II of the technological process. Technological parameters used for setting values sample P2 to base the optimization stage I. A micro-alloying high obtained in stage I create the basis to obtain adequate results in stage II (induction thermal processing).

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