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NITRIDING PROCESS MODERNIZATION ON THE EXAMPLE OF CHOSEN PARTS FROM AIRCRAFT ENGINE

MODERNIZACJA PROCESU AZOTOWANIA NA PRZYKŁADZIE WYBRANYCH CZEŚCI LOTNICZYCH

World industry increasingly takes advantage in nitriding process to produce technological surface layer (TSL) of machine and device components working in conditions of high contact loads. In industry practice not only the traditional gas nitriding has found application, but also whole range of its modern modifications which lets to shape the TSL structure in arbitrary way.

Application of ion nitriding has let (by the control of conditions of the process steering) the thickness limiting of the zone of compounds on the surface or even its total elimination. Thanks to this it is possible to limit the allowance for the necessary, costly and the time consuming finish machining like grinding operation or even resigning of carrying it out. As the effect it will result not only in the considerable savings, but also proper stresses distribution of the internal stresses in nitrided layer, what is particularly important in components which are used in the aviation. Additional advantage of this method is also considerable shortening of the time of the nitriding itself and also the energy-saving process, what unquestionably will have the impact on production cost reduction.

Modernizing the traditional nitriding process by substituting it with ion nitriding will allow to eliminate the copper plating operation in manufacturing process. This galvanizing process is used in selective protection against nitriding. Copper plating is particularly expensive, energy and water consuming, threat-creating to the natural environment. Due to this not only the production cost will be reduced but it will also considerably limit usage of substances dangerous for the natural environment and maintenance - such as chromic acid anhydride. Usage of chromic acid anhydride as per directive of European Parliament will be prohibited by the 1st of January 2012.

In work the examples of modernizing of the nitriding process were discussed on chosen parts having the application in the aviation. The economical analysis has also been presented as well as ecological and legal aspects which argue for the modifications implementation to technological process used up till now. The investigation results acknowledged possibilities of practical use of proposed processes in industry practice.

Keywords: gas nitriding; Ion nitriding; Galvanic treatment; Environment protection; Production costs

Przemysł światowy w coraz większym stopniu wykorzystuje proces azotowania do wytworzenia technologicznej warstwy wierzchniej (TWW) elementów maszyn i urządzeń pracujących w warunkach wysokich obciążeń stykowych. W praktyce przemysłowej zastosowanie znalazło nie tylko tradycyjne azotowanie gazowe, ale także cały szereg nowoczesnych jego odmian pozwalających na dowolne kształtowanie struktury TWW.

Zastosowanie azotowania jonowego pozwoliło, poprzez kontrolę warunków prowadzania procesu, na ograniczenie grubości strefy związków na powierzchni lub nawet jej całkowite wyeliminowanie. Dzięki temu możliwe jest ograniczenie naddatku na niezbędną, kosztowną i czasochłonną obróbkę wykończającą w postaci szlifowania lub nawet rezygnacja z jej wykonania. W efekcie uzyska się nie tylko znaczne oszczędności, ale także prawidłowe widmo naprężeń własnych warstwy azotowanej, co jest szczególnie istotne w elementach mających zastosowanie np. w lotnictwie. Dodatkową zaletą tej metody jest także znaczne skrócenie czasu azotowania i energooszczędność procesu, co niewątpliwie wpływać będzie na zmniejszenie kosztów produkcji.

Modernizacja procesu azotowania poprzez zastąpienie metody tradycyjnej azotowaniem jonowym pozwala na wyeliminowanie z procesu technologicznego szczególnie drogiej, energo- i wodochłonnej, stwarzającej zagrożenie dla środowiska naturalnego obróbki galwanicznej w postaci miedziowania, stosowanej do ochrony wybranych powierzchni części przed azotowaniem. Dzięki temu nie tylko zmniejszy się koszty produkcji, ale także znacznie ograniczy stosowanie niebezpiecznych dla środowiska naturalnego i obsługi substancji niebezpiecznych np. bezwodnika kwasu chromowego, którego stosowanie w myśl dyrektywy Parlamentu Europejskiego będzie zakazane od 01 stycznia 2012.

W pracy omówiono przykłady modernizacji procesu azotowania wybranych części mających zastosowanie w lotnictwie. Przedstawiono analizę ekonomiczną, jak również aspekty ekologiczne i prawne przemawiające za wprowadzeniem modyfikacji

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dotychczas stosowanego procesu technologicznego. Uzyskane wyniki badań potwierdziły możliwość praktycznego zastosowania zaproponowanych technologii w praktyce przemysłowej.

1. Introduction

Nitriding process is right next to the carburizing and cyaniding mostly spread in industrial practice thermochemical treatment. Specific properities of nitriding layers and relatively low temperature of the process and small distortions, what makes this process more often applied in very conservative aviation industry.

As it is known structure of TSL obtained in gas nitriding may be quite complex. Next to the internal nitriding zone there can be also distinguished consisting from ε and γ ' phases so called "white layer". It characterizes itself considerable brittleness and porosity, therefore its appearance in TSL in most cases of machine parts is disadvantageous. Therefore these parts are submitted to grinding after nitriding process, in which the "white layer" is being removed. Nevertheless what stays in connection with a total cost and time of production increase, what is well known that in age of growing competitiveness between producers should be avoided. Moreover this kind of processing sometimes is plainly impossible to carry out. Grinding stays also with connection with appearance in surface layer unfavorable tension stresses for the sake of working conditions of he components.

Applying of ion nitriding by steering such as parameters as: pressure, reactive gas composition, voltage-current conditions of glow discharge in fact arbitrarily form the structure of nitrided layer produced in relationship with forecasted working conditions of produced parts [1-3]. Additionally ion nitriding process in comparison to gas nitriding as the result of smaller influence of hydrogen increases the plasticity of layers obtained.

Ion nitriding in comparison to classic method unquestionably has many advantages. First of all it is energy-saving and less material consumable. Instead of arduous ammonia NH3 as working gases more ecological are used: nitrogen N2, hydrogen H2 and argon Ar. So the need of utilization of waste gases is being eliminated. The process of nitriding itself is shorter and enables more uniform and repeatable layers. Above that it can be used to process on very complicated components. Unquestionable advantage of ion nitriding is also maintaining of surface smoothness of processed components, and also possibility of usage of so called cathode sputtering until final dimensions correction and also traces of nitrogen compounds zone removal [1-4]. Pointed advantages cause, that the process of ion nitriding is more often used in machine, automotive and aviation industries. Crankshafts of compression-ignition engines, highly loaded gears, cams and cam shaft, casings of injection

pumps for fuel injection in diesel engines, tools for plastic cold and hot forming are being treated this way and so on. Considerable group of treated compounds are also parts manufactured in powder metallurgy way [1,3,5].

As it is commonly known that the privileged places for chemical reaction initiation on the surface of the processed compound are the areas which characterize themselves as the areas with elevated internal energy, as grain boundaries, surface defects different phases compounds release [6]. In these areas as the effect of intensive superficial reactions (adsorption, dissociation) what occurs is relatively fast saturation of substrate with nitrogen and nucleation of nitric phases. One should to treat the surface of nitrided parts in way to increase in highest degree the amount of active centers on it. In industrial practice technological operations are often used in way to achieve that such as [1]:

- mechanical cleaning (sand blasting, zirconium blasting),

– phosphatizing,

- adding the reactive substances for example halides to processing atmosphere.

The process of mechanical cleaning can be carried out in heat treatment shop (PMH). Anyway it requires of usage special stock of machine tools and also special abrasive material (brand, granulation), which will be not only slightly detrimental for surface of processed parts but also will fulfill requirements of Polish legislation in field of natural environment protection and also staff healthiness.

Activation of nitrided surfaces with usage of galvanic processing (etching, phosphatizing) requires of carrying out series of treatments (operations) on specially equipped galvanic processing shop (PMG). These operations are not only time consuming, but also water and energy-consuming. Above that the wastes which are formed during their carrying out are considerable threat for natural environment and that is why they should be properly neutralized and utilized, so they fulfill the requirements defined by legislative authorities acts (Dz.U. z 2001r Nr 62 poz.627, Dz.U. z 2001r Nr 62 poz.628, Dz.U. z 1991r Nr 116 poz.503, Dz.U. z 1999r Nr 50 poz.501 and others), what is really costly.

Oxidation is most often carried out in the same installations as for the nitriding process. Nevertheless it prolongs the time of thermochemical treatment. Except that this method cannot be used for materials which easily passivate such as heat-resisting steel. In these cases

[–] etching,

⁻ oxidation,

quite popular is usage of proper substances chemically reactive for example ammonium chloride NH_4Cl or carbon tetrachloride CCl_4 , which are added to nitriding atmosphere. However in spite of high significant toxicity their use is serious threat heat treatment shop staff and also for natural environment [1].

Presented above processes of surface activation before gas nitriding are nowadays commonly used in industrial practice. However they are connected with unnecessary costs generating, which have fundamental influence on the total costs of the process of thermochemical treatment carrying out, and its profitability itself. Above that they make the real source of threat for natural environment and staff. This is the reason why it has been searched for such activation method, which in considerable degree would eliminate the faults of currently used methods. Most advantageous effect has been obtain by usage of cathode sputtering. By high energy ion bombardment of processed batch, surface oxides layers are removed and gases adsorbed. Above that the surface of the processed component is being unfolded, and also new structural defects are being generated which have essential role in process run as chemisorption and diffusion. The period of incubation and nucleation of nitric phases is being shortened this way. There is no problem with passivating of materials which are generally difficult to nitride [1, 6-7].

Significant advantage of cathode sputtering is, that it takes place all the time during the ion nitriding process. Thanks to this the surface of processed part during the whole process stays active. So there is no need to use costly operations which activate the surface what takes place in gas nitriding. Considerable savings of time and also internal costs are obtained. Above that one eliminates usage of substances which can be hazardous both for natural environment and people operating the process. It can be stated, that this is pro ecological method, which will have more common use in industrial practice.

If only possible the processed components should be nitrided totally. So performed operation is the simplest to carry out and at once the cheapest. However there are cases, when there are chosen areas to be nitrided. In this case surfaces not nitrided are protected by applying the protective coating. It can be both special stop-off paste available in trade and what is more often used in industrial practice galvanic coatings of tin, nickel, or copper.

In WSK KALISZ as the protective layer the electrolytic copper is used with thickness not less than $50\mu m$. Crucial disadvantage of copper plating usage in manufacturing process is a need to use hazardous chemical compounds, both on stage of preparation of batch (washing, degreasing) and also during the copper layer deposition. As an agent for degreasing organic sol-

vents are used of TRI or PER types, and also water solutions containing: sodium hydroxide NaOH, tribasic sodium phosphate Na₃PO₄·12H₂O and sodium carbonate Na_2CO_3 with proper concentration [8]. In case of etching, having on purpose thin oxide layers removal from the surface of components and also surface activation to assure good adhesion of layer to substrate, the aqueous solutions of sulfuric acid H₂SO₄ and nitric acid HNO₃ are used. The copper plating process itself is performed in baths containing toxic chemical compounds like: cuprous cyanide CuCN, sodium cyanide NaCN (inhibiting tissue oxidation quickly lead to death living organisms). Above that their composition consist also of: sodium hydroxide NaOH, sodium carbonate Na₂CO₃ and sodium-potassium tartrate NaKC₄H₄O₆·4H₂O [8-9]. Of course instead cyanide baths the acid baths (sulfate) or pyrophosphate can be used, which characterize themselves with less throwing power and covering power than cyanide baths. Moreover they cannot be used for direct copper plating of steel. For cyanide bath chemical yield is assumed to be of the order of $0.8m^2$ copper plated surface for 1m³ of bath. In case of sulfate baths this chemical yield equals yet about $0.4m^2$ from $1m^3$ of bath. This is also the reason why the cyanide baths regardless toxic compounds which are in composition have found the wider appliance in industrial practice.

Quite crucial problem which create the highest toxic threat when it comes to usage of deposited copper layer is its removal from thermo-chemically treated components. Most often used way of copper stripping is chemical dissolving method. The copper stripping process is performed in solutions containing chromic acid anhydride CrO₃ and sulfuric acid H₂SO₄ [8]. Total time of copper stripping takes about 4÷6 hours using new bath. Maximum time of copper stripping of parts takes 12 hours. Used in bath chromic acid anhydride causes the biggest threat both for the crew and natural environment, therefore usage of it on territory of European Community will be prohibited by the 1st of January 2012. This compound is carcinogenic and mutagenes one. It act very toxically through breathing ways and can be the cause both hereditary genetic faults and also fertility disorders. After getting to aqueous environment it can cause long lasting unfavorable changes [10]. That is the reason why it is so important to guarantee proper ventilation during the copper stripping process and also neutralization of sewers formed and utilization wastes as per the Polish legislation acts requirements (Dz.U. z 2001r Nr 62 poz.627, Dz.U. z 2001r Nr 62 poz.628, Dz.U. z 1991r Nr 116 poz.503, Dz.U. z 1999r Nr 50 poz.501, Dz.U. z 1998r Nr 55 poz.355 and others), what is costly.

To sum up, it can be said that galvanic treatment of components designated for nitriding is not only time consuming, but also water and energy-consuming and in addition dangerous for staff and also natural environment. Used in galvanic processes substances form local sources of air contamination because they can be emitted to the atmosphere in form of gases or aerosols. Most arduous for the atmospheric air arise during etching the steels in sulfuric and nitric acids – source of hydrogen chloride nitric oxides emission, and also in cyanide and chromic baths – source of hydrogen cyanide and aerosol of chromium compounds. Above that big threat for natural environment are also post galvanic sewers which are to be properly neutralized before letting them into the sewage system.

By usage the ion nitriding one obtains by applying special mechanical covers possibility of protection chosen surface regions e.g. threads against nitriding. One eliminates this way galvanic treatment which is bigger threat for natural environment and crew, more over elevating the cost of part manufacturing. Applying the covers lets also to eliminate not much expensive but arduous in carrying out pasting operation. Thanks to this one can avoid mechanical working necessary for paste (stop-off) removal and in unison cost of parts manufacturing elevation.

2. Nitriding process modernization

Modernization of nitriding process has been made on two chosen parts being in series production in WSK KALISZ. As research parts were chosen: side connecting-rod pin (sample No 1) showed in figure 1, made of steel grade 41CrAlMo7-4 and also ring gear (sample No 2) presented in figure 2, made of steel grade 32CrMoV12-9-3. Chemical composition of steels has been determined by certificates supplied by steel producers given in tab.1.



Fig. 1. Side connecting-rod pin

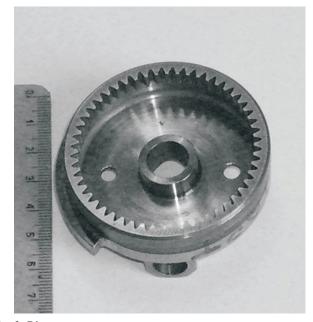


Fig. 2. Ring gear

TABLE 1

Chemical composition of 41CrAlMo7-4 and 32CrMoV12-9-3 steels

Grade	C	Mn	Si	Р	S	Cr	Ni	Mo	Cu	Al	Ti	W	V	Sn
41CrAlMo7-4	0,38	0,42	0,35	0,011	0,001	1,45	0,09	0,19	0,19	0,95	0,045	0,05	0,01	-
32CrMoV12-9-3	0,34	0,64	0,30	0,007	0,001	2,99	0,07	0,89	0,08	0,028	_	-	0,29	0,008

Grade steel 41CrAlMo7-4 has been delivered in softened state therefore it has been hardened and tempered as per the parameters given in the tab.2. This process has been taken into account in manufacturing operation sheets of the pin. After so carried out treatment its strength was equal to Rm = 1078MPa, what as per PN-93/H-04357 responds to hardness 345HV. The steel 32CrMoV12-9-3 has been delivered by a supplier in hardened and tempered state. Its tensile strength was

equal to $Rm = 1138 \div 1143MPa$, what as per DIN 50.150 responds to 363HV.

TABLE 2

Parameters of hardening and tempering of 41CrAlMo7-4 steel

Operation name	Device	Temperature	Quenching medium
Hardening	PEK – 4	940 °C	Water temp. $20 \div 45^{\circ}C$
Tempering	PEK – 4	610 °C	Air

Samples which were real production parts were sub-

jected to a gas nitriding (designation A) and ion nitriding (designation B) as specified in table 3. Ion nitriding components were not plated with an electrolytic copper on surfaces which were not supposed to be subjected to nitriding. In this case special steel S235JRG1 mechanical shields were designed.

TABLE 3

	Gas Nitrid	ing	Ion nitriding		
Sample designation	1A	2A	1B	2B	
Furnace type	GVN 80/180	GVN 80/180	Plasimp 900/900	Plasimp 900/900	
Temperature	I grade 510°C II grade 540°C	500°C	525°C	500°C	
Time	65h	25h	26h	3h	
Discharge current	-	-	10A	5,5A	
Discharge voltage	-	-	460V	420V	

Nitriding process parameters

Samples which were used for metallographic inspection were wet cut by a disk-type grinding wheel. After mounting the samples they were grinded on papers SiC with 60-800 gradation. After that they were polished on a STRUERS MS Allegro and MD Plus polishing wheels applying DP Suspension P with grains of size $6\mu m$ and $3\mu m$. Microscopic inspection of surface layer were made using a AXIOUERT 25 Carl Zeiss microscope. Microhardness measurements were made using a Vickers method by LECO type LM-700AT microhardness tester with load of 500 grams.



Fig. 3. Microstructure of nitrided layer - sample Nº 1A etched with Mi1Fe, mag. x500



Fig. 4. Microstructure of nitrided layer – sample $N^{\rm o}$ 1 A etched with Mi1Fe, mag. x500

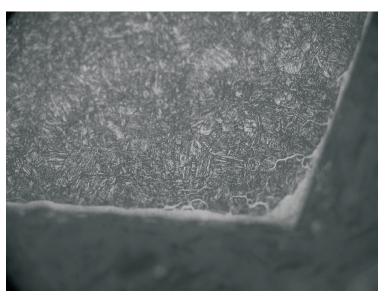


Fig. 5. Microstructure of nitrided layer – sample N $^{\rm o}$ 2 A. x500 etched with Mi1Fe, mag. x500

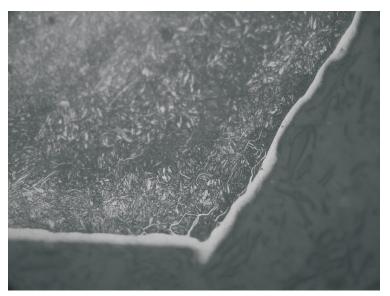


Fig. 6. Microstructure of nitrided layer – sample N° 2 B. etched with Mi1Fe, mag. x500 $\,$

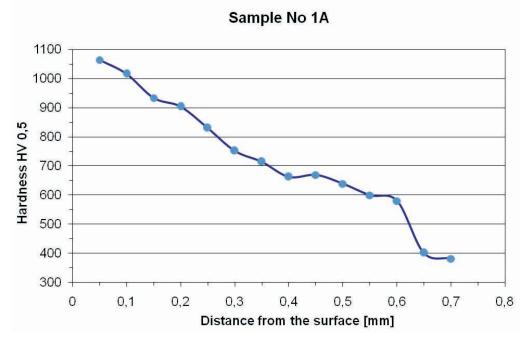
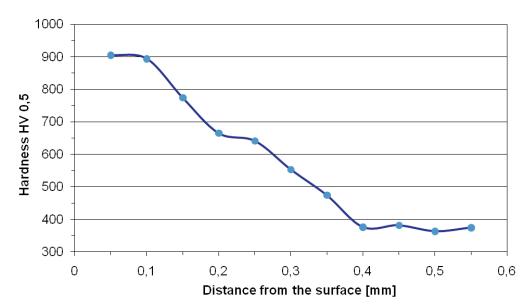


Fig. 7. Microhardness pattern in surface layer of sample Nº 1 A



Sample No 1B

Fig. 8. Microhardness pattern in surface layer of sample Nº 1 B

Sample No 2A

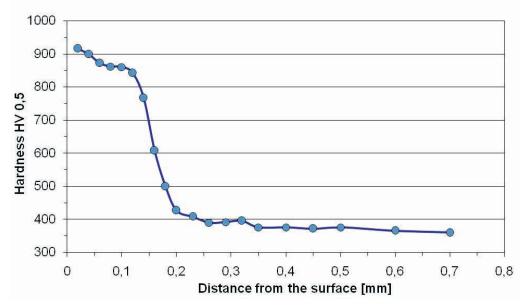
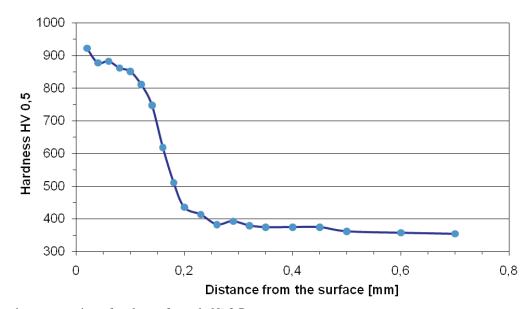


Fig. 9. Microhardness pattern in surface layer of sample $N^{\rm o}$ 2 A



Sample No 2B

Fig. 10. Microhardness pattern in surface layer of sample $N^{\rm o}$ 2 B

	Sample N ^O 1 A	Sample N ^O 1 B	Sample N ^O 2 A	Sample N ^O 2 B
Surface hardness	1093HV	917HV	923HV	941HV
Core hardness	345HV	345HV	363HV	372HV
Nitrided layer thickness	0,63mm	0,38mm	0,19mm	0,18mm
"White layer" thickness	16µm	-	8μm	6µm

TABLE 4

TABLE 5

Requirements for nitrided layers

	Surface	Core	Nitrided layer	"White layer"
	hardness	hardness	thickness	thickness
Sample N ^O 1	min. 793HV	310÷345HV	0,35÷0,70mm	-
Sample Nº 2	800÷950HV	340÷400HV	min. 0,15mm	max. 0,01 mm

The result which were obtained during the thermochemical treatment of inspected samples and the conditions which are to be fulfilled on produced surface layers of the inspected components were put together in the tables 4 and in tab.5. On these basis we can affirm, that a sample in a gas nitration is significantly higher hardness on a surface than in comparison to ion nitriding (tab. 4). Moreover for both variants of nitriding of the sample Nº 1 different profiles of nitriding microhardness were obtained (fig.7-8). This effect is caused mainly by an appearance of nitrides in a structure of a gas nitrided layer (sample 1A) which create so called "white layer" 16µm thick (fig. 3). Because of working character (load) of a side connecting-rod pinion, these nitrides are unwanted. Future manufacturing process for this part forecasts grinding operation in purpose to grind off the "white layer". Due to additional grinding operation extra allowance was applied of $0.12 \div 0.18$ mm in radius. Therefore the nitrided layer on a sample 1A is much thicker (tab.4) and with different profile of microhardness than on a sample 1B (fig.7-8). By applying ion nitriding (sample 1B) much more advantageous structure was obtained, which does not contain nitric compounds which create "white layer" (fig. 4). Taking this into consideration there is no need to grind the parts after nitriding, and the layer which has to be produced can be thinner (0,38mm) than in a gas nitriding (0,63mm).

In case of a ring gear (sample N^O 2) not only the same structure of nitrided layer was obtained (fig. $5\div6$) but also similar micro-hardness traces (fig. $9 \div 10$). The similarities result from so far applied manufacturing pro-

cess which did not forecast grinding operation and additional allowance on nitrided surfaces related to this. Applying of ion nitriding in this case was to limit the work consumption in manufacturing process of gear.

To define the work consumption of manufacturing the part there are two times given – setup time (Tpz) and basic time (Tj). The Tpz time contains the time which an operator needs to get acquainted with the task and make preliminary steps before starting the main work (for example picking up the components for work, all of the instructions, necessary tools etc.) This time isn't defined in thermal and galvanic treatment. Basic time (Tj) is the principal time provided for processing one component during one operation. Taking that into consideration in tab.6, the course of the technological process of a side connecting-rod pin with specification of Tpz and Tj time executing for given stage of production process for each component is presented.

In table 6. technological process run has been showed for side connecting-rod pin with pointing of setup time (Tpz), and the basic time (Tj), which is assigned for specified phase of technological process. As it can be seen total setup time (Tpz) after modernizing of nitriding process equals Tpz = 666 min. So it has been shortened by 14,72%. In case of the basic time (Tj) it has been correspondingly Tj = 62,88 min., what makes reduction by 10,77% relatively to original time. Reduction of Tpz and Tj was possible by eliminating galvanic treatments from technological process and limiting mechanical working (grinding) after thermochemical treatment.

TABLE 6

Sample Nº 1 A		Sample Nº 1 B			
Processing type	Tpz [min.]	Tj [min.]	Processing type	Tpz [min.]	Tj [min.]
Heat treat. – toughening	–	3,09	Heat treat. – toughening	–	3,09
Mechanical working	466	38,2	Mechanical working	466	38,2
Galvanic treatment - copper plating	_	0,82	Galvanic treatment – copper plating	_	_
Mechanical working	105	3,2	Mechanical working	105	3,2
Galvanic treatment – phosphatizing	-	0,27	Galvanic treatment – phosphatizing	-	-
Thermochemical treatment	_	2,15	Thermochemical treatment	-	0,75
Mechanical working	190	15,93	Mechanical working	75	8,0
Galvanic treatment – brass plating	_	3,5	Galvanic treatment – brass plating	-	3,5
Mechanical working	20	2,98	Mechanical working	20	2,98

Technological process run of side connecting-rod pin (sample Nºr 1A and 1B)

In case of ring gear (tab.7.) the time was not shortened because none of the mechanical working processes were removed. The difference in processes only consist in that in case of ion nitriding usage whole mechanical working is done before the nitriding. Nitriding is carried out in this case on "blue print" made part. The Tj time saving has been obtained, because it has been shortened by 8,54%. Saving on this results from ion nitriding usage, and also by eliminating from the process the galvanic treatment.

TABLE 7

Technological process run of side connecting-rod (sample Nº 2A and 2B)

Sample N ^o	2A	Sample N° 2B				
Processing type	Tpz [min.]	Tj [min.]	Processing type	Tpz [min.]	Tj [min.]	
Mechanical working	410	48,9		780		
Galvanic treatment – copper plating	-	1,2	Mechanical working		202,0	
Mechanical working	110	73,6				
Thermochemical treatment	-	21,4			2.4	
Mechanical working	260	79,5	Thermochemical treatment	-	3,4	

3. Summary

By modernizing thermochemical treatment it is possible to:

- reduce the cost of production of part by lowering the labor consumption,
- eliminate the harmful and hazardous substances (i.e. chromic acid anhydrate) for natural environment and the treatment operators from technological process,
- reduce of usage electrical energy and working media (water, technical gases),
- production of surface layer totally fulfilling design requirements.

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