Chemical Compounds Analysis Developed on the Micro Alloying Area of Coating Layers Obtained by Impulse Discharge Method

MANUELA CRISTINA PERJU, PETRICA VIZUREANU*
„Gheorghe Asachi" Technical University of Iasi, Faculty, of Materials Science and Engineering, 41 Dimitrie Mangeron Blv.., 700050, Iaşi, România

The paper presents the chemical analysis of coating layers obtained by impulse discharge method, using X-Ray Diffraction Analysis (XRD) and highlights the presence inside this layer of complex chemical compounds that explains the mechanical proprieties of surface layer (micro-hardness). Electrical impulse discharge method is used for coating some installation components that work in heavy abrasive conditions in wet and dry environments.

Keywords: chemical compounds, XRD, thin layers

The wear, fatigue and corrosion phenomenon are the most aggressive factors that lead to scrapping metallic parts. From this situation results the high importance for the physico-chemical research of the phenomenon that takes place into these superficial layers in connection with coating technologies [9].

One of the methods to process the materials is electrical impulse discharge method that can allow to coat complex shaped and reduced dimensions parts by extracting material from the surface of the part or developing coating layers by applying the compact material electrodes or by introducing into working area of powders or mix of powders. It is necessary that the surface layers to have a good adherence to the part and especially a good connection to the sub-layer. In some surface areas have to exist high hardness layers. Most studies focused on chemical, physical and mechanical proprieties of deposited layer and on mass transfer behaviour, to obtain the proprieties imposed, and to control as much as possible the coating layer thickness, [1-3, 9-11].

The technology can be defined as a micro-pulsed welding technique, that allows deposition of one "electrode material" on a metallic sub-layer; mass transport made in high current intensity and in short time periods, [5].

Impulse discharge method involves thermal and mass transfer phenomenon. Practically "we discuss" about multiple phenomenon, heating to incandescence, melting and eventually vaporizing the electrode material.

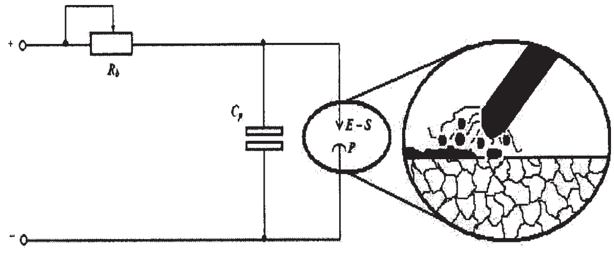

Fig. 1. Metallic surface processing with electric impulse method; a) Electric sketch of processing device: C_p=condenser, R_b=variable resistance, E-S=electrode connected to anode, P=part connected to cathode; b) Superficial sketch of the process

* email: peviz2002@yahoo.com; Tel.: 0744793984

An electron flux is developing as well as ions and, fast neutral atoms inside electric field between electrodes. Electric field is concentrated on a micro-portion from the part where the melting metallic bath is forming. Micro-alloying between base material and electrode melting has as effect nitride forming reactions, carbide and carbide-nitrides and plasma forming by air decomposition (nitrogen and active oxygen). This entire phenomenon, and also others, describe in a succession, the way of hardened by deposition superficial layer is formed.

The study based on using electric impulse discharge method in textile industry focused on friction cam parts and gears from looms which are made of ferrite-perlitic grey cast-iron.

Experimental part

Method and materials

To obtain thin layers, impulse discharge method was used, based on polarized erosion effect and on anode material transfer (electrode) on the cathode (part).

Tests were made with the Elitron 22A type machine [12].

Materials

As base material for the coating, ferrite-perlitic cast iron was used, whose chemical composition is presented in table 1, [7]. The chemical composition was determined with Foundry Master Spectrometer.

Table 1
CHEMICAL COMPOSITION OF BASE MATERIAL, %

C	Si	Mn	P	Cr	Ni	Cu
3.97	2.87	0.25	0.06	0.28	0.12	0.17

The choice of the base material is given by the advantages of this material in textile industry machines and installations (cams, friction skates and gears in looms), [4, 6, 8]. Grey cast-iron is a material with a good thermal shocks resistance and does not require lubrication. Expanding and contraction coefficients are low and have the propriety to absorb easily apparent tensions during work because of the graphite from the matrix which is creating discontinuities that can absorb vibrations and shock exposure.

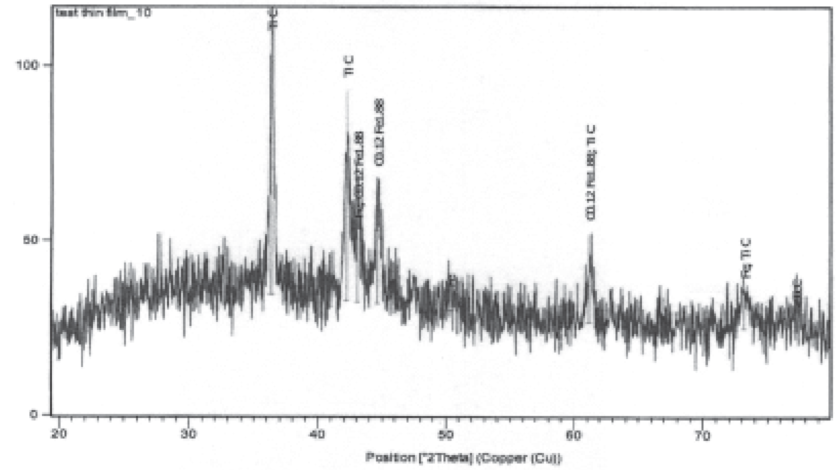

Fig. 2. XRD analysis for ferrite-perlitic sample coated with TiC simple layer highlighting the maximum areas for chemical compounds (TiC, $C_{0,12}Fe_{1,88}$)

Table 2
ANGLED ANNALYSIS FOR TiC SIMPLE LAYER COATING

Nr.	Name component	Chemical formula	Quantity, %
1	Austenite	Fe	12
2	Martensite	$C_{0.12}Fe_{1.88}$	21
3	Titanium carbide	TiC	67

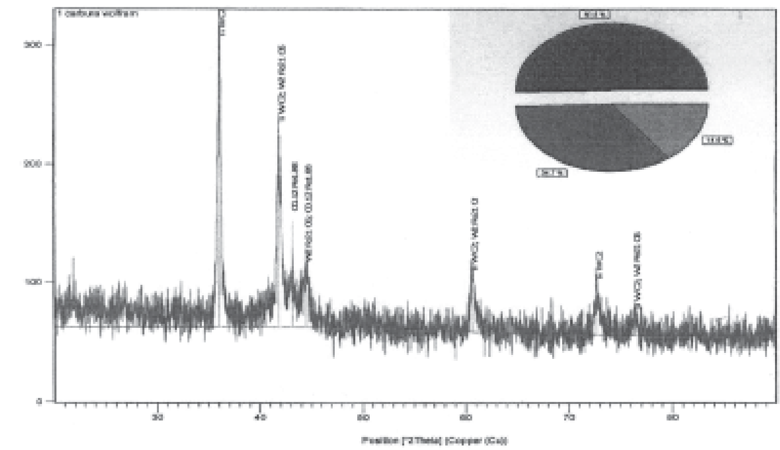

Fig. 3. XRD analysis for ferrite-perlitic cast-iron sample part coated with Ti/WC highlighting maximum areas for chemical compounds ($W_2Fe_{21}C_6$, $TiWC_2$, $C_{O,12}Fe_{1,88}$)

Table 3
ANGLED ANALYSIS FOR Ti/WC COATING

No.	Component name	Chemical formula	Quantity, %
1	Ti carbide	TiWC2	35
2	Iron and Wolfram carbide	$W_2Fe_{21}C_6$	50
3	Martensite	$C_{0.12}Fe_{1.88}$	15

Electrodes used for deposition are Ti, TiC and WC. TiC one layer and Ti/WC, WC/TiC two layers coatings were made. Ti and TiC electrodes give good compactness and adherence, and WC electrode creates surface hardness.

XRD Technique

X-ray scattering techniques are a family of non-destructive analytical techniques which reveal information about the crystallographic structure, chemical composition, and physical properties of materials and thin films. These techniques are based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy.

The atomic planes of a crystal cause an incident beam of X-rays to interfere with one another as they leave the crystal. The phenomenon is called X-ray diffraction.

Results and discussions

XRD spectrum is formed by a series of peaks that represent the photoelectrons that come from different energetic levels from the atom. Presence of a particular energy peak is indicating the presence of a specific element inside the studied sample, even more, the intensity of the peak is related to the concentration of the element in that area. High percentage of some elements can be identified at the surface and their concentration can be measured on areas with diameters from few microns to millimeters. Connection stages in which these elements are engaged and the depth distribution can be determined on few nanometers. For this, angled analysis can be applied; particles are emitted in certain angles.

Analysis were made on coated samples with TiC one layer Ti/WC double layer and WC/Ti double layer with X raze diffract-meter "X Pert Pro Philips Analytical", where the maximum energetic peaks give indication about chemical compounds of the element from the energy area corresponding to photo-electronic spectrum of kinetic energy.

XRD analysis for TiC simple layer coated sample

Sample part was analyzed using angled method (fig.2). Like this were determined chemical connections corresponding to compounds found in the superficial layer: 67% mass percentage of TiC, martensite ($C_{0,12}Fe_{1,88}$) 21% mass percentage and austenite 12% mass percentage (table 2).

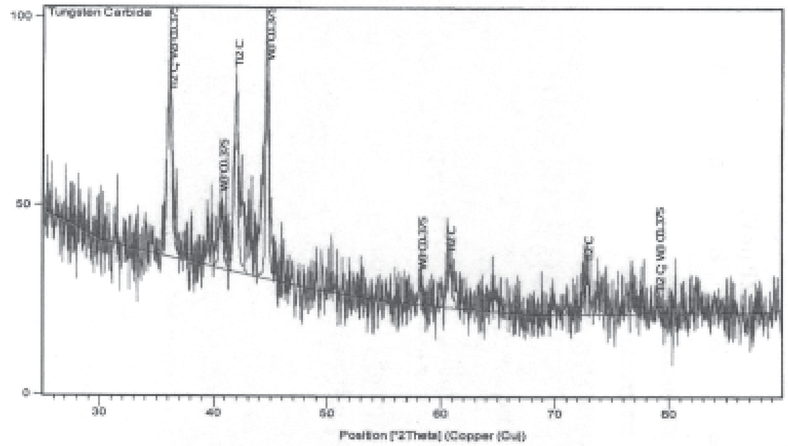

Fig. 4. XRD analysis for ferrite-perlitic cast-iron sample part coated with WC/Ti highlighting maximum areas for chemical compounds ($W_3C_{0,375}$, Ti_2C)

Table 4
ANGLED ANALYSIS FOR WC/Ti COATING

Nr.	Component name	Chemical formula	Quantity, %
1	Titan carbide	Ti2C	89
2	Wolfram carbide	$W_3C_{0.375}$	11

XPS analysis for Ti/WC coating double layer

According to the measurements on few nanometers depth the following structure was determined (table 3): 50.5% $W_2Fe_{21}C_6$, 34.7% $TiWC_2$, 14.9% $CO_{12}Fe_{1,88}$. For this sample XRD analysis for chemical connections, angled type, of incident ray at the surface of ferrite-perlitic cast-iron base material on which double Ti/WC layer was deposited was applied(fig.3).

XRD analysis for WC/Ti coating double layer

Angled analysis method was used to determine the chemical compounds that appear at the surface of the sample of ferrite-perlitic cast-iron coated with double WC/Ti layer (fig.4). Mentioned combination has generated according to measurements, mainly, two compounds: $W_3C_{0,375}$ and Ti_2C, 89% Ti_2C mass percentage and 11% $W_3C_{0,375}$ mass percentage (table 4).

Conclusions

For TiC one layer coating we observe martensite and residual austenite at the surface of the sample part, what means that besides coating a superficial quenching takes place. Adherence on a hardened layer improves a lot the resistance of the material. Martensite and austenite presence was possible on this sample because only one layer was deposited which usually is under 25μm, so in depth analysis could reach underneath the coating area where thermal influenced area developed.

For Ti/WC deposition, titan as intermediary layer, favours the complex carbides $TiWC_2$, $W_2Fe_{21}C_6$ type, what leads to exterior hardness increase and anchors the coating layer by mixing the phases also in case of melting and splashing dynamics but also by forming the complex chemical combinations.

In case of WC/Ti coating, the carbon absorbed from the active elements (chemical activated carbon) present in the metallic bath of the melting, from the deposited "drops" combines with titanium forming Ti2C. Mainly we can observe chemical compounds in detriment to pure stage elements what means that coating elements, like titanium, are developed due to temperature conditions, pressure and chemical reaction. These are favorable to hardness, adherence and interconnection of layers.

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