



"GREEN TECHNOLOGY" – TRANSFORMATION OF STAINLESS STEEL SURFACE BY ELECTROCHEMICAL BLACKENING



Michael Pasamanik, Shoshana Tamir, Malka Rotel
 Institute of Metal, Technion-Israel Institute of Technology, Haifa, Israel

ABSTRACT

Blackening of stainless steel is a method of final processing of products. It improves anti-corrosive properties of the product, it is used for decorative purposes, it also forms anti-glare surface layer and increases absorbability of electro-magnetical waves in infrared range. For these purposes chemical and electro-chemical methods are usually applied though being unsafe to the environment and to the potential users of the product. Our lab has developed a new ecologically safe technology of blackening of stainless steel, the products of which do not include any harmful components.



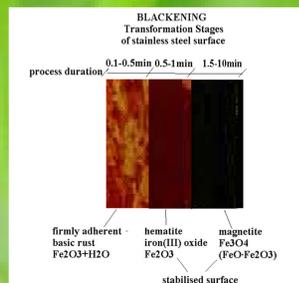
Electrochemical Reactor



Blackened Samples

INTRODUCTION

Physical meaning of blackening (obtaining a black oxide layer on the surface of stainless steel) is the concurrent oxidation-reduction reactions with iron, oxygen and hydrogen. The process of blackening of the stainless steel surface is the process of transformation of iron and other alloy components to a mixture of their oxides, in which the most significant component is the oxide of iron called "magnetite", which is the one that causes the surface to be black. The processes of both chemical and electrochemical blackening are actually modeling the classic oxidation/reduction reactions that take place in high-temperature oxygen/hydrogen steam-gas atmosphere or in the atmosphere of overheated water steam. The essential disadvantages of such oxidation methods are the following: high temperature heating of the whole sample which harms its mechanical properties for future applications, and ecological risks. Our goal was to construct the electrochemical oxygen-hydrogen reactor enabling controlled creation of local fast-moving micro areas of high temperature where the steam-air problems. Basing the research on the provided above reasoning which is related to the physical essence of phenomena occurring in steel blackening, we succeeded to select and optimize the above mentioned process parameters and to obtain the magnetite coating with the predefined properties. We have determined and researched the optimal parameters of blackening process for stainless steel under regular temperature and ecologically safe conditions during 5-7min. We have developed double-stage process of steels blackening: (fig.1) the first stage is an anode polarization during t1 which hematite is formed - color red -Fig.3a&3b., the second stage is a cathode polarization during t2 which hematite will be transformed in magnetite - color black.



Stages of Oxidation



Fig.3a Red color of a solution after an anode stage of blackening

RESULTS

Coating composition

Examination of the surface of blackened samples using electron microscope SEM/EDS, X-ray Photoelectron Spectroscopy method and Time of Flight Secondary Ion Mass Spectrometry method has demonstrated that the black oxide film on the surface has the thickness of about 5 micron in average and it consists of composite material – magnetite and chrome oxide at the ratio corresponding to the initial ratio in the basic metal. Coating composition of several black coating obtained are listed in table 1

All results in weight%			All results in atomic%			All results in compound%	
Spectrum	O	Cr	Fe	Spectrum	O	Cr	Fe
2	29.27	15.11	55.62	2	58.71	9.33	31.96
3	37.37	9.32	53.32	3	67.32	5.16	27.52
4	45.44	10.28	44.29	4	74.14	5.16	20.70

Table 1

Corrosion resistance

The corrosion resistance of the coating was evaluated by electrochemical corrosion measurement. Electrochemical measurements have shown that the OCP (Open Corrosion Potential) of the blackened sample corresponds to the OCP of magnetite and its potential +0.1V (vs. Saturated Calomel electrode) is much more positive than that at the initial sample -0.3V. The passivation area is increased from 0.3 V at the uncoated sample to 1.35 V at the blackened sample, thus our coating carries out good additional protection against corrosion.

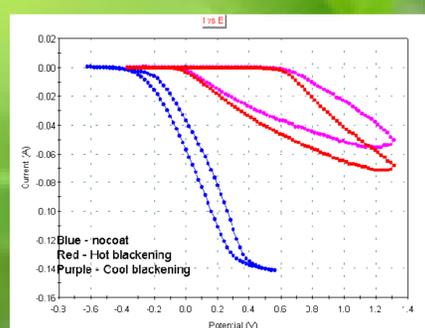
Fig. presents the graphs of electrochemical measurements:

For coated samples by "cool" method of blackening

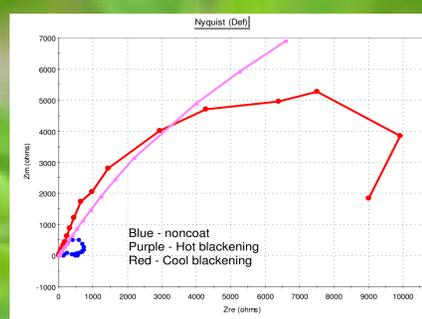
For non-coated samples

For samples coated using the known "hot" method of blackening

These graphs 1,2 show that corrosion resistance and impedance characteristics of "cold" and "hot" coatings are comparable, which proves our theoretical assumptions about the common physical meaning of blackening processes no matter what the technology is.



Graph1-Cyclic Polarization



Graph2 - Impedance



Rockwell Hardness	
1 - basis surgical tool without blackening	HRC - 46.2
2 - blackening surgical tool	HRC - 50.7
3 - blackening surgical tool	HRC - 50.8
4 - blackening surgical tool	HRC - 50.1
5 - blackening surgical drill	HRC - 44.7
6 - blackening metalworker's drill	HRC - 47.1

Hardness - Rockwell-test have shown that improvement of hardness is observed after blackening



Fig.1 – uncoated sample after Cyclic Polarization

The increased firmness against corrosion of the coated samples in comparison with uncoated samples is shown in pictures from the Optical microscope of figs 1,2. On uncoated samples (fig.1) it is visible strongly pronounced pitting after Cyclic Polarization – Pitting is not observed on the coated samples (fig.2)



Fig.2 – coated sample after Cyclic Polarization



Fig.3b Red color of a sample after an anode stage of blackening

Conclusion

We have determined and researched the optimal parameters of blackening process for stainless steel under regular temperature and ecologically safe conditions.

We were unable to find description of any similar process in the overviewed literature.

Our theoretical assumptions about the physical meaning of the process have been confirmed in experimental part of research.

The accumulated experience and knowledge of this process enable further research on the specific samples of different stainless steel.