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THE INFLUENCE OF TEMPERATURE AND TIME OF EXHIBITION ON A CHANGE OF Al-Si COATING THICKNESS AND SURFACE TEXTURE ON THE STEEL PLATES

WPŁYW TEMPERATURY I CZASU EKSPOZYCJI NA ZMIANĘ GRUBOŚCI I STRUKTURY GEOMETRYCZNEJ POWIERZCHNI POWŁOKI SILUMINOWEJ NA STALOWYCH TAŚMACH

The paper presents the results of the testing of the influence of temperature and time of heat treatment of aluminized steel plates on the change of the Al-Si thickness, Al-Fe-Si inter-metallic layer thickness and the geometrical structure of the coating surface. Also, tests on the influence of heat treatment on the changes in the surface appearance such as partial or total graying and dulling due to iron diffusion to the coating surface have been conducted. Characteristics showing the dependence of temperature on the speed with which initial and total changes in the appearance of the surface coating occur have been made. Tests have been conducted on samples of DX52D+AS120 aluminized steel plate with the surface coating thickness of ca 20 μm, before and after heat treatment in temperature 250-700°C within 1 – 10620min. Equipment for non-destructive measurements of coatings thickness and roughness has been used in these tests. A digital camera, an optical microscope and a scanning electron microscope have been used to show surface changes after heat treatment.
Keywords: aluminized steel plate, coating, thickness, inter-metallic layer, roughness, surface

W artykule przestawiono wyniki badań wpływu temperatury i czasu obróbki cieplnej, aluminiowanych taśm stalowych, na zmianę grubości powłoki Al.-Si, grubości warstwy międzymetalicznej Al.-Fe-Si oraz struktury geometrycznej powierzchni powłoki. Dodatkowo przeprowadzono badania wpływu obróbki cieplnej na zmianę wyglądu powierzchni powłoki, w postaci częściowego lub całkowitego jej zszarzenia i zmatowienia na skutek dyfuzji żelaza do powierzchni powłoki. Opracowano charakterystyki przedstawiające zależności wpływu temperatury na szybkość pojawienia się początkowych i całkowitych zmian wyglądu powierzchni powłoki. Doświadczenia prowadzono na próbkach z aluminiowanej taśmy stalowej z gatunku DX52D+AS120, o grubości powłoki ok. 20 μm, w stanie przed i po obróbce cieplnej, w temperaturze 250-700°C, w czasie 1 – 10620 minut. W badaniach wykorzystano przyrządy do nieniszczących pomiarów grubości i chropowatości powłok. Do zobrazowania zmian powłoki po obróbce cieplnej wykorzystano cyfrowy aparat fotograficzny, mikroskop optyczny i skaningowy mikroskop elektronowy.

1. Introduction

Aluminized steel plates have the characteristics which combine the advantages of steel and aluminum. The modulus of elasticity of steel is three times as big as compared to pure aluminum and the thermal expansion coefficient of steel is ca half as low as compared to aluminum. The advantages of aluminum which are transferred to aluminized steel plates are: resistance to corrosion, oxidation, high temperatures and thermal reflectivity up to 90%.

Steel is coated with aluminum during continuous processes, on lines similar to the ones used in hot galvanizing. Cold-rolled steel plate is heated and immersed into liquid aluminum or aluminum-silicon alloy (8 – 11 % Si).

The most important parameters of the aluminizing process include the temperatures of the steel plate and the coating alloy, contact time of the plate with the liquid alloy and the cooling method. These parameters influence the geometrical structure of the coating surface, coating thickness and the thickness of the alloy layer, which largely influence mechanical and physical properties of the aluminized plate as well as its corrosion resistance.

Hot dip coating has numerous advantages, such as good adhesion, the possibility of making an alloy with steel due to diffusion, density and smoothness of the

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coating, wear resistance, water resistance and the possibility of an automated production process. These advantages make hot dip coating a better method than other coating methods.

Requirements for aluminized steel plates are presented in [1-2]. Standard [3] specifies types of steel which can be coated with aluminum layers. The type of steel should be selected taking into consideration, among others, the need of forming of steel products.

Aluminized steel plates are used in car industry, household appliances, heat exchangers, heat screens, tanks, etc.

Parts of exhaust systems, where aluminized steel plates are found, operate in broad temperature ranges [4]. Changes in the temperature have an influence on changes of the structure and properties of the coating and the base and this influence is proven by the test results [5-13]. There is, however, no information about the changes in the thickness of the coating, the inter-metallic layer and the coating surface. Therefore, the decision about multi-variant tests has been made. These tests aimed at the specification of the influence of temperature and exposure time on the change of Al-Si coating thickness, Al-Fe-Si inter-metallic layer thickness and the geometrical structure of the coating surface as characterized by roughness parameters R_a and R_z. Also, tests enabling obtain the characteristics concerning the speed with which initial and total changes – graying and dulling – in the coating surface appearance after its heating, have been conducted. The tests made it possible to determine boundary values for the time during which the material can be kept under a specified temperature without any fear for the appearance of changes on the coating surface, taking into consideration the thickness of the coating. Considering the test results [8, 12], temperature which appears during the production of pipes, in the areas close to the weld zone [10] and under the operation conditions of exhaust systems [4], a wide range of temperature-time exposure of Al-Si-coated steel plates has been selected for the tests.

2. Experimental technique

Al-10wt%Si-coated steel plates type DX52D+AS120, which are most often used to produce parts of exhaust pipes, have been selected for testing. The chemical composition of the aluminized steel used in this study is shown in Table 1. Chemical composition was tested using optical emission spectrometry SPECTROLAB M7.

TABLE

Chemical composition of strip (wt.%)

C	Mn	Si	P	S	Cr	Ni	Nb	Cu	Al	Fe
0,004	0,14	0,006	0,009	0,011	0,02	0,022	0,014	0,017	0,045	Bal.

Tests were conducted in two stages. The first stage covered tests of Al-Si coating thickness, Al-Fe-Si inter-metallic layer thickness and coating roughness depending on heat treatment parameters. For this purpose, samples of 50x25mm were cut out of the plate. Heat treatment was performed in a chamber furnace in atmospheric air. The furnace was heated up in the range of 250-700°C, every 50°C. When the temperature became stabilized, samples were put into the furnace and were soaked for 30, 180 and 1440min. Then the samples were taken out of the furnace and were air-cooled up to 23±2°C. For each combination of time and temperature, three samples were assigned. Measurements were done on the samples before and after heat treatment. Observation of cross-section of coating structure and local and linear microanalysis of the chemical composition in initial state and after heat treatment were done, using Hitachi scanning electron microscope S3500N (SEM), equipped with NORAN Energy Dispersive Spectroscopy analyzer (EDS).

Thickness was tested using a non-destructive method with the help of Elkometer 345, which had been scaled on metal samples with a specified coating thickness. The samples were specially prepared for this purpose. Because of the fact that plates are made with a coating on both sides, coating thickness was measured on them accordingly. 13 measurements of coating thickness on each side of the each sample were done. Depending on the measurement result, sides of the sample were marked A for the smaller coating thickness and B for the bigger coating thickness for a given sample (Fig. 1). Results of the measurements are presented as graphs showing the dependence of the medium coating thickness on heat treatment parameters.

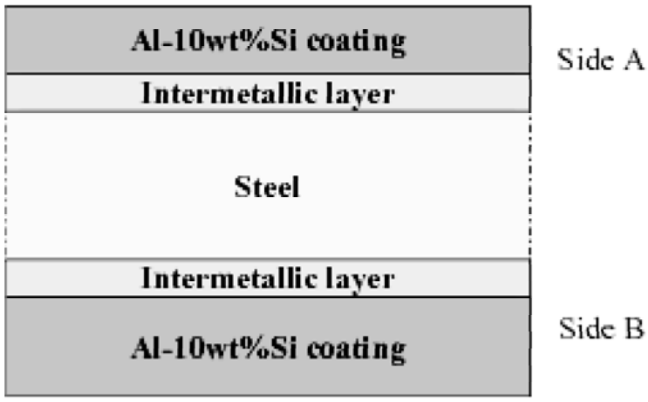


Fig. 1. Schematic illustration of the cross-section of Al-10wt%Si coating steel plate with sign of the sides of measurements A and B

Measurements of the inter-metallic layer thickness were done on samples selected for coating thickness measurements. Metallographic specimens were prepared and they were observed using SEM with EDS. Then, on the base of sectional coating structure pictures, 10 measurements of inter-metallic layer thickness were done, only on the side A. The quantity of the scale, which was present on the picture of the coating structure, was taken as a reference. Results are presented as a graph showing the dependence of the inter-metallic layer thickness on heat treatment parameters. Exemplary results of microscopic observations are also shown.

Measurements of coating surface roughness were done with the tracer method using Taylor Hobson Form Talysurf with an optical head. Two parameters specified by the standard [12], i.e. R_a and R_z, were measured. Medium numerical values of R_a and R_z parameters on the coating surface were obtained after repeating the measurements on each side (side A and B) ten times. A sampling length l_e = 0,8mm and a measuring length L_c = 4mm were used in the tests. No other surface errors, namely corrugation and shape deviation were considered. Results are presented as graphs showing the dependence of the roughness parameters R_a i R_z values on heat treatment parameters. Changes on the coating surface were recorded as pictures of exemplary macroscopic observations done using a digital camera and microscopic observations done using Neophot optic microscope.

The results of the observations of changes on the coating surface caused to do the second stage of the tests, which aimed at determining the characteristics concerning the speed with which initial and total changes of the appearance of the coating after heating, namely graying and dulling. Similarly to the first stage, samples of 50x25mm were cut out from the plate. Then, 10 measurements of the coating thickness on each side were carried out in order to select the plate side with a thicker coating for further observations. Samples with the coat thickness of 25±1 m were selected for the tests. Heat treatment was performed in a chamber furnace in atmospheric air. The furnace was heated up to the range of 100-700°C, within the temperature range of 100-450°C every 50°C, within the temperature range of 450-600°C every 10°C and 600-700°C every 20°C. When the temperature became stabilized, samples were put into the furnace and were soaked. Then, the soaking time, after which the first changes on the coating surface such as grey spots appear and the time, after which the coating surface is totally grey and dull, were determined. Also, some intermediate times, when the changes on the surface became more intensive, were set up. The samples were taken out from the furnace and were air-cooled up to 23±2°C. For each combination of time and temperature, three samples were assigned. The appearance of the coating surface on the samples was done before and after the heat treatment. The results of the measurements are shown on the graph. Changes on the coating surface were recorded as pictures of exemplary macroscopic observations done using a digital camera.

3. Results and discussion

3.1. Characteristics of the coating in initial state

Fig. 2 shows the result of the observation of the Al-10wt%Si coating surface in the initial state. The coating has a bright and smooth surface without peeling and irregularity of surface.

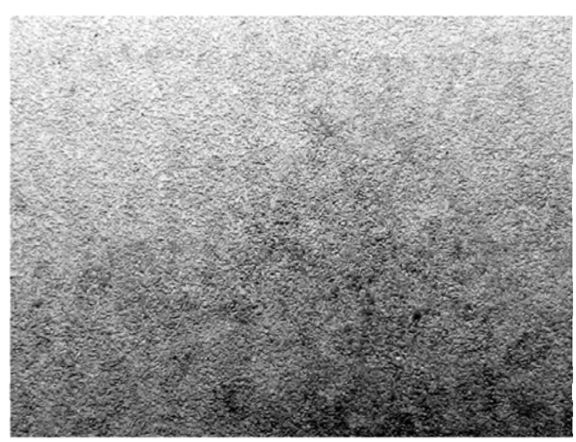
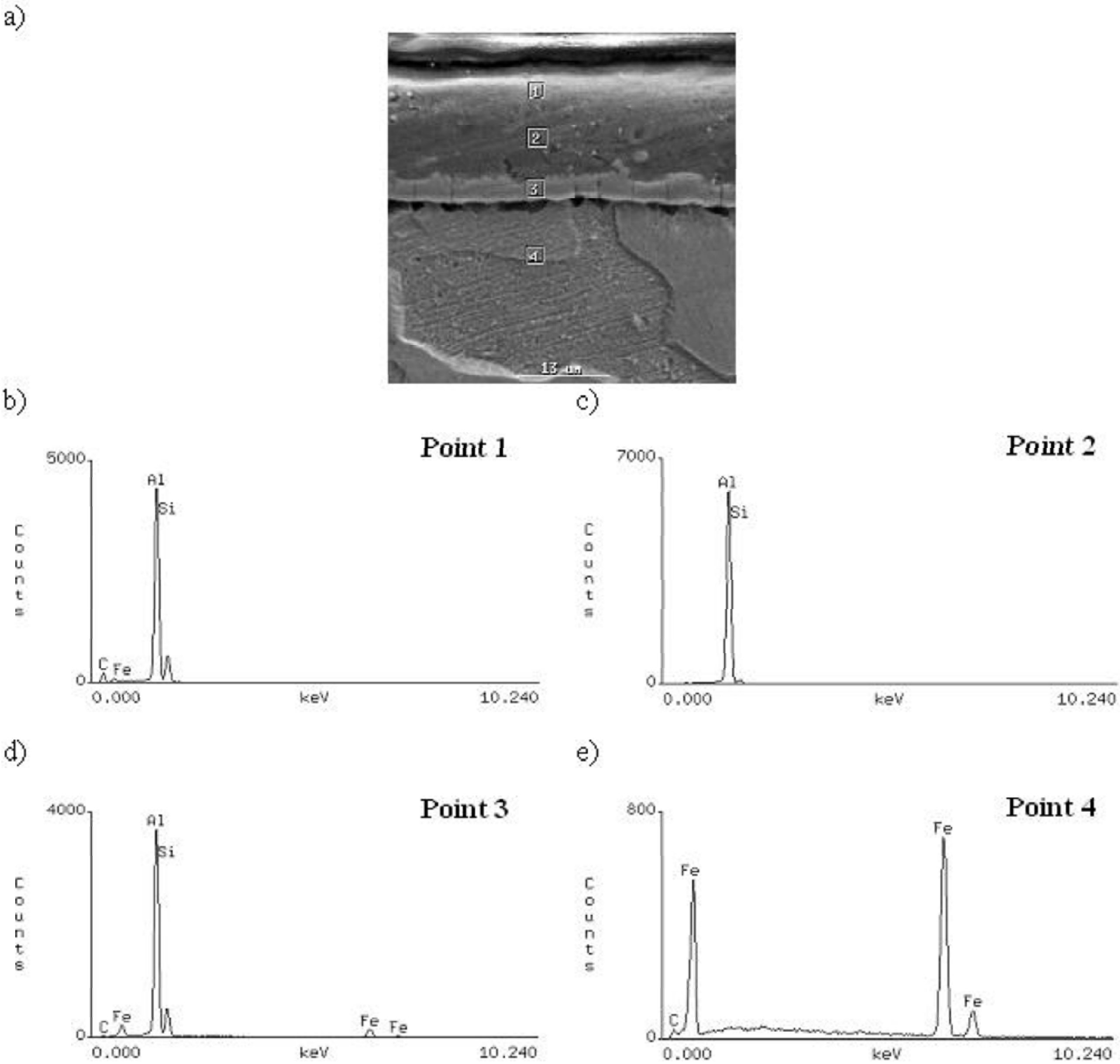


Fig. 2. Surface of Al-10wt%Si coating in initial state

Fig 3. shows the results of the observation of the cross-section of coating structure and local and linear microanalysis of the chemical composition using SEM with EDS.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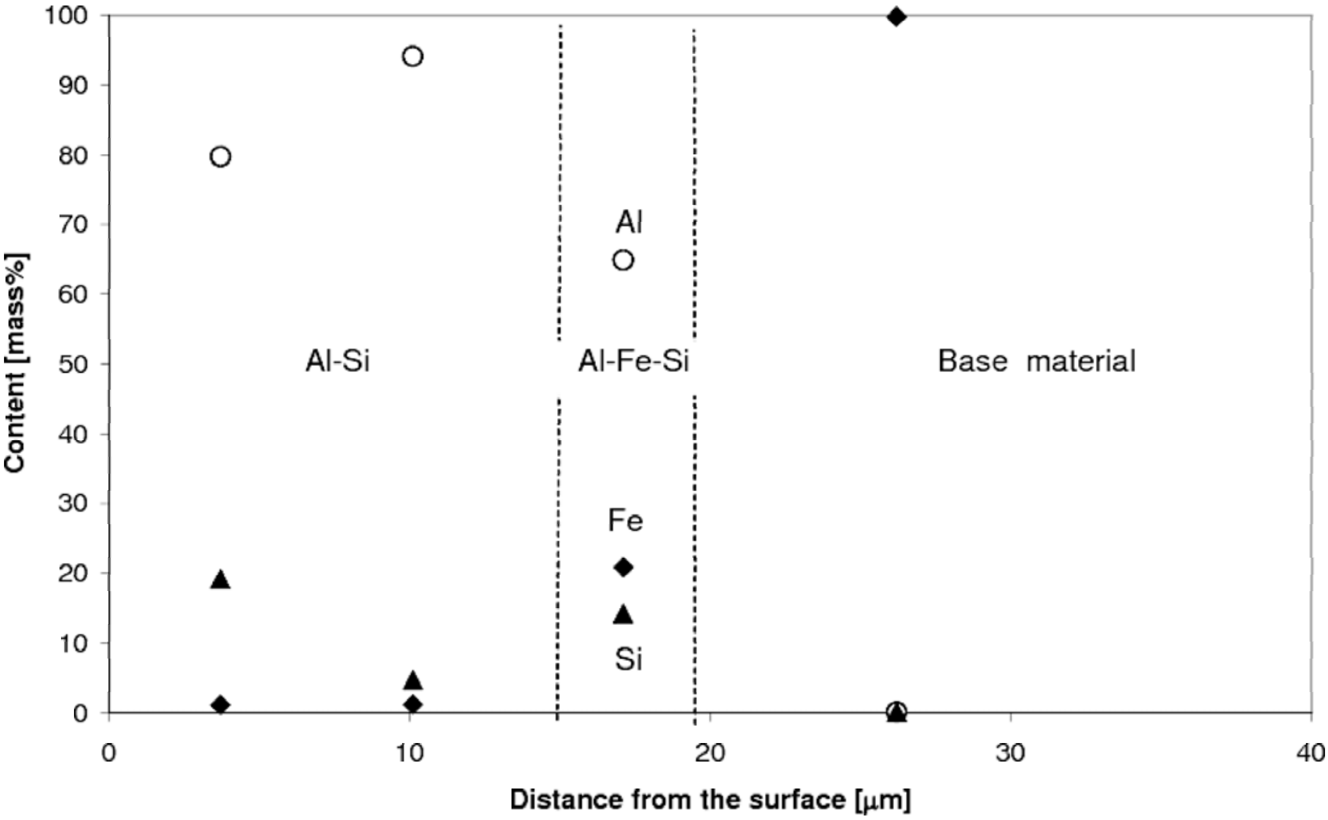

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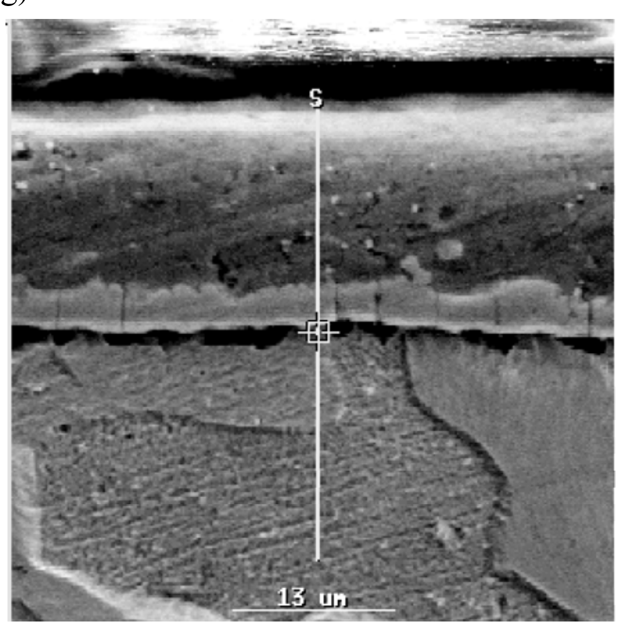

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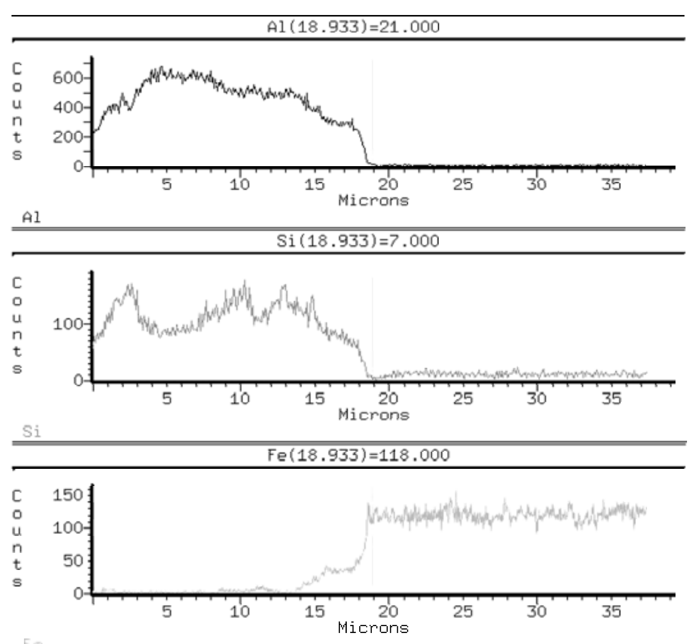


Fig. 3. Coating cross-section and local and linear microanalyses of chemical composition – SEM a) coating cross-section with measuring points of chemical composition microanalysis, b) EDS spectrogram of Al, Si, Fe concentrations in point 1, c) EDS spectrogram of Al, Si, Fe concentrations in point 2, d) EDS spectrogram of Al, Si, Fe concentrations in point 3, e) EDS spectrogram of Al, Si, Fe concentrations in point 4, f) change of Al, Fe and Si concentrations in thickness direction of sample corresponding to Fig 3a, g) coating cross-section with the line along which linear analysis was made, h) linear analysis results – changes in Al, Si, Fe concentrations

The coating in the initial state has two layers (Fig. 3a). It consists of the main two-component Al-Si coating with the prevailing concentration of aluminum (Fig. 3b, c) and the three-component Al-Fe-Si (Fig 3d) inter-metallic layer connecting the coating with the base and having the thickness of ca 20% of the coating thickness. The linear analysis (Fig. 3g) shows a characteristic sudden change of the elements concentration in this area (Fig. 3h). Al concentration in the coating amounts to 80-95% and is strictly related to Si concentration. Local silicon emissions, which increase its concentration in the tested area, lower Al concentration. In the inter-metallic layer, Al concentration is lowered below 70% until it nearly disappears from the base material (Fig. 3f, h). Fe is not present in the coating, whereas its concentration in the inter-metallic layer is ca 20%, and, obviously, amounts to ca 100% in the base material (Fig. 3e).

3.2. Tests of Al-Si coating thickness

Fig. 4 shows the results of the measurements of Al-Si coating thickness on side A and B in initial state and after heating. A diversified coating thickness in initial state is characteristic. Medium thickness of the coating in initial state is 18,6 μm on side A and 22,4 μm on side B. Coating thickness after heat treatment depends on soaking time and temperature. In 250-400°C, throughout the whole time period, medium coating thickness changes very little (max. 19,6 μm on side A and 23,7 μm on side B, as compared to the initial material). After heating in temperatures 450-500°C the growth of medium coating thickness depends on exposure time. For short soaking times (30min and 180min for 450°C and 30min for 500°C) the growth of the coating thickness is small, whereas longer soaking times (1440min for 450°C and 180min and 1 for 500°C) cause the growth of the thickness (max 28 μm). After heating in temperatures 550-700°C throughout the whole time range medium coating thickness grows in comparison to the thickness in the initial state maximum to 26,7 μm on side A and to 31,1 μm on side B.

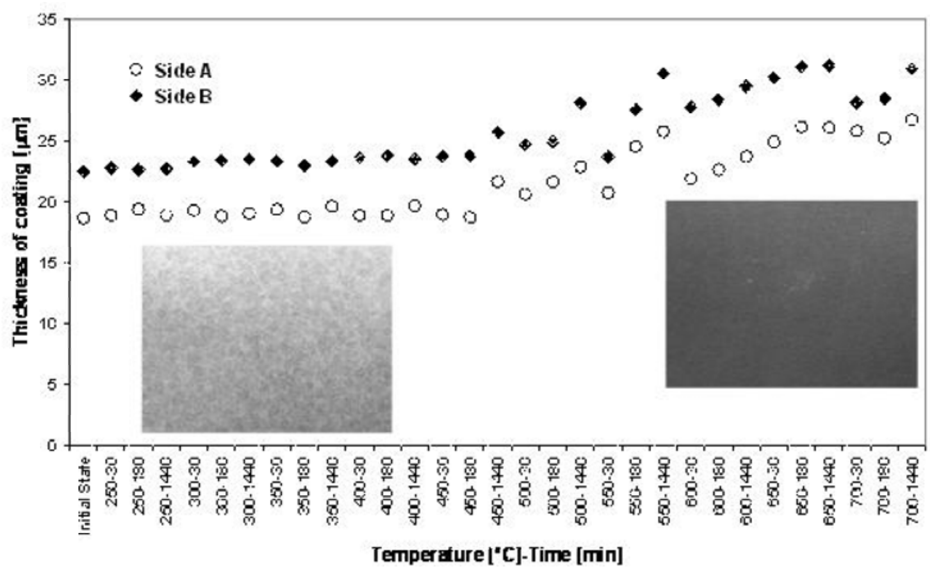


Fig. 4. Medium thickness of the coating depending on heat treatment parameters. Side A and B

The increase of the coating thickness depends on the raise of soaking time and temperature, thus on the changes in the coating appearance. The most significant growth in the coating thickness appears when the temperature ranges from 600-700°C. At the same time, in this time-temperature range, the coating surface becomes dark, dull and porous (Fig.4). The percentage change in the coating thickness on sides A and B is more or less identical.

3.3. Tests of Al-Fe-Si inter-metallic layer thickness

Fig. 5 shows the results of the measurements of the medium thickness of the Al-Fe-Si inter-metallic layer in initial state and after heat treatment.

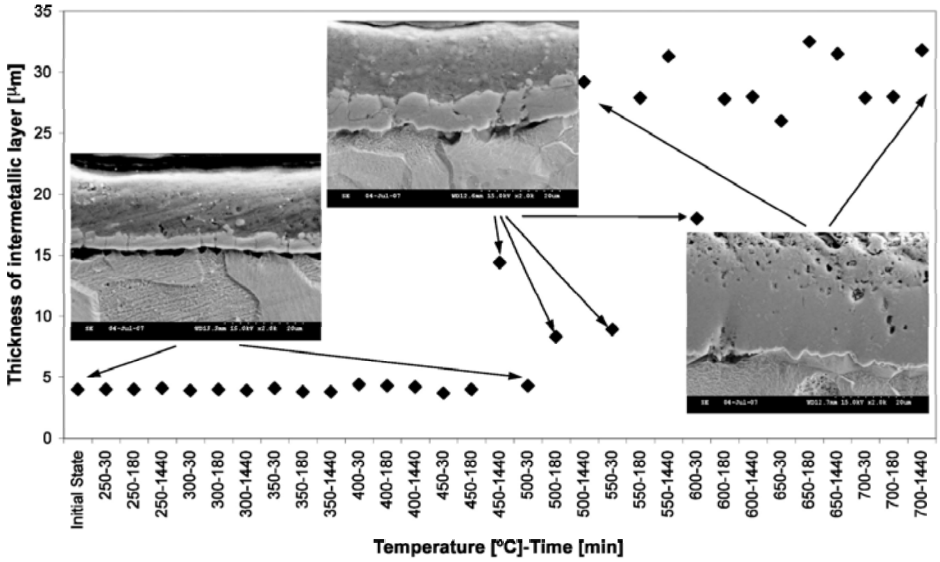
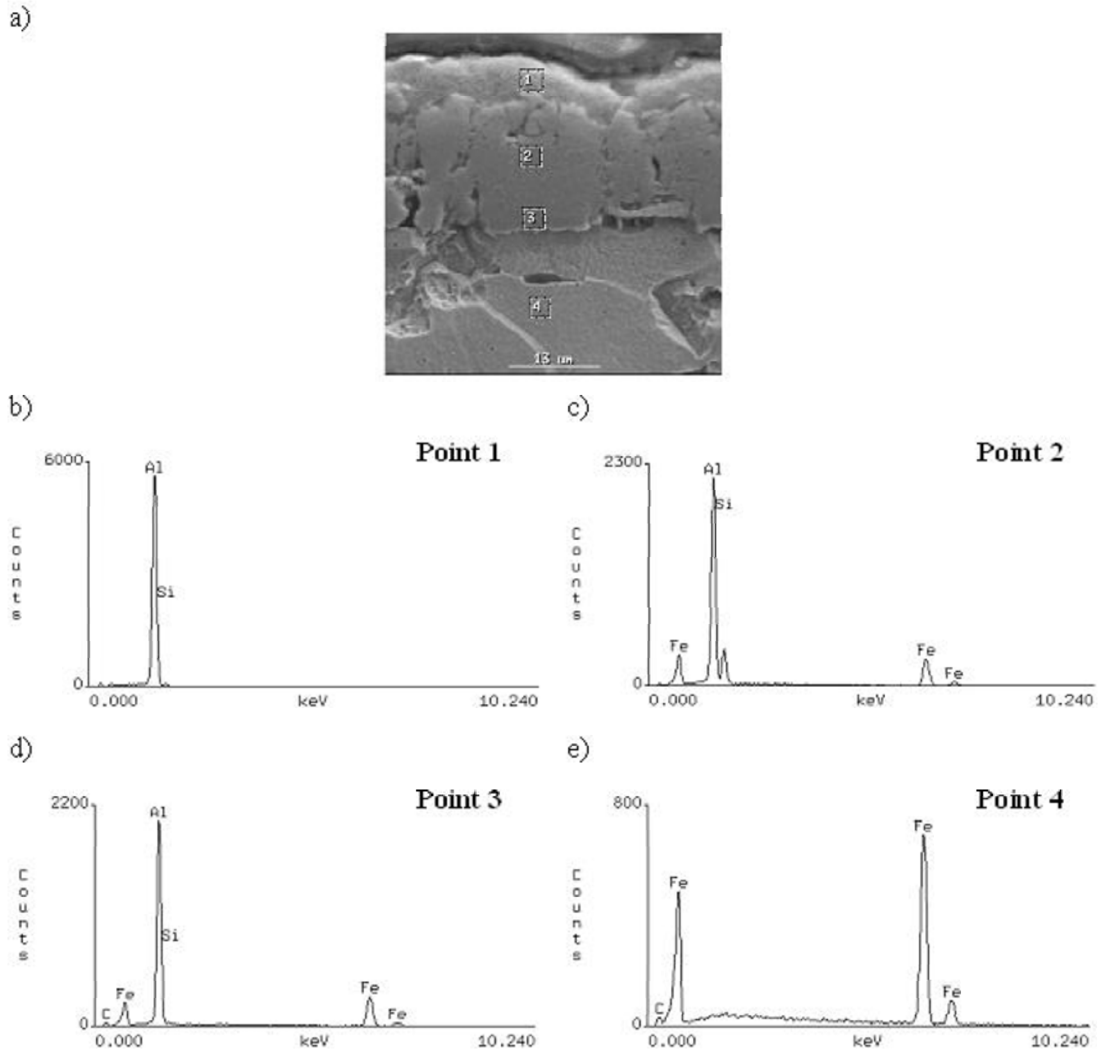


Fig. 5. Medium thickness of the inter-metallic layer Al-Fe-Si depending on heat treatment parameters

Temperature and time have a significant influence on the change of Al-Fe-Si inter-metallic layer thickness. The thickness of the inter-metallic layer in initial state is ca 15-20% of the coating thickness. After heating in temperatures 250-400°C the thickness of the inter-metallic layer is near to the thickness of the alloy layer in the material in the initial state, i.e. it makes ca 15-20% of the total coating thickness. After heating in temperatures 450°C, together with the growth of the exposure, the thickness of the inter-metallic layer grows. After 1440min its thickness amounts to ca 60% of the coating thickness. After heating in temperature 500°C, after 30min, the thickness of the inter-metallic layer amounts to ca 15% of the coating thickness, after 180min – ca 30% and after 1440min the coating changed into a three-component Al-Fe-Si one-layer throughout its total thickness. In 550°C, after 30min the alloy layer amounts to ca 30% of the coating thickness. For other exposure times in these temperatures and other variations, the coating changed into Al-Fe-Si one-layer. Changes in the thickness of the inter-metallic layer are caused by the diffusion of iron into the coating. The higher soaking time and temperature, the quicker the diffusion occurs. It is proved by the local and linear microanalysis of the chemical composition of the sample which was heat-treated in 600oC during 30min. (Fig. 6).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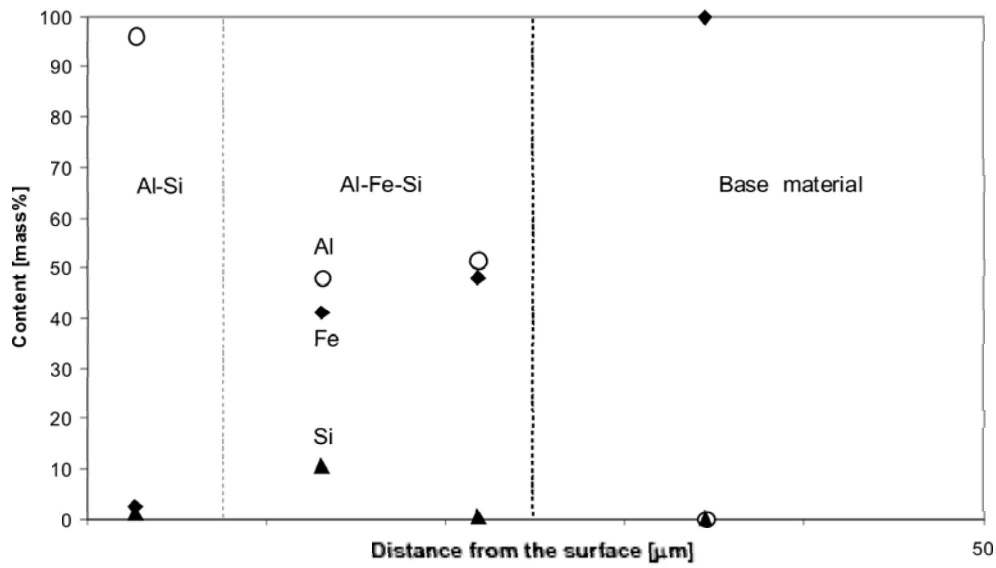

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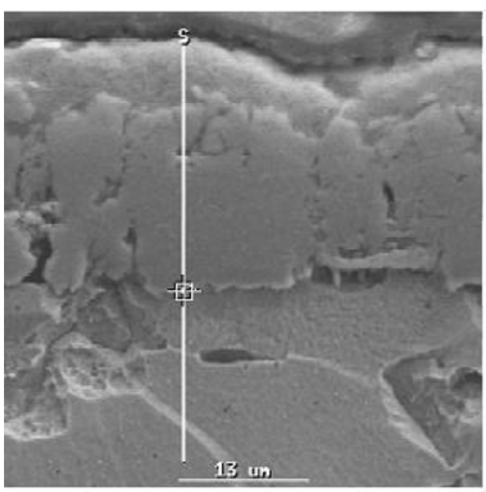

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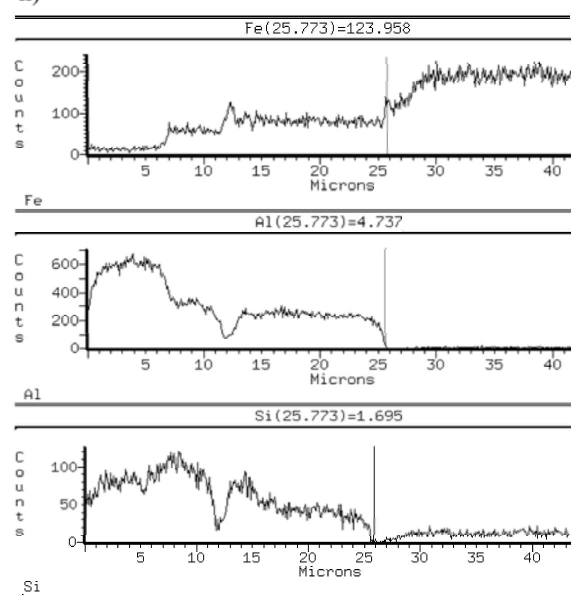

Fig. 6. Coating cross-section and local and linear microanalyses of chemical composition after heating in temperature 600 °C during 30min – SEM

a) coating cross-section with measuring points of chemical composition microanalysis, b) EDS spectrogram of Al, Si, Fe concentrations in point 1, c) EDS spectrogram of Al, Si, Fe concentrations in point 2, d) EDS spectrogram of Al, Si, Fe concentrations in point 3, e) EDS spectrogram of Al, Si, Fe concentrations in point 4, f) change of Al, Fe and Si concentrations in thickness direction of sample corresponding to Fig 6a, g) coating cross-section with the line along which linear analysis was made, h) linear analysis results – changes in Al, Si, Fe concentrations

The coating after heating in temperature 600°C and 30min still has two layers (Fig. 6a). It consists of the main two-component Al-Si coating with the prevailing concentration of Al (Fig. 6b, c) and the three-component Al-Fe-Si inter-metallic layer (Fig 6d). However, in this case, the thickness of the inter-metallic layer amounts to ca 70-80% of the coating thickness. Al concentration in the coating amounts to over ca 90% and is strictly related to Si concentration. Local silicon emissions, which increase its concentration in the tested area, lower Al concentration. Fe is not present in the coating. In the inter-metallic layer, Al concentration drops down from ca 90% to ca 50% and Fe concentration grows to ca 40-50% (Fig. 6f, h). The linear analysis (Fig. 6g) shows a characteristic sudden change of the elements concentration in this area. Fe concentration in the base material amounts to ca 10% (Fig. 6e).

3.4. Al-Si coating surface roughness testing

Fig. 7 shows the results of the measurements of the geometrical structure of the coating surface, on sides A and B, characterized by the roughness parameters R_a and R_z.

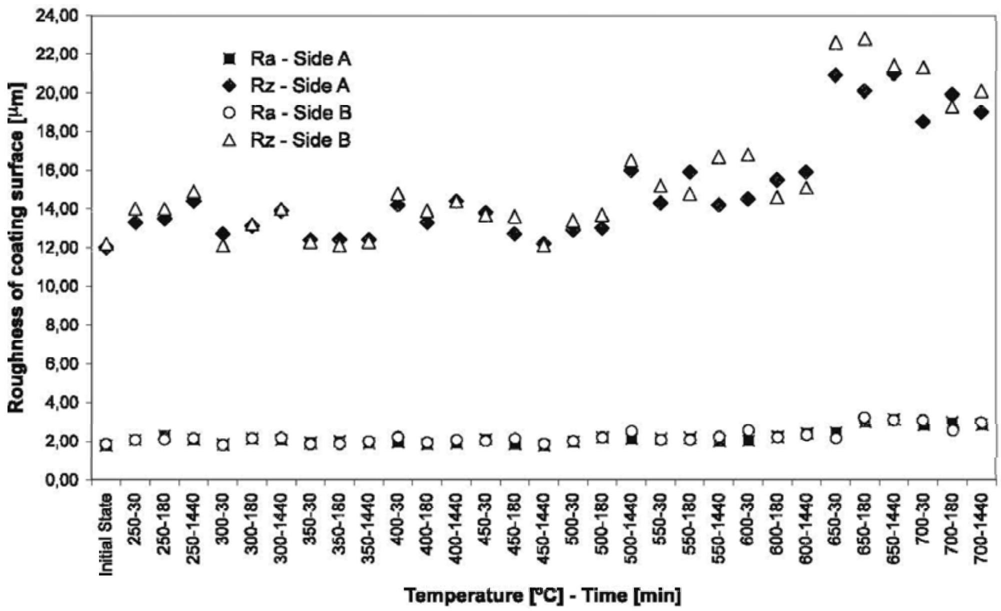

Fig. 7. Medium R_a and R_z values depending on heat treatment parameters. Side A and B

Roughness of the coating surface is, similarly to the coating thickness, closely correlated to temperature and exposure time. In the initial state roughness of the coating surface amounts to R_a = 1,8 m and R_z = 12 m. In 250-600°C, nearly throughout the whole time range, R_a parameter varies between 1,8 and 2,2 µm, whereas R_z parameter varies between 12 and 14 µm. Samples on the surfaces where darker areas or dulling appear are an exception. It was for samples after heat treatment in 550°C during 1440min and 600°C during 180min and 1440min. Then, R_a amounts to ca 2,4 µm and R_z ca 16 µm. In 650 – 700°C, roughness grows to R_a = 2,5 - 3,3 µm and R_z = 19 – 23 µm values. It is caused by the change in the coating appearance from smooth, bright and glossy to rough, dark, porous and dull. The percentage change in the roughness of coating surface on sides A and B is more or less identical. Fig. 8 shows some of the results of macro- and microscopic observations of Al-Si coating surface after heat treatment.

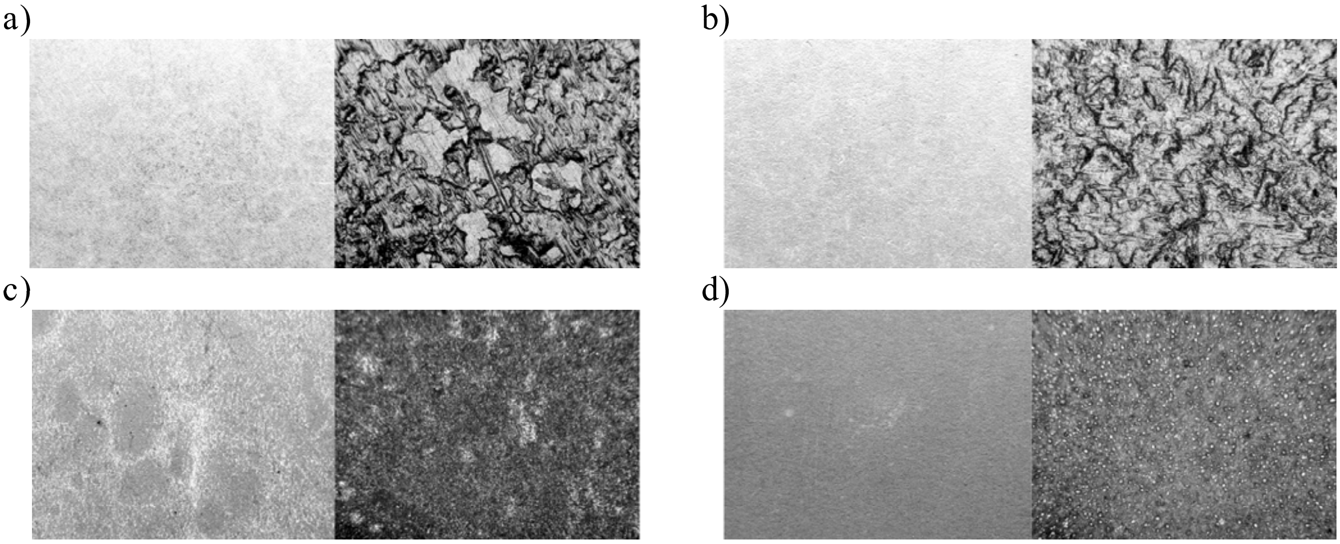

Fig. 8. Macro- and microscopic observation of the surface of the Al-Si coating after heat treatment a) 300°C – 1440min, b) 450°C – 1440min, c) 500°C – 180min, d) 700°C – 30min

The appearance of the coating surface depends on soaking temperature and time. In the range of 250-450°C the coating surface is bright and glossy and has no dark areas (Fig. 8a, b). In temperatures 500-600°C the coating surface becomes dull and irregular dark areas appear (Fig. 8c). In temperatures 650-700°C throughout the whole time range, whole coating surface is dark and dull (Fig. 8d).

3.5. Changes on the coating surface after heat treatment

Fig. 9. shows the results of the first changes and total dulling and darkening of the coating surface

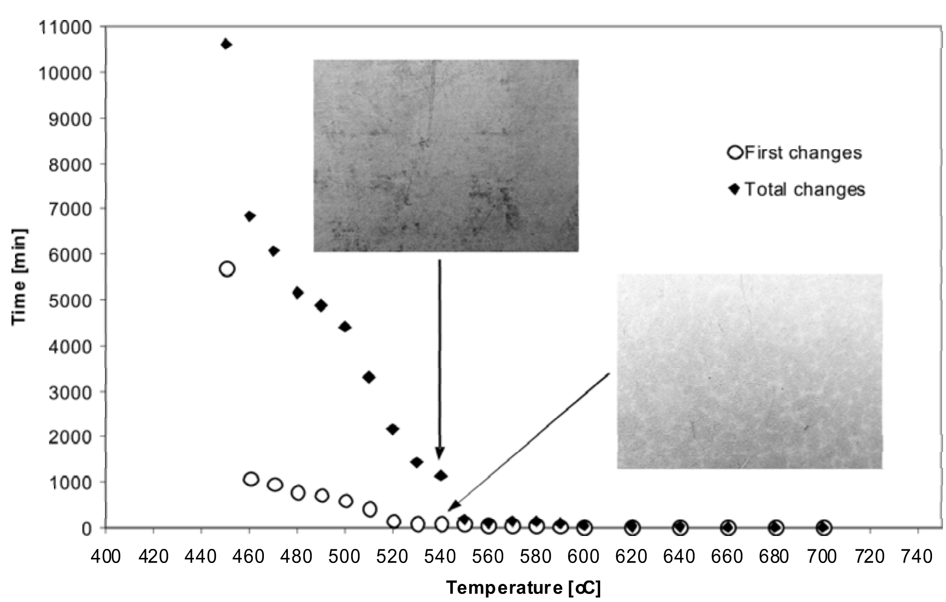

Fig. 9. First and total changes of the coating surface depend on temperature and time of heat treatment

In 250-450°C there is no changes on the coating surface. In 450°C first changes appear after 5700min. and the total changes after 10620min. In 460-510°C first changes appear after 1080-420min and total changes after 6840-3300min. In 520-700°C time needed for the first changes to appear drops down from 150min to 1min and for the total changes to appear drops down from 2160min to 6min. It is very probable that in temperature over 700°C time needed for the first changes to appear will be shorter than 1min and for the total changes to appear will be shorter than 5min.

4. Conclusions

The paper presents the results of the influence of heat treatment on the changes in Al-Si coating thickness, Al-Fe-Si inter-metallic layer thickness and roughness on steel plates and the speed of changes in the coating appearance such as dark spots and dulling. Tests were conducted in 100-700°C, during 1-10620min. Basing on the analysis of the test results the following conclusions can be drawn:

1. Tests have proved that time and temperature of heat treatment has a significant influence on the surface condition, structure and chemical composition of Al-Si coating.
2. In the initial state Al-Si coating has two layers – the first one is a two-component Al-Si basic coating and the other one is a three-component Al-Fe-Si diffusion intermediate layer, which appears between the coating and the steel base.
3. Under the influence of temperature and time Al-Si coating changes from the two-component into a three-component Al-Fe-Si layer. This effect appears during heat treatment above 500°C.
4. Coating thickness and roughness in the temperature range of 600-700°C increases with the growth of soaking temperature and time.
5. Coating thickness in the initial state has a significant influence on the coating behavior during heat treatment. The smaller the thickness, the quicker changes on the coating surface are observed.
6. First changes on the coating surface during soaking, which is seen as dark areas, or dulling of the coating appear above 450°C. With the raise of temperature, the time necessary for the first changes of the coating surface as well as its total dulling and darkening to appear becomes shorter.

Acknowledgements

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