

## EDS-INVESTIGATION OF Fe-Al INTERMETALLIC COMPOUNDS\*

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Steels are often coated by other metallic materials in order to obtain better surface properties. In several cases this layer is made by hot dip methods. Due to the concentration gradient between the base metal and the layer, phase transformations occur in the diffusive interface layer. These phase transformations, of course, influence the kinetics of the coating process and the properties of the coating itself. The aim of our work was to clarify the characteristic features of the aluminium coating process, which are in connection with the phase transformations in the diffusive interface layer. The development of the compound layer, its microstructure and phases were investigated in order to determine the effect of the carbon content of the base metal.

### INTRODUCTION

During hot dip coating of steel intermetallic compounds develop due to diffusion processes between the solid Fe and the liquid Al. In the equilibrium diagram of Al-Fe six different equilibrium intermetallic compound can be found. They have a certain concentration-interval, which is especially wide in case of the FeAl compound (this compound is practically a solid solution). On the other hand, it is impossible to isothermally create Fe<sub>3</sub>Al compound over the melting point of Al, therefore in our case this phase can be created only during cooling. It should be mentioned that, due to the rapid cooling, metastable phases can be observed also, e.g. FeAl<sub>6</sub> and Fe<sub>2</sub>Al<sub>9</sub>.<sup>1</sup> The Fe<sub>2</sub>Al<sub>7</sub> compound, which is very close to the FeAl<sub>3</sub> compound, is signed as an individual phase by certain authors,<sup>2,3</sup> and this phase was identified in this work too, by EDS-analysis.

The main question in this work was the role of carbon content in the kinetics of the development of Fe-Al compounds. The carbon-solubility of Al is low, only 0.22-0.71 at % between 800-1200 °C. It is interesting that in the Fe-C-Al equilibrium system a biner carbide, the Al<sub>4</sub>C<sub>3</sub>, and a tertiary carbide, the C<sub>x</sub>Fe<sub>3</sub>Al<sup>4</sup> ( $\chi$ -phase) appear, which can act in the following reaction at 780 °C:



During hot dip coating, the diffusion process is carried out only at lower temperature (close to the melting point of Al) and for a short dipping periods. For describing the diffusion process, several authors apply the well-known formula:

$$y = K \cdot t^n \quad [2]$$

where  $y$  is the thickness of the coating developed in  $t$  time, and  $K$  and  $n$  are constants. The value of  $n$  is generally kept as 1/2 or very close. At higher temperature, however, the value of  $n$  can deviate

from this constant.<sup>5</sup> Therefore the experimental test were performed in such a way that the variation of  $n$  can be followed.

## EXPERIMENTAL

Two different, non-alloyed steel wires were applied for the tests. One of them (steel A) had a lower carbon content (0.11 wt%), while the other (steel B) had a higher carbon content (0.77 wt%). The diameter of the specimens were 1 mm, and the length of them was 120 mm.

The melting point of Al is lower than the initial temperature of  $\alpha \rightarrow \gamma$  transformation of steel, therefore it was possible to investigate the coating process in the ferritic, austenitic and two-phase state of the steel. By altering the temperature and duration of dipping, the velocity of the development of the coating was analysed. Table 1 shows the temperature and time values applied during the tests.

Table 1. Parameters of hot dip coatings

Duration of dipping									
2s	5s	10s	30s	60s	120 s	300 s			
Temperature of the Al-liquid during dipping of 0,11 % C-steel									
680 °C	700 °C	730 °C	760 °C	790 °C	820 °C	850 °C	880 °C	910 °C	940 °C
Temperature of the Al-liquid during dipping of 0,77 % C-steel									
680 °C	790 °C				910 °C				

The coating could be handled as isothermic process, since on the phase boundary, where the reactions took place, it took only a few ms to reach the temperature of the liquid. Before hot dipping the samples were descaled, degreased and electrically polished. The Al bath contained about 1 kg very pure (99.99%) aluminium. After cooling the wires were cut and polished, the thickness of the coatings were measured, and the micro-hardness of the layers were determined. After that scanning electron microscope investigations, EDS/WDS/XRD analyses were performed.

## RESULTS

Figure 1 shows the thickness of the hot dipped coating as a function of time. Continuous lines refer to the low carbon steel, dashed lines refer to the higher carbon steel. These lines are parallel straight lines until the value of  $n$  in eqn. (2) is more-or-less uniform. As it can be seen in figure 1 the development of coating on the surface of higher carbon steel is continuous, but much slower than that of the lower carbon steel. It follows from the foregoings that the amount and C-content of austenite restrain the development of the compound layer, therefore the role of ferrite and austenite in the coating process is just the opposite. The ferrite, which practically does not contain carbon, reacts with the aluminium very rapidly, while the austenite reacts much slower, although the diffusivity of Al in austenite is twenty times higher than that in the ferrite.

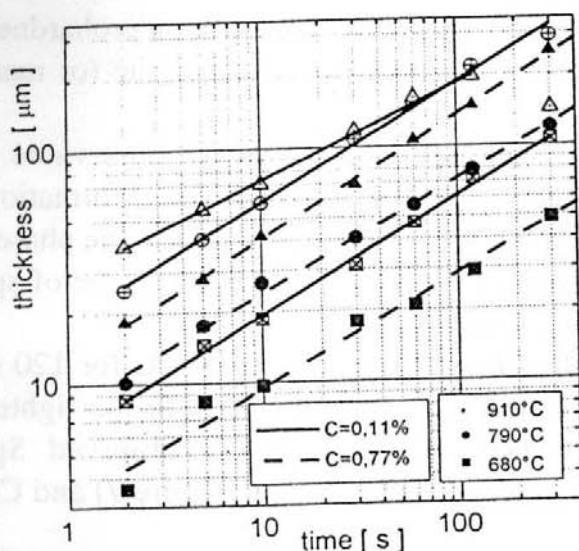


Fig. 1. The thickness of coating as a function of dipping time.

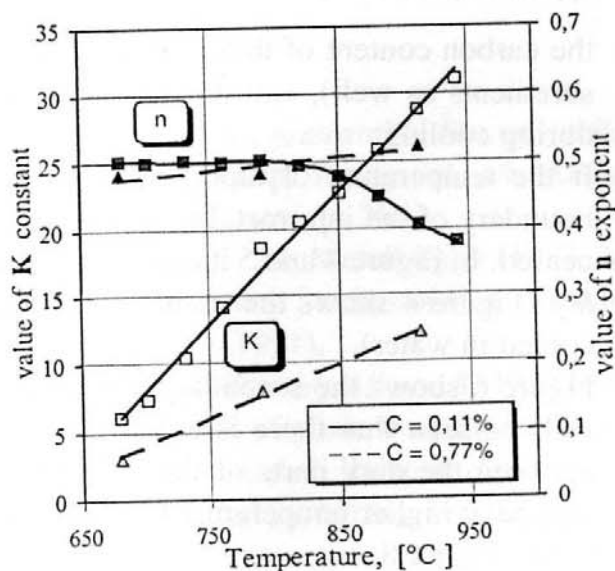


Fig. 2. The values of  $K$  and  $n$  as a function of Al-bath temperature.

The measurable data of the compound-formation as the function of temperature can be illustrated by the values of  $K$  and  $n$  of equation (2). This can be seen in Figure 2. It can be observed that the value of  $n$  for steel A decreases as the temperature reaches 850 °C, while that for steel B is practically constant. It should be mentioned that the temperature of isothermal  $\alpha \rightarrow \gamma$  transformation for low carbon steel ( $C = 0.11\%$ ) is about 860 °C. It can also be observed in Figure 2 that the coating process of steel containing higher amount of carbon ( $C = 0.77\%$ ) is much slower, the slope of the  $K$  parameter is smaller.

Metallographic investigations showed that the compound layer formed at temperature below  $A_1$  was zigzagged. On the Al-side the  $\text{FeAl}_3$  phase could be identified. Close to the Fe-side the  $\text{Fe}_2\text{Al}_5$  phase could be measured, and this phase formed the biggest part of the compound layer. By increasing the temperature the zigzagging of the layer decreased.

In Figure 3 the metallographic image of the compound-base metal interface of steel A formed at 790 °C during 120 s and cooled at air atmosphere can be seen. Pearlitic regions of steel were transformed into austenite, while only a small part of ferrite became austenite, the remaining part recrystallized. Along the compound layer the carbon content of the darker area increased (in some cases

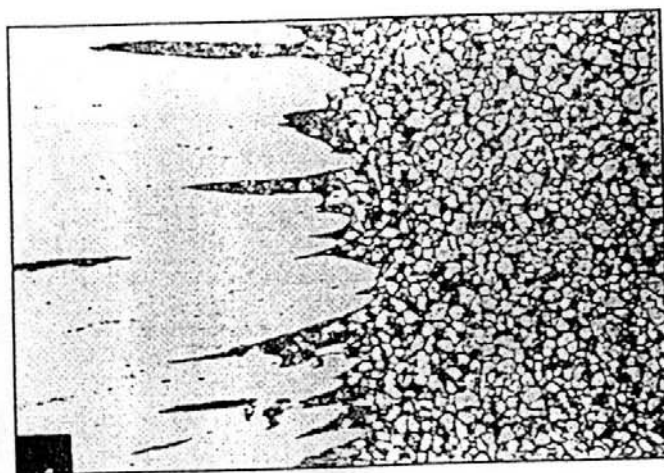


Fig. 3.  $G = 500 \times$ .



the carbon content of these regions was over 0.7 wt%, which was proven by microhardness measurements as well), and the austenite formed there was transformed into pearlite (or martensite) during cooling.

If the temperature of dipping exceeded the A3 temperature of steel it was observable that the boundary of the intermetallic phase on the Fe-side is uniform, and the zigzagged formation disappeared. In Figure 4 and 5 it can be seen that there was a high carbon enrichment at the phase boundary (Figure 4 shows the tissue of specimen cooled at air, Figure 5 shows the tissue of specimen cooled in water).

Figure 6 shows the secondary electron image of steel B after dipping at 680 °C for 120 s. It can well be seen that there is a boundary between the FeAl<sub>3</sub> and the Fe<sub>2</sub>Al<sub>5</sub>. In the lighter bands between the dark parts of the compound layer a carbon enrichment can be observed. Specimen dipped at higher temperature (910 °C, 120 s) showed also the FeAl edge (Figure 7) and C-enrichment (Figure 8).

None of the measured composition correspond to any of the equilibrium phases, which means that the concentration changed heavily in the measured regions. These regions, however, can be equilibrium phases, e.g. the identified Fe<sub>4</sub>Al<sub>3</sub> and Fe<sub>5</sub>Al<sub>3</sub> phases.



Fig. 4. G = 500 x.

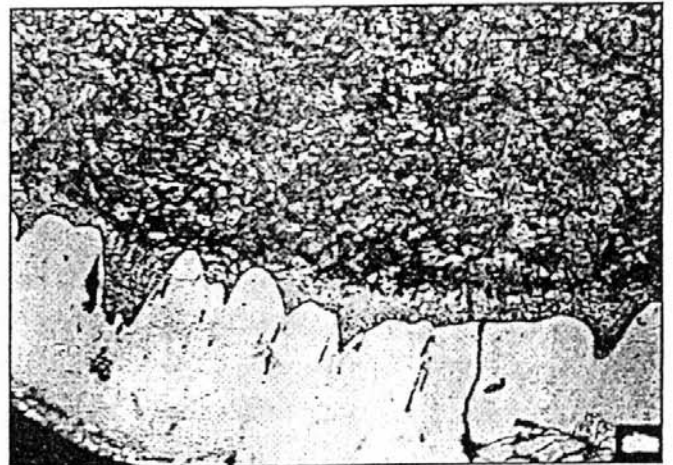


Fig. 5. G = 500 x.

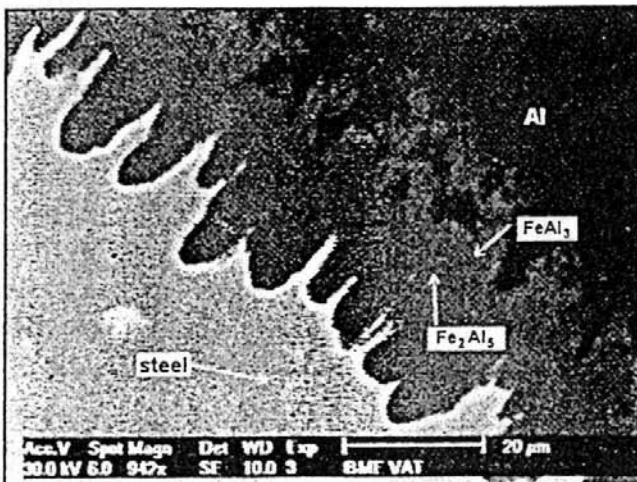


Fig. 6.

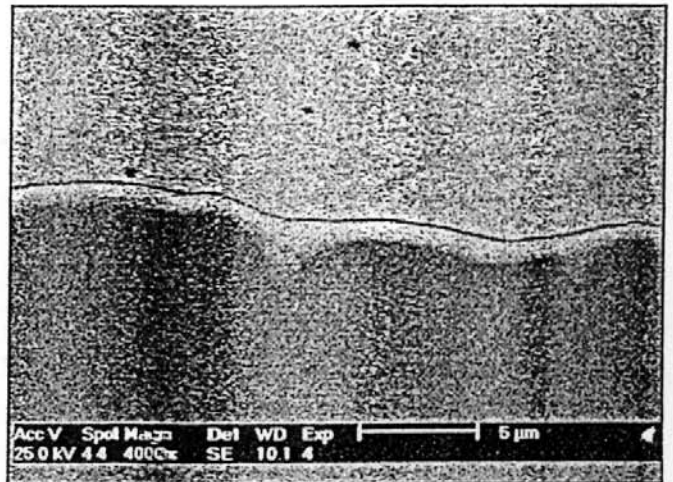


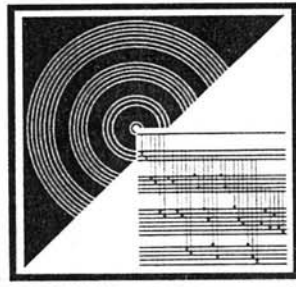
Fig. 7.

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