OBSERVATION OF SELECTIVE OXIDATION OF STEEL SHEETS WITH ATOMIC FORCE MICROSCOPY

Madeira, L.[1*], Lins, V.C.F.[1], Vilela, J.M.C.[2], Andrade, M. S.[2], Guimarães, J.P.[3], Alvarenga, E. A.[3]

[1]Department of Chemical Engineer – Federal University of Minas Gerais, Brasil – laureannymadeira@hotmail.com, vlins@deq.ufmg.br
[2]Department of Metallurgy – Fundação Centro Tecnológico de Minas Gerais, Brasil - jose.mario.vilela@cetec.br, margareth.spangler@cetec.br
[3]Usiminas – Brasil – mailto:juliana.guimaraes@usiminas.com, evandro.alvarenga@usiminas.com.br

Hot galvanized steels have been extensively used in the automobile industry due to their excellent corrosion resistance, good weldability and good formability. However, the presence of scratches, iron powder, dirt, oil flecks, high roughness, and oxide formation affect the reactions at the coating/substrate interface inducing several defects in the final product [1].

One of the main problems in the galvanizing process is the unsatisfying wettability of the steel surface during Zn coating due to the presence of oxides. After cold rolling the steel sheets undergo an annealing to accomplish the recrystallization. Alloying elements such as Mn, Al, Si, Cr, and Ti present a high affinity to oxygen, forming oxides during this annealing cycle. The oxides grow in the form of islands rather than in a continuous layer and are not completely removed in the reduction-annealing step before galvanizing [2]. Nitride agglomerates such as BN can also be formed during the annealing step. An important aspect of these agglomerates is that they can grow selectively [3] depending on the grain orientation of the steel. The most important parameter to control the oxidation is the dew point of the reduction-annealing atmosphere. Changing the dew point allows the amount of water in the annealing atmosphere to be controlled. As well as the annealing conditions the steel composition plays an important role in the oxidation [4].

In this work the morphology and the distribution of oxide and/or nitride agglomerates grown during the annealing step on the surface of a steel sheet were analysed with atomic force microscopy (AFM). Three different dew points were investigated.

The analyses were performed on commercial interstitial free (IF) steel. Samples with 5cm diameter were taken from a cold rolled steel sheet, annealed at 900^oC for 60s in $5\%H_2$-N2 atmosphere with dew points of -60, -30 and 0^oC. They were observed with AFM operating in contact mode using silicon nitride commercial probes.

Figure 1 shows AFM images of the steel surface after annealing with dew points of -60^oC (a), -30^oC (b) and 0^oC (c). Globular agglomerates are observed in the three images. Selective oxidation can be observed in images 1a and 1b. As the water content in the annealing atmosphere increases with the dew point, the density and size of the agglomerates also increase (1c) suggesting that they are oxide agglomerates. Preferential growth sites were not observed at dew point of 0^oC, with agglomerates evenly distributed over the surface. In figure 2a the sample annealed with dew point of 0^oC is shown with a larger magnification. Figure 2b shows the topographical profile obtained of the line drawn in 2a. Figure 3 presents the size distribution of the agglomerates observed on the surface of the sample annealed at 0^oC. Two agglomerate size distributions can be observed. The larger agglomerates with mean diameter of a 680nm are not globular, but present geometrical facets, being probably boron nitride. The smaller agglomerates with mean diameter of 200nm are globular in shape. The two agglomerate size distributions suggest that different oxides or nitrides are present after annealing with dew point of 0^oC. From these results it can be concluded that AFM can be applied to the study of selective oxidation of steel sheets.

After these morphological analyses, X-Ray Photoelectron Spectrometry (XPS) and Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS) will be used to characterize the nucleus of the oxide/nitride agglomerates.

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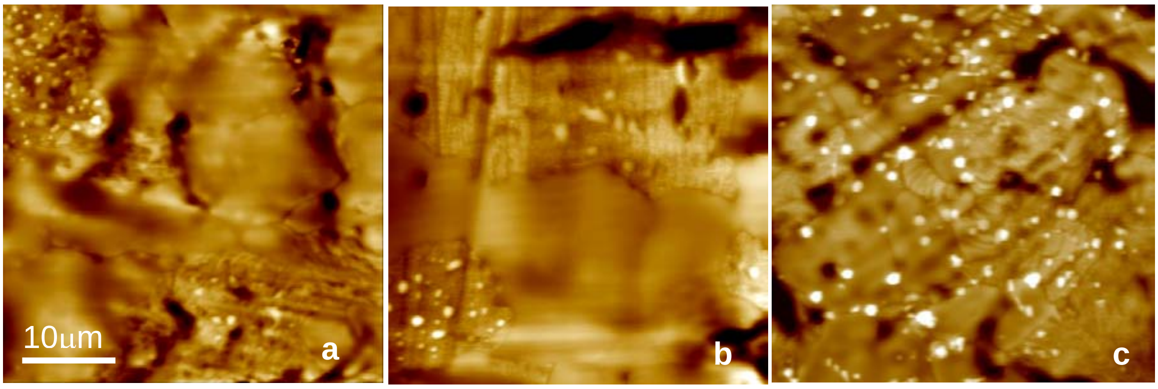

Figure 1- AFM images of steel surface after annealing with dew points of -60°C (a), -30°C (b) and 0°C (c). Selective oxidation is observed in (a) and (b).

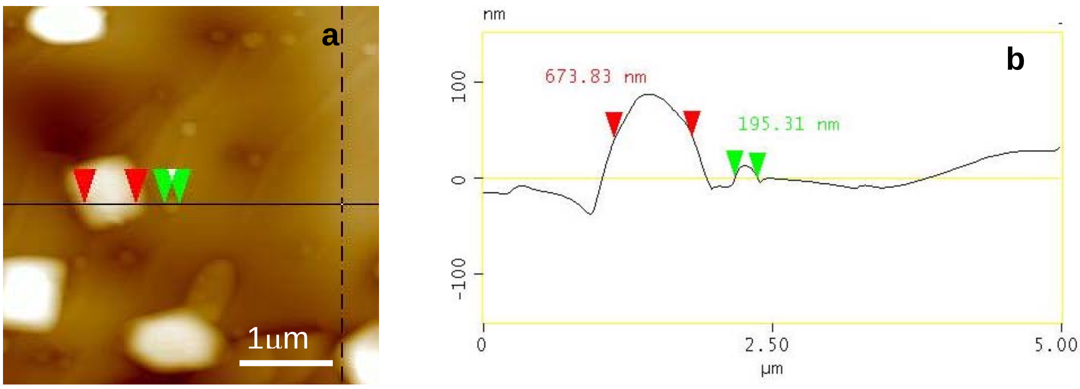

Figure 2 – AFM image of steel surface after annealing with dew point of 0°C (a) and topographical profile (b).

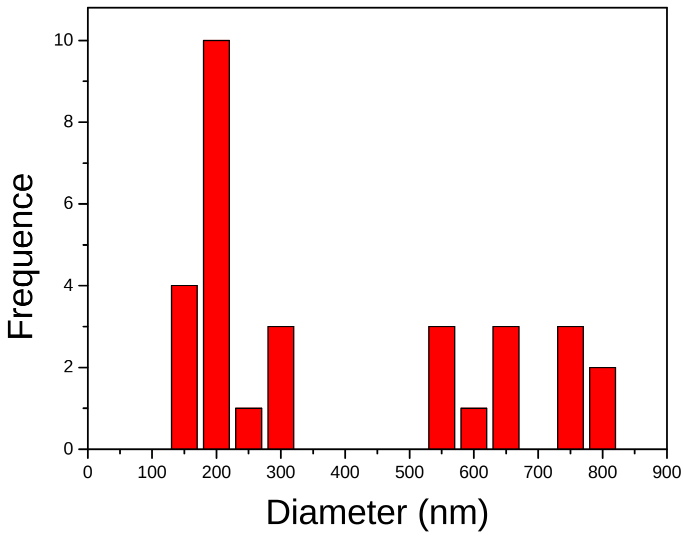

Figure 3 – Size distribution of agglomerates after annealing with dew point of 0°C.