

Aluminum-silicon co-deposition by FB-CVD on austenitic stainless steel AISI 316

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Abstract. Aluminum-silicon coatings were deposited on stainless steel AISI 316 in the temperature range of 540 to 560°C by CVD-FBR. It was used a fluidized bed with 2.5% silicon and 7.5% aluminum powder and 90% inert (alumina). This bed was fluidized with Ar and as an activator a mixture of HCl/H₂ in ratios of 1/10 to 1/16. Furthermore, the deposition time of the coatings was varied between 45 minutes to 1.5 hours, with a 50% active gas, neutral gases 50%. Thermodynamic simulation was conducted with the Thermocalc software to get the possible compositions and amount of material deposited for the chosen conditions. The coatings presented the follow compounds FeAl₂Si, FeAl₂ and Fe₂Al₅. Aluminum-silicon coatings were heat treated to improve its mechanical properties and its behavior against oxidation for the inter diffusion of the alloying elements. The heat treatment causes the aluminum diffuse into the substrate and the iron diffuse into coating surface. This leads to the transformation of the above compounds in FeAl, Al₂FeSi, Cr₃Si, AlFeNi and AlCrFe.

1. Introduction

The Chemical Vapour Deposition (CVD) involves the chemical reaction of a precursor gas mixture inside a vacuum chamber for depositing a thin layer on the substrate. The reaction byproducts are evacuated outwards [1, 2]. This technique uses the chemical reactions of the precursor gases which are activated by heating, plasma, electromagnetic radiation, etc. The chemical deposition reactions can be classified into homogeneous and heterogeneous. Chemical vapor deposition is a very versatile process which allows several applications. Some applications are for protecting materials against wear, corrosion and oxidation at high temperature. It also allows the deposition of amorphous elements, mono crystalline, polycrystalline thin layers, dielectrics, for microelectronics and optoelectronics in energy conversion devices. Also can be manufactured ceramic fibers and composites ceramic matrix, as well as nanostructured materials for the electronics industry and biotechnology [3-5].

The chemical vapor deposition fluidized bed is a variant of the technique of chemical vapor deposition that combines the advantages of thermal activation by heating and the fluidized bed. The CVD-FBR takes advantage of the fluidized beds, as are the high transfer heat and mass between the gas, the bed and samples immersed in the reactor. The CVD-FBR allows a more uniform temperature and good mixing of reactive gases with the fluidized particles. This produce a fast reaction of the activated species in all the bed, because there fluidization in excellent contact between the solid



particles and the gaseous fluidizing medium. This helps reduce the temperature and operating time compared to other processes such as pack cementation. The deposition reaction between the chemical components of the donor and the activator precursor vapors form the element layer, the layer being formed by the reaction of the substrate with the vapors produced from the precursor [6-8].

2. Results

Pre-eliminate tests were conducted in the CVD-FBR reactor using a fluidized bed formed by a 5% aluminum powder, 5% silicon powder and 90% bed inert (alumina). It was fluidized with 61% argon and as gas activators a mixture of hydrogen 37.1% and hydrochloric acid 1.9%, for a time of 90 minutes at 580°C. This was done to find the best conditions of the deposition process on the stainless steels. The above conditions are producing good coatings with a thickness approximately of 10 microns ($\pm 4 \mu\text{m}$). The amount of silicon is incorporated into the coating is below 0.5%. Because the amount of silicon was minimal in the co-deposition aluminum-silicon we analyzed the simulation thermodynamics and literature results [5-10] and we determine the lowest the deposition temperature was 540°C. This is due to the greater amount of ClH_3Si present in the process of deposition. Therefore there is a greater possibility the silicon is deposited and best thermal activation increasing the probability of obtain a good aluminum-silicon coating. The best aluminum-silicon coatings were obtained when deposition was carried out at 540°C with a ratio of active gases $\text{HCl}/\text{H}_2:1/15,3$ and 2.5 g of aluminum and 7.5 g of silicon in the bed and a 50% active gases, neutral gases 50%. The best samples are reached in the early stages of fluidization.

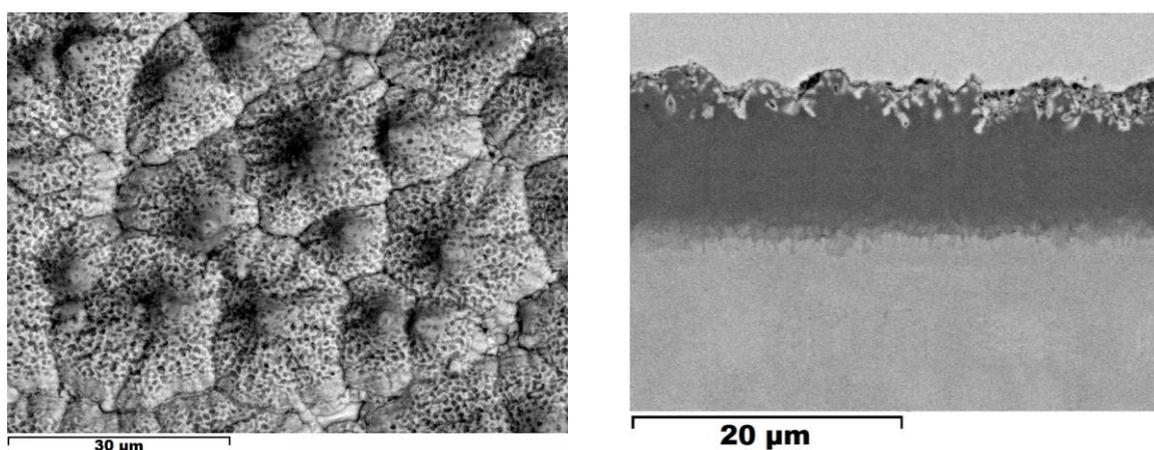


Figure 1. Morphological surface (a) and cross-sectional (b) image of aluminum-silicon coating.

The figure 1. Shows the SEM results for aluminum-silicon coating. The fig 1a shows a cone-shaped morphology of the surface. The chemical composition of the coating is 54.59% Al, 24.35% Fe, 8.05% Ni, 7.67% Cr, 3.62% Si, 0.62% Mg and 1.10% Mo. The aluminum is found like FeAl_2Si , Fe_2Al_5 and FeAl_2 . These coatings were heat treated to improve its mechanical properties by the interdiffusion of the alloying elements. These coating heat treated are formed by compounds of FeAl , Al_2FeSi , Cr_3Si , AlCrFe_2 , and AlFeNi . The FeAl is the greater extent on the outside of the layer. The coatings no heat treatment have a higher amount of aluminum than heat treated coatings. After heat treatment disappear the compounds of Al_3Fe_2 , FeAl , decreasing the percentage of aluminum and increasing the percentages of iron, chromium and nickel.

The heat treatment favoring the transformation of aluminum rich phases, in other more iron content, thereby improving the coating mechanical properties and corrosion resistance. Finally, it can be concluded that by the process FBCVD can obtain coatings of aluminum-silicon 10 microns thick, for the deposition temperature of 540 °C and a deposition time of 1 hour. After a heat treatment such layers can grow up to 14 μm thick, because appears a inter diffusion zone within the substrate. The

thermodynamic simulation is a good tool to select the working conditions and know the aluminum and silicon halides that are taking the deposition of the coating.

3. Conclusions

Were optimized deposition conditions of the coatings of Al-Si by FBCVD at temperatures below 600 °C. These temperatures do not affect the microstructure of the steels. We obtained thicknesses greater than 14 microns. The Temperature and flow of the precursor gases are directly related to the kinetics of growth of the coatings. The temperature of deposition of the coatings should not exceed 560 °C because they begin to chlorinate the coatings and not able to obtain good layers. This paper had the highest coating thickness for the aluminum-silicon alloy at 540 °C to 60 minutes and with active gas mixture of 50% argon, 46.83% hydrogen and 3.17% HCl, with a bed of 2.5 g Al, 7.5 g Si, and 90g of Al₂O₃. conductivity. Any additional amount of HEM produces a negative effect on PANI/HEM conductivity.

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