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GAS CORROSION DAMAGE IN Ti-STABILIZED INTERSTITIAL FREE STEEL

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Abstract

The selective oxidation damage in the Ti-stabilized interstitial free steel during 60 s of recrystallization annealing at 820 °C under the different compositions of protective H₂-N₂ atmosphere at low dew point (-40 °C) was investigated using various experimental techniques. It was found that Mn, Al and Si oxide particles are the main products of external and internal oxidation. Increase of the H₂ content in gas atmosphere favors external oxidation and leads to appearance of greater nonwetted surface areas.

Introduction

The surface segregation and selective oxidation during recrystallization annealing of high strength steels used in automotive industry can cause the poor wettability of the steel surface by molten baths during hot-dip galvanizing [1], reducing the quality of corrosion-protective coatings [2]. The external presence and appearance of nonwettable Mn-oxides have the main influence on the coatings coverage. Since it is impossible to prevent external oxidation [3], it is very important to determine the annealing conditions and surface state which would improve the wetting and spreading of the coating melts, changing the nature, morphology and distribution of oxide particles. In this work, the effects of protective H₂-N₂ atmosphere composition on the selective oxidation mode and external/internal particle characteristics were investigated in the case of Ti-stabilized interstitial free steel (Ti-IF).

Experimental Procedure

The investigated steel with the chemical composition: Fe-0.0028C-0.0033N-0.085Mn-0.006Si-0.044Al-0.0001B-0.016Cr-0.006P-0.006S-0.073Ti (in wt.%) was supplied in the form of 0.8mm thick cold-rolled sheets. Metallographically prepared samples were annealed for 60 s at 820 °C under the 5 vol.% H₂-N₂ and 15 vol.% H₂-N₂ protective atmosphere with traces of water (dew point of -40 °C). The characterization of sheet surface composition before and after annealing was carried out using a LEO 1550 VP field emission scanning electron microscope (FE-SEM) equipped with energy dispersive spectrometer (EDS) at a 15 kV voltage and PHI Quantum 2000 X-ray photoelectron spectrometer (XPS). The XPS depth profiles were recorded with a sputter rate of 2.67 nm/min.

Results and Discussion

The XPS analysis of cold rolled sheet surface showed that it is covered with 38.9 nm thick oxide layer, mainly containing Fe_2O_3 and Fe_3O_4 . The presence of these oxides is confirmed with appearance of O 1s and Fe 2p peaks on XPS spectrum shown in Fig. 1a. A weak Si 2p peak indicates that SiO_2 is also present, while an appreciable amount of C may only be present as a contaminant.

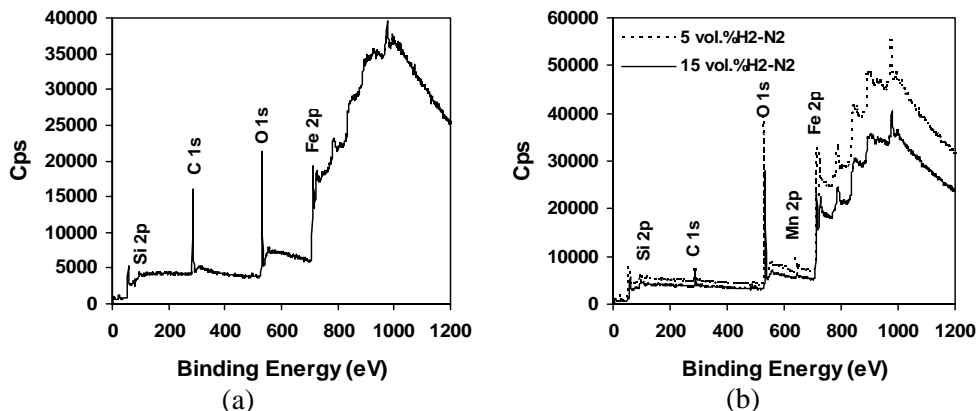


Fig. 1. XPS spectra recorded on as-cold rolled sheet surface of Ti-IF steel (a) before and (b) after annealing under $\text{H}_2\text{-N}_2$ protective gas atmosphere for 60 s.

During annealing at 820 °C the native Fe-oxides are reduced which is confirmed with the shift of the Fe 2p peak towards lower binding energy, Fig. 1b. Simultaneously, the external and internal oxidation of alloying elements is progressive, Fig. 2. The Mn oxides growing at the sheet surface are the main product of selective oxidation. Double Mn 2p and Si 2p peak, Fig. 1b, at the position which does not correspond to the formation of SiO_2 , indicate that different types of external Mn oxides are present, Fig. 3. Nodular MnO islands formed inside the grain are found only after annealing under 15 vol.% $\text{H}_2\text{-N}_2$ atmosphere, Fig. 3b. Another Mn oxide detected as complex MnSiO_3 oxide is precipitated at the grain boundaries during annealing under both conditions. Since the partial pressure of oxygen is decreased with increased H_2 content in the gas atmosphere, segregating elements have enough time to diffuse to the surface. As a result, the outer oxide layer is thicker than on the sheet surface annealed under 5 vol.% $\text{H}_2\text{-N}_2$ atmosphere. The formation of 39.2 nm thick oxide layer is primarily consequence of the expressive external oxidation. Large surface coverage with external oxides limits the oxygen penetration into the steel and decreases the internal oxidation as shown in Fig. 2b. As can be seen, the concentration of Al increases slightly with depth indicating the presence of a small amount of internal Al_2O_3 oxide. The behavior of Ti-IF steel during annealing under 5 vol.% $\text{H}_2\text{-N}_2$ atmosphere is quite different. The smaller surface coverage is observed. Except separate MnSiO_3 islands, Fig. 3a, the particles of other oxides are not present. Moreover, the beneficial TiN particles are sporadically present at the surface. Intensive internal oxidation and deeper presence of Al_2O_3 oxide led to formation of 50.4 nm thick oxide layer.

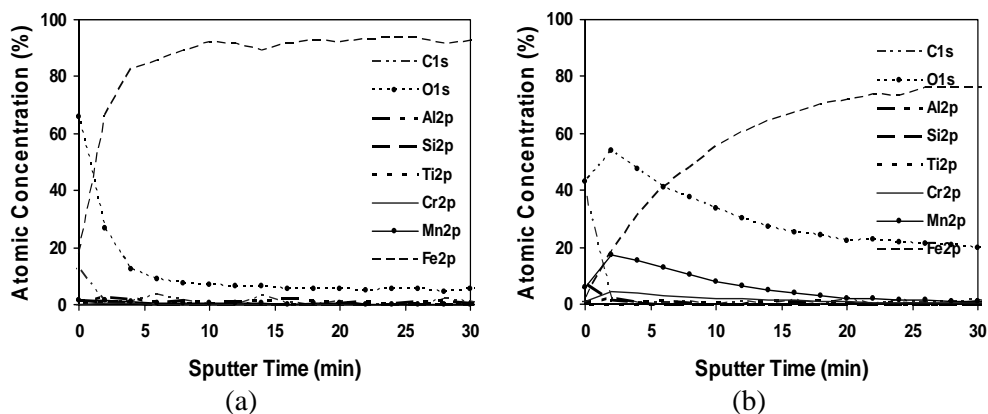


Fig. 2. XPS in-depth profiles on Ti-IF steel after annealing under (a) 5 vol.% H₂-N₂ and (b) 15 vol.% H₂-N₂ atmosphere for 60 s.

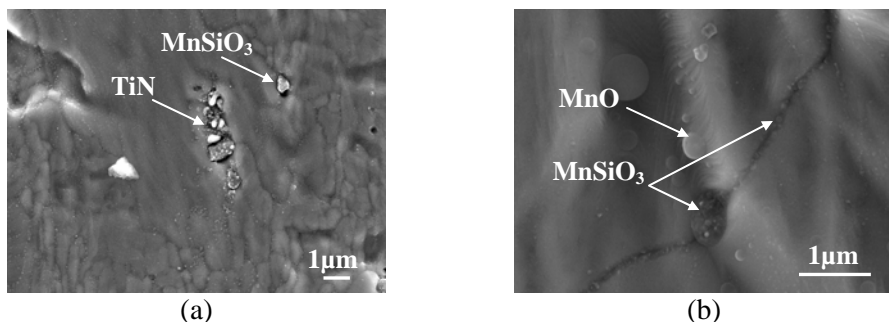


Fig. 3. FE-SEM micrographs of Ti-IF steel surface after annealing under (a) 5 vol.% H₂-N₂ and (b) 15 vol.% H₂-N₂ atmosphere for 60 s.

Conclusion

The selective oxidation of Ti-IF steel, occurring externally and internally during annealing at 820 °C, is strongly dependent on the H₂-N₂ protective atmosphere composition. External formation of MnO and MnSiO₃ islands is predominant. Internal appearance of Al₂O₃ oxide contributes to the overall thickness of oxide layer. As the H₂ content increases from 5 to 15 vol.% the surface coverage with nonwetable oxides increases. On the other hand, decrease in the H₂ content favors internal oxidation and leads to increase of oxide layer thickness for 11.2 nm.

Acknowledgements

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