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TRANSMISSION ELECTRON MICROSCOPICAL STUDY OF THE STAINLESS STEEL SPECIMENS AFTER ION-PLASMA TREATMENT

[1,2]Zagibalova E.A., [1]Moskvina V.A.

[1]*Institute of Strength Physics and Materials Science SB RAS, Tomsk*

[2]*National Research Tomsk Polytechnic University, Tomsk*

Using the transmission electron microscopy (TEM) methods, we studied phase composition and microstructure of the surface composite layers obtained by ion-plasma treatment in austenitic stainless steel specimens (Fe-17Cr-13Ni-2.7Mo-1.7Mn-0.6Si-0.01C mass.%, AISI 316L) with different initial structures. Two portions of homogenized and solution-treated AISI 316L billets were cold rolled to 80% reduction (Regime 1 – R1) and 40% reduction (Regime 2 – R2). Further, R1-specimens were investigated in as-rolled state, R2-specimens were annealed in a helium atmosphere at the temperature of T=1050°C for 5 h with quenching into water. In cold-rolled 316L steel specimens (R1), a misoriented grain-subgrain structure (d<1 μm) with a high density of deformation defects (dislocations, subboundaries, twins, localized bands, etc.) and both high- and low-angle misorientations between structural elements was obtained. In cold-rolled and annealed specimens (R2), a coarse-grained austenitic structure with an average grain size of d = 55 μm was formed. R1 and R2 specimens were subjected to a surface ion plasma treatment (IPT) in a mixture of gases 70% Ar + 25% N_2 + 5% C_2H_2 at pressure 300 Pa and temperature of 540°C for 12 hours using ELU-5 device. TEM microstructural analysis was carried out using the foils obtained in cross-section of IPT-processed surfaces.

Under IPT-processing of stainless steel, the surface-hardened regions with the depth of ≈150 μm in R1 and ≈50 μm in R2-specimens are formed. By the features of the microstructure and phase composition with depth, in the IPT-processed surfaces of both R1 and R2 specimens three characteristic regions were revealed: a composite layer, a diffusion zone and a base material. For R1-specimens, the microstructure of the composite layer is inhomogeneous and consists of extended regions doped with nitrogen and carbon austenite Fe-$\gamma_{N,\,C}$, ferrite Fe-$\alpha_{N,\,C}$, and ultra-fine carbonitrides and nitrides Fe_4(N, C) and Cr(N, C) located along numerous boundaries and subboundaries and within grains. The diffusion zone of the R1-specimens is characterized by a microstructure with extended regions with the Fe-$\gamma_{N,\,C}$ phase and high density of deformation twins. As moving from the IPT-assisted surface towards the center of R1-specimens, the microstructure becomes typical to the initial base material, i.e., the grain/subgrain austenitic structure. According to TEM phase analysis, the phase composition of the composite layers in both R1 and R2 states is similar, but the phase distribution, their morphology and volume fraction are completely different. In case of R2-specimens, the microstructure of IPT-assisted composite layer is more homogeneous in comparison with that in R1 specimens. In the composite layer, from the top surface up to depth of ≈15-18 μm, the initially coarse grains with the Fe-$\gamma_{N,\,C}$ phase are filled with Fe_4(N, C) particles with a strip-like arrangement inside these grains. Particles of the Fe_4(N,C) phase have a length of 1÷5 μm and a width of 30÷120 nm. The areas next to the grain boundaries are depleted by Ni, Cr and Mn but enriched with Fe, C and N according to EDS data. Strip-like Fe_4(N,C) particles are parallel to each other and they are located along certain crystallographic planes in the austenite structure. Areas ≈1 μm in size with fine particles of Cr(N, C) + Fe-α phases are observed only up to 10 μm in depth near the boundaries and inside of grains. At ≈ 50 μm depth, the microstructure of R2-specimens is similar to that in base material - coarse-grained austenite.

Thus, it is experimentally shown that the initial structure in 316L steel affects the phase distribution and morphology of the composite IPT-assisted surface-hardened regions despite the formation of similar set of secondary phases in these regions.

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