Application of Mössbauer spectroscopy for surface investigation of 316L stainless steel after temperature treatment

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Austenitic stainless steel tends to form a protective layer under the influence of high temperatures. Mössbauer spectroscopy can be applied to the characterization surface and bulk of the material. The experiment requires an iron-containing alloy to observe the effect of resonant absorption of gamma quanta. Conversion X-ray Mössbauer spectroscopy (CXMS) and conversion electron Mössbauer spectroscopy (CEMS) provide information about the material structure at different depths, 10 μm and 0.3 μm, respectively. This method can give information on the inhomogeneity of the surface layer of the material.

The studied samples were made of metal powder of austenitic stainless steel 316L by selective laser melting (Concept Laser, Germany). Initial size of finished samples 25x25x2 mm^3. After 3D printing samples were grinded by the sand and annealed in air in the temperature range 550 – 1000 °C during 2, 8, 32 hours. In addition to Mössbauer spectroscopy, X-ray diffraction (XRD) was used to study the phase composition of the oxide layer. Scanning electron microscopy (SEM) and energy-dispersive analysis (EDS) were used for the investigation of surface morphology and elemental composition, respectively.

The CXMS spectra for the samples annealed at 550 ℃ showed the presence of FCC austenite (γ - phase). The obtained spectrum suggests that iron atoms are located in the FCC lattice in the presence of alloying elements, which caused a slight quadrupole splitting of the spectrum. No oxide phases were identified at this depth. CEMS studies have shown that the tradition FCC structure is present in all spectra. In addition to the austenitic phase, there is also the presence of the BCC structure (α – phase). The sextet corresponding to the α - phase increased with increasing annealing time. The samples studied using the CEMS method showed also the presence of Fe^{3+} doublet, which corresponds to the iron-containing oxide layer. The combination of Mössbauer spectroscopy and other methods (EDS, XRD) allowed to recognize the oxide layer as spinel oxide $(Fe\text{-}Cr)_2O_3$. According to elemental analysis, the amount of chromium increases in the surface layer of the sample. Based to the obtained data, the approximate depth of the oxide layer is 0.6 – 1 μm. The CXMS spectra for all samples annealed in the temperature range 700 – 1000 ℃ showed the presence of a singlet peak corresponding to the FCC phase of iron. With an increase in the temperature and holding time of the samples in the furnace, an oxide layer appears on the surface of the samples. On the basis of CEMS spectra, this oxide layer is thin and contains Fe^{3+} atoms. This oxide layer is not homogeneous and its thickness increases with increasing temperature and exposure time. EDS analyses demonstrate that chromium contest increases during annealing. At the same time, at the 1000 °C annealing, the manganese concentration began to grow on the surface of the sample. XRD analysis showed the presence of spinel type $(Fe\text{-}Cr)_2O_3$ oxide and Mn-rich oxide $Mn_2(Fe\text{-}Cr)O_4$. This formation of oxides can be explained by the fact that chromium has a great affinity for oxygen and under the influence of the temperature prone to diffusion. Meanwhile, manganese easily diffused in the oxide layer due to the higher diffusion coefficient.