

RIETVELD QUANTITATIVE ANALYSIS OF SUPER DUPLEX STAINLESS STEEL

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The widespread industrial applications of the so-called “super duplex” stainless steels is based upon their remarkable mechanical and chemical properties. The microstructure of these alloys is composed of approximately equal proportions of BCC ferrite and FCC austenite phases. However, high-temperature processes such as heat treatment and welding cause embrittlement and loss of corrosion resistance due to precipitation of intermediate phases, principally sigma-phase, in the microstructure. This phase is a complex intermetallic compound of Fe and Cr, which considerably affects the main properties of the alloy. The structure type of the compound is based upon an ideal stoichiometric composition AX_2 , Pearson's code tP30, and space group $P4_2/mnm$. Since the chemical composition in the Fe-Cr system is approximately Cr_6Fe_7 , the Fe and Cr atoms are disorderly located with fractional site occupation factors among the suitable equivalent positions in the space group, disclosing a polyhedral array of the Frank-Kasper type. Although sigma-phase is known to form quite rapidly in the Fe-Cr-Ni system, detailed phase identification is not easily obtained. In order to interpret highly superimposed diffraction patterns, the Rietveld method together with conventional XRD techniques were conveniently carried out on several heat-treated weldments of cast ASTM A890 super duplex stainless steel. The room temperature data collection from each specimen was carried out over the range of 30 to 85° (two-theta) with step size 0.02° and measurement time 15 s, using Bragg-Brentano geometry (theta-theta scan) and Cu K-alpha radiation with graphite monochromator for the reflected beam. The analysis was performed assuming the split Pearson VII function for the simulation of the peak shapes, while the background was modeled by a 4rd order Chebychev polynomial for all reflections; the refinement cycles were based upon the variations of atom positions and isotropic thermal parameters, scale factors, cell parameters, preferred orientations coefficients, surface roughness, background polynomial coefficients, specimen transparency, specimen displacement and others. This procedure permitted an accurate quantification of phases such as austenite, ferrite and sigma-phase in all studied samples. Hence the combination of XRD and Rietveld processing is a powerful analytical technique for the quantitative structural characterization of phases in the Fe-Cr-Ni system. Moreover, the main advantage of Rietveld method over conventional quantitative metallography, is that image analysis by computer-aid microscopy is highly depending upon the ability of the optical system to resolve the subject in question. From the metallurgical point of view the XRD results are in accordance with the results of complementary techniques such as emission spectrometry, scanning electron microscopy and magnetic measurements. Finally, the data evaluation of the diffraction patterns derived in the metallurgical results shown in the figure below, where the content (mass %) of sigma-phase is plotted versus processing time of a weldment annealed at 1123 K.

