Conversion of MX Nitrides to Modified Z-Phase in 9-12%Cr Ferritic Steels (12/12/2018)

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The 9-12%Cr ferritic steels are extensively used in modern steam power plants at service temperature up to 620°C. Currently the best performing ferritic creep resistance steel is the ASTM Grade 92, whose high temperature strength has recently been assessed by European Creep Collaborative Committee in 2005 as 600°C/113MPa/10^5h. All previous attempts made in the last twenty years to develop ferritic steels for 650°C applications have failed due to the incapacity to combine the superior oxidation resistance, given by 12%Cr content, with excellent creep resistance of high-alloyed ferritic steels. Indeed the fast conversion of finely distributed MX nitrides, which highly promote creep strengthening, to larger and thermodynamically more stable modified Z-phase, Cr(V,Nb)N, led to an unforeseen drop of creep resistance of 12%Cr steels. Since chromium content was confirmed to be the main driving force for Z-phase formation, this explains why 12%Cr steels (i.e. P122, VM12, NF12) suffer from fast and abundant Z-phase precipitation, while 9%Cr steels (i.e. Grades 91, 92, 911) do not. In this thesis the role of vanadium and niobium nitrides in the formation of Z-phase in 9-12%Cr steels is investigated. With this purpose in mind, two 12%Cr model alloys, 12CrVNbN and 12CrVN, with ultra low carbon content, were manufactured. Both model alloys consisted of Cr-, V- and Nb-nitrides only. The first model alloy, 12CrVNbN, was especially designed to quickly convert the complex V- and Nb-nitrides into modified Z-phase. The second model alloy, 12CrVN, was selected to investigate the transformation of pure V-nitride into V-based Z-phase, CrVN, and through comparison to understand the effect of Nb. Thus, without the disturbing interferences of carbides and intermetallic phases (i.e. M23C6, NbC, Fe2(Mo,W)) it was possible to identify all stages of MX conversion to Z-phase particle during ageings at 600°C, 650°C and 700°C up to 10^4 hours. Transmission Electron Microscopy (TEM) and X-Ray powder Diffraction (XRD) were applied to follow the microstructural evolution of the nitrides of model alloys during ageings: morphology, crystal structure, chemical composition and equivalent diameter of precipitates were assessed over times and temperatures. The mechanism of Z-phase formation was identified in the model alloys; hybrid MX/Z particles were found as mid-stage phases of this conversion. The key-role of niobium as accelerator for Z-phase formation was highlighted during the studies. Several 9-12%Cr commercial steels with prolonged high-temperature exposures have been investigated, too. The same mechanism of Z-phase formation observed in 12%Cr model alloys was identified in industrial 9-12%Cr steels after thousands of hours of high-temperature service. The first MX/Z-phase hybrid particles in Grade P91 and P92 were identified during the Ph.D. project.

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