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## Thermal Ageing of a 316 Nb Stainless Steel: From Mechanical and Microstructural Analyses to Thermal Ageing Models for Long Time Prediction

Authors: Julien Monnier, Isabelle Mouton, Francois Buy, Adrien Michel, Sylvain Ringeval, Joel Malaplate, Caroline Toffolon, Bernard Marini, Audrey Lechartier

Abstract: Chosen to design and assemble massive components for nuclear industry, the 316 Nb austenitic stainless steel (also called 316 Nb) suits well this function thanks to its mechanical, heat and corrosion handling properties. However, these properties might change during steel's life due to thermal ageing causing changes within its microstructure. Our main purpose is to determine if the 316 Nb will keep its mechanical properties after an exposition to industrial temperatures (around 300 °C) during a long period of time (< 10 years). The 316 Nb is composed by different phases, which are austenite as main phase, niobium-carbides, and ferrite remaining from the ferrite to austenite transformation during the process. Our purpose is to understand thermal ageing effects on the material microstructure and properties and to submit a model predicting the evolution of 316 Nb properties as a function of temperature and time. To do so, based on Fe-Cr and 316 Nb phase diagrams, we studied the thermal ageing of 316 Nb steel alloys (1%v of ferrite) and welds (10%v of ferrite) for various temperatures (350, 400, and 450 °C) and ageing time (from 1 to 10.000 hours). Higher temperatures have been chosen to reduce thermal treatment time by exploiting a kinetic effect of temperature on 316 Nb ageing without modifying reaction mechanisms. Our results from early times of ageing show no effect on steel's global properties linked to austenite stability, but an increase of ferrite hardness during thermal ageing has been observed. It has been shown that austenite's crystalline structure (cfc) grants it a thermal stability, however, ferrite crystalline structure (bcc) favours iron-chromium demixion and formation of iron-rich and chromium-rich phases within ferrite. Observations of thermal ageing effects on ferrite's microstructure were necessary to understand the changes caused by the thermal treatment. Analyses have been performed by using different techniques like Atomic Probe Tomography (APT) and Differential Scanning Calorimetry (DSC). A demixion of alloy's elements leading to formation of iron-rich ( $\alpha$  phase, bcc structure), chromium-rich ( $\alpha$  phase, bcc structure), and nickel-rich (fcc structure) phases within the ferrite have been observed and associated to the increase of ferrite's hardness. APT results grant information about phases' volume fraction and composition, allowing to associate hardness measurements to the volume fractions of the different phases and to set up a way to calculate  $\alpha'$  and nickel-rich particles' growth rate depending on temperature. The same methodology has been applied to DSC results, which allowed us to measure the enthalpy of  $\alpha'$  phase dissolution between 500 and 600 °C. To resume, we started from mechanical and macroscopic measurements and explained the results through microstructural study. The data obtained has been match to CALPHAD models' prediction and used to improve these calculations and employ them to predict 316 Nb properties' change during the industrial process.

**Keywords:** stainless steel characterization, atom probe tomography APT, vickers hardness, differential scanning calorimetry DSC, thermal ageing

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