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Sampling of P91 base metal for in-situ neutron powder diffraction and dilatometer evaluation / characterisation

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ASTM SA-335 Grade P91 / T91, a 9%Cr creep strength enhanced ferritic (CSEF) steel, is commonly used in modern power industry for manufacturing critical pressure components and vessels, tubing, piping and headers. The objective of the current study is to determine the temperatures at which transformation of BCC ferrite to FCC austenite starts and finishes on heating (the so-called Ac1 and Ac3 temperatures). In addition, the transformation of austenite to delta ferrite (a BCC structure) at higher temperatures must be characterized. As reported previously, neutron powder diffraction and dilatometry were selected to characterize these phase transformations. A small number of isothermal heat treatments, followed by water quenching was also done, to verify neutron diffraction results at temperatures that could not be achieved using the dilatometer. The objective of the current paper is to describe calibration and sampling methods in P91 base metal for such phase transformation.Temperatures in the dilatometer and neutron furnace were verified by comparing the experimentally measured phase transformation temperatures of pure iron rod with the theoretical temperatures.By using room temperature neutron powder diffraction, the crystallographic preferred orientation in a tubular sample of P91 base metal was established along the longitudinal, through-thickness and tangential direction by comparison of intensity ratios of the diffraction peaks with those of Fe powder. It was found that the tangential sample machined exhibited intensity ratios closer to those of Fe powder. The longitudinal sample presented severe preferred orientation. Dilatometer results were insensitive to the orientation of the sample. The Ac1 and Ac3 temperatures in the different oriented samples were determined in the range 809-811℃ and 875-811℃ respectively in the dilatometer. The quenching methods revealed that at 1150℃, the onset of delta ferrite in P91 base metal has already commenced, which agrees with previous temperature range obtained by in-situ neutron diffraction (between 1140 and 1160℃). The calibration results indicate that the error in temperature (between published and experimentally determined temperatures, and between neutron diffraction and dilatometry results) was less than 5℃, and that the combination of neutron diffraction, dilatometry and isothermal heat treatments resulted in an assessment of the phase transformation behavior over a wide range of temperatures, not achievable using only one characterization technique.

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P.G.H.Pistorius, UP, Pieter,Pistorius@up.ac.za
A.M. Venter, Necsa SOC, andrew.venter@necsa.co.za
J.P.R. deVilliers, UP,johan.devilliers@up.ac.za

Primary authors: Dr VENTER, Andrew (Necsa Limited); Prof. DEVILLIERS, Johan (Department of Materials Science and Metallurgical Engineering, University of Pretoria); Prof. PISTORIUS, Pieter (Department of Materials Science and Metallurgical Engineering, University of Pretoria); Ms SENTSHO, zeldah (necsa)

Presenter: Ms SENTSHO, zeldah (necsa)

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