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Verification of phase transformation temperatures of 9%Cr ferritic steel using dilatometry and neutron powder diffraction

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Creep strength enhanced ferritic steels [1] are used in the manufacture of critical pressure components and vessels in modern power industry due to high thermal conductivity, low thermal expansion, high strength and resistance to creep at elevated temperatures. 9%Cr steel [2] is of technological relevance since it contains elements which provide precipitation strengthening by forming $M_{23}C_6$ carbides and niobium containing carbonitrides. As for many other steels that have a ferritic structure at room temperature, a number of phase transformations occur in 9%Cr steels on heating. At the so-called A_{c1} temperature, about 800°C , the transformation of ferrite to austenite begins. At the A_{c3} temperature the structure is fully austenitic. On further heating, the transformation of austenite to delta ferrite occurs, at about 1390°C [3].

Phase transformation temperatures are traditionally established with dilatometry measurements [4] with the onset and progress of transformations inferred from changes in the slope of a graph of sample length vs temperature. The change in slope is in the first instance due to the phase transformation of the bcc ferrite structure to fcc austenite. Notwithstanding dilatometry being sensitive for revealing the consequences of phase transformations, it remains an indirect measure of the onset of phase transformation. If phase transformation results in a dual phase structure with approximately constant volume fractions, the dilatometric curve will show an approximately constant slope. Such a slope can, incorrectly, be interpreted as indicative of a single phase structure.

By using the capability of thermal neutron diffraction [5] as direct probe of crystallographic phase changes averaged over the volume of the sample, in conjunction with in-situ heating, direct quantification of the respective phases through the application of the Rietveld method is possible at a range of temperatures. In addition, by doing neutron diffraction at pre-determined sample temperatures, an alternative method exists for compiling/verifying phase transformation temperatures. The completion of transformation is better defined with neutron powder diffraction and also the study of the austenite to delta ferrite transformation at about 1300°C is possible. Preliminary results of the heat treatments conducted at Necsa indicate the range of temperatures for the beginning and end of the transformation of ferrite to austenite on heating. Results from both approaches will be compared and discussed.

Summary

The following references were used in the write up of this abstract:

- [1] B. Lun Wang, J. C. Lippold, and A. S. Sudarsanam Babu, "Development of Predictive Formulae for the A_{c1} Temperature in Creep Strength Enhanced Ferritic Steels," The Ohio State University, 2010.
- [2] J. Parker, "EPRI Project Manager Guidelines and Specifications for High- Reliability Fossil Power Plants Best Practice Guideline for Manufacturing and Construction of Grade 91 Steel Components disclaimer of warranties and limitation of liabilities," 2011.
- [3] D. J. Abson and J. S. Rothwell, "Review of type IV cracking of weldments in 9–12%Cr creep strength enhanced ferritic steels," Int. Mater. Rev., vol. 58, no. 8, pp. 437–473, 2013.
- [4] K. W. Andrew, "Empirical Formulae for the Calculation of Some Transformation Temperatures," J. Iron Steel Inst., vol. 203, pp. 721–727, 1965.

[5] S. C. Vogel, "A Review of Neutron Scattering Application to Nuclear Materials," ISRN Mater. Sci., pp. 1–24, 2013.

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