

In Situ Neutron Diffraction Study of Phase Transformation in Zr-Excel Alloy

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An in situ heating experiment was performed to measure the phase transformation temperatures of a Zr alloy, known as Excel. The Excel alloy, developed by AECL, is an α/β zirconium alloy proposed for use in the CANDU-concept Gen IV nuclear reactor, with relatively high alloying content (3.5%Sn-0.8%Mo-0.8%Nb-0.15%Fe). The high alloying content and competing diffusion rates and effects of the different solutes leads to difficulty in predicting phase transformation temperatures. Previous experimental studies on the phase transformation temperatures of this alloy involved quenching from elevated temperatures followed by ex situ metallography [1]. A direct assessment of the $\alpha+\beta \rightarrow \beta$ temperature cannot be made by this technique, due to the combined effects of the formation of α' martensite from β phase during quenching and the retention of metastable β at room temperature. This experiment will be compared with the results of the previous ex situ study, avoiding the problems associated with heating (as oxygen uptake has been noted as impacting relative phase stability [1]) and quenching.

Samples were machined from Excel pressure tube, provided by AECL. Samples were handled with gloves and cleaned with acetone prior to testing to prevent contamination, which could alter the apparent phase transformation temperatures. Samples were heated with in situ neutron diffraction at the C2 beam line at NRU at Chalk River Laboratories, in Chalk River, Ontario.

The sample was held in vacuum during heating to prevent oxidation. The thermocouple was protected from direct heating with a Nb foil shield, which also provides an indirect measure of the sample temperature by via thermal expansion which can be determined from the displacement of Nb diffraction peaks. Good resolution of peaks was achieved. Sample peaks were fit with a Gaussian peak profile for initial analysis. A small section of the diffraction pattern is shown in Figure 1. Five peaks are shown – three from the α -Zr phase, one from the β -Zr phase, and a very broad peak which is believed to be from the ω -Zr phase.

The evolution of the integrated intensity of the β (110) peak during one heating cycle is summarized in Figure 2. There is good qualitative agreement between the ex situ metallography data reported by Sattari et al. [1] on β phase volume fraction and the behaviour of the β (110) peak intensity measured here. Further analysis and Rietveld refinement of the experimental data will be required to extract phase volume fractions at different temperatures, which will then be compared against theoretical models in development to fully characterize the phase behaviour of this alloy.

Reference

- [1] Sattari M, Holt RA, Daymond MR. J Nucl Mater 2013;435:241

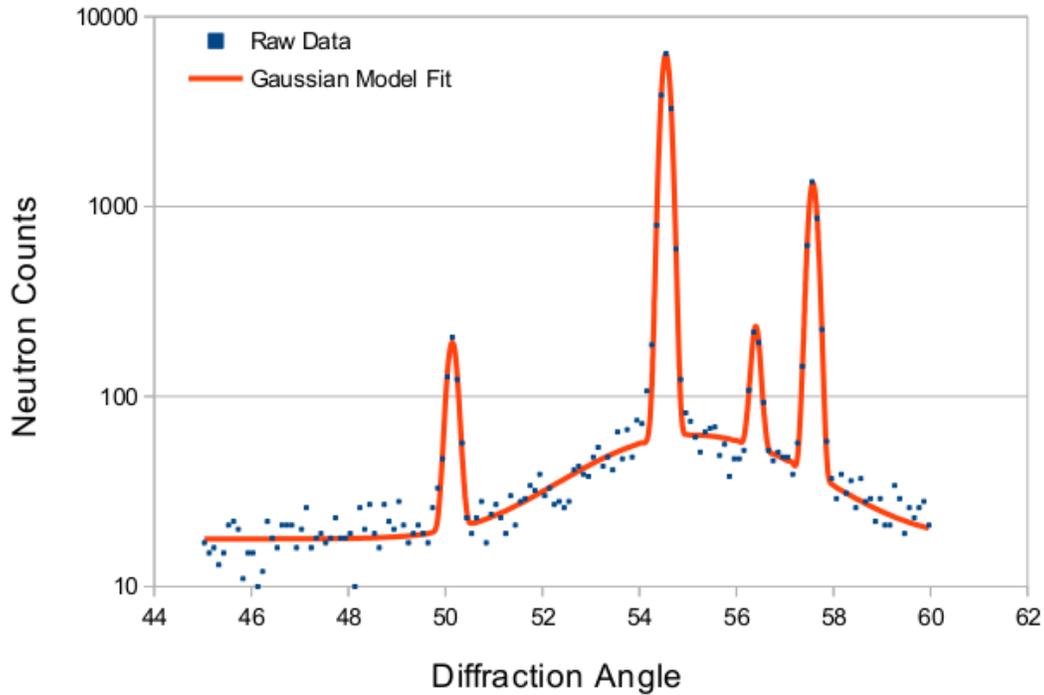


Figure 1 Selected region of diffraction pattern for Zr-Excel alloy measured at C2 at 650°C. Three peaks belonging to the α phase and one peak belonging to the β phase are easily resolved. The presence of a very broad peak is apparent (centred at 55°), which is believed to be due to the ω phase, which is known to have very fine precipitates.

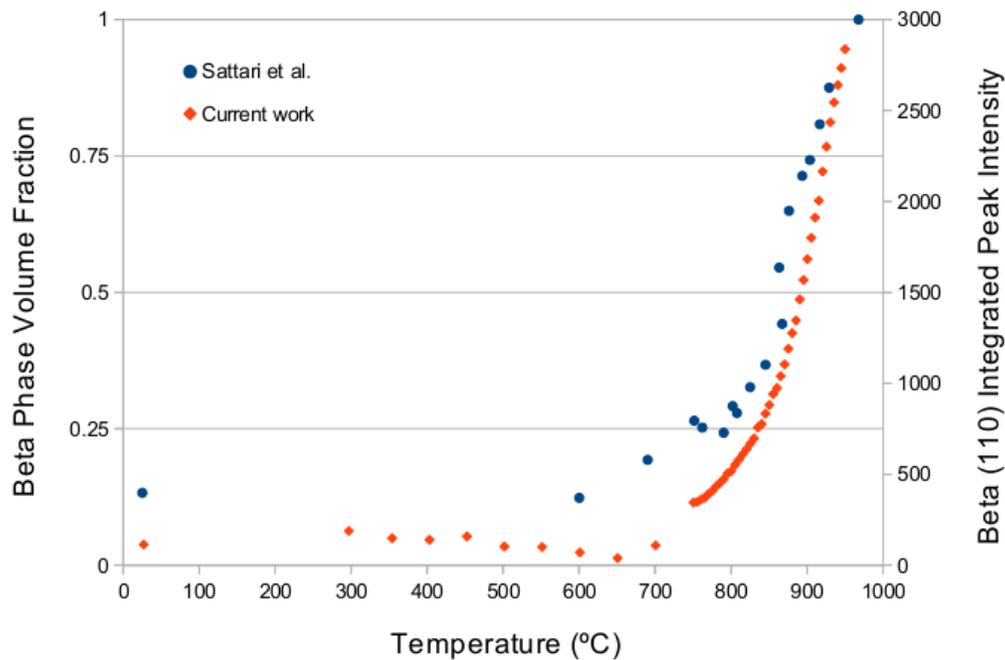


Figure 2 Comparison of the current work with results presented by Sattari et al [1]. Good qualitative agreement is seen between the two data sets. The $\beta(110)$ peak integrated intensity appears to increase rapidly with heating above 700°C.