Measurement of Minority Phase Fraction Following Processing of a Heat Treated Zirconium Excel Alloy

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Materials Science

This series of measurements was performed to investigate the kinetics of two opportunities for metallurgical strengthening of the proposed Zr Excel alloy: the growth of a precipitate ω phase, and transformation-induced plasticity. The Excel alloy, developed by AECL, is an α/β zirconium alloy proposed for use in the CANDU-concept Gen IV nuclear reactor. Previous studies on the effect of heat treatment on this alloy have revealed that quenching from below 850ºC results in a retained metastable β phase. These measurements allowed us to investigate the possibility of utilizing this retained metastable β phase to further enhance mechanical properties, through either conversion to ω phase, or mechanically induced transformation to the α phase.

Growth of the ω Phase

The first of our goals, the growth of precipitate ω phase, was motivated by the use of precipitation hardening in the Zr-2.5wt%Nb alloy, the current alloy used for pressure tubes in CANDU reactors. Heat treatments were performed *ex situ* on cubes of Excel (1.2mm side length), heating at time intervals from 0 – 100 hours at a temperature of 450ºC followed by a water quench. Pole figures were measured at E3 spectrometer of the heat treated specimen, in order to track the growth and decline of both the precipitate ω phase and the metastable β phase. Results of these measurements indicate that the ω phase forms from the metastable β following the Burgers relationship, as expected, and that the growth and dissolution of the ω phase is complete following 100 hours at 450ºC. Analysis of the results was complicated by sample-to-sample variations in heat treatment and the potential for oxygen pickup during heat treatment, and *in situ* heating tests have been proposed to control these factors.

Mechanically induced transformation of β phase

The second goal of our study was to provide preliminary evidence supported a proposed transformation-induced plasticity in Excel alloy, analogous to the TRIP effect in steels. Samples were deformed ex situ in tension along both of the plate rolling and plate transverse directions. Pole figures in both the α and β phase were measured both before and after deformation using the E3 spectrometer. Texture evolution during deformation was observed, although some of the reorientation may be due to twinning, which has been previously observed in many zirconium alloys. Evidence of a phase transformation is present, however, due to the apparent orientation relationship between the new regions of basal intensity following tension in TD and the initial (110)β pole figure (Figures 2b and 2c). This relationship is in agreement with the Burgers relationship, {110}β||{0002}α, which is observed in the β→α transformation that occurs on cooling from the β to β+α phase fields. Further tests, both *ex situ* and *in situ*, have been proposed to better understand this phenomena.

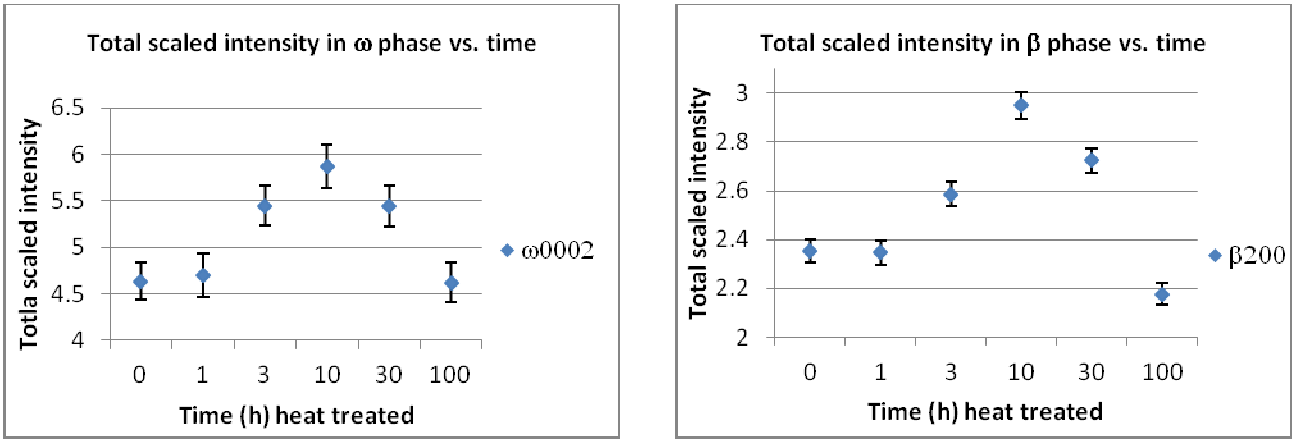

Figure 1 Total scaled intensity of the (left) ω and (right) β phase as a function of heat treatment time for a furnace temperature of 450ºC. Both phases increase in total scaled intensity after 3 hours of heat treatment. The ω phase returns to its initial intensity (comparable to volume fraction) after 100 hours. The scale intensity of the β phase is lower after 100 hours than before the heat treatment.

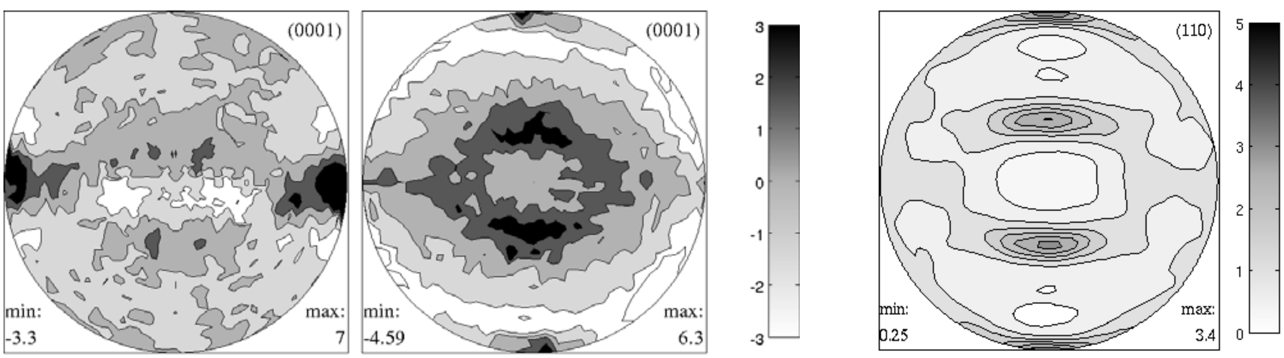

Figure 2 Difference α(0002) pole figures for the samples deformed (left) along the plate rolling direction and (middle) along the plate transverse direction. These pole figures are the difference between the initial α(0002) orientations and the final α(0002) orientations, and thus show the change in texture that occurred as a result of deformation. The difference pole figure for the sample deformed along the transverse direction (middle) is highly correlated to the pole figure for the undeformed β(110) peak (right).