Effect of Fe and O on the $\beta/(\alpha + \beta)$ boundary in Zr-2.5 wt% Nb

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This work proposes to investigate the influence of Fe and O on the $\beta/(\alpha + \beta)$ boundary shift that affects the fraction of α and β phases during extrusion. As principal impurities in Zr-2.5Nb CANDU pressure tube material, Fe and O have opposite effects on the relative volume fractions of alpha and beta phases at the extrusion temperature. Fe, being a beta-phase stabilizer, tends to increase the volume fraction of beta phase at a given extrusion temperature – this is equivalent to increasing the extrusion temperature. Conversely, O, an alpha-phase stabilizer, tends to increase the volume fraction of alpha phase at the extrusion temperature – this is equivalent to lowering the extrusion temperature. Current pressure tube production employs a specification with an Fe content of 900 to 1300 wt ppm, that is higher than that for pressure tubes made between 1987 and 1997, i.e. about 650 ppm Fe. The change in Fe specification can influence the extrusion response of the material and affect the final microstructure that controls the physical properties of the pressure tube. Also the oxygen specification is modified depending on the requirements of the reactor owner because of the empirical relationships between oxygen and deformation that could be related to the microstructure that evolves during extrusion and is a function of the phase boundary structure.

In-situ neutron diffraction experiments were performed on four Zr-2.5wt% Nb billet materials that have various amounts of Fe and O. Typically the samples were heated in a vacuum furnace to a temperature in the β-field, and cooled slowly past the appearance of α-Zr well into the $(\alpha + \beta)$-phase field. The temperature at which the α-Zr first appeared was taken as the $\beta/(\alpha + \beta)$ transus. Results are given in Table 1 below.

The data in Table 1 shows that while the $\beta/(\alpha + \beta)$ transus temperature increases with increase in O content, it increases with Fe content as well, contrary to expectation. It is noted that O may be much more efficient in raising the $\beta/(\alpha + \beta)$ transus temperature than Fe in reducing it, but this still does not explain the apparent difference in the measured transus temperatures for materials A and B above. To resolve the difficulty, we intend to make further measurements on a few samples taken from a batch of micro-pressure tube materials of Zr-2.5Nb that have amounts of Fe varying from 74 to 2950 wt ppm. Because these are samples from thin-walled tubes rather than billets, the O content in the material would be more uniform, so that the effect of Fe can be apparent. An application in this work would be to measure the $\beta/(\alpha + \beta)$ transus temperatures of some Zr-2.5Nb pressure tube specimens that were from the same material but have seemingly different microstructures after extrusion.

Table 1. Measured $\beta/(\alpha + \beta)$ transus temperatures

Material	Iron (wt ppm)	Oxygen (wt%)	$\beta/(\alpha + \beta)$ transus temperature
A	< 11	0.128 ± 0.01	1138 K (865 °C)
B	910 ± 90	0.115 ± 0.008	1166 K (893 °C)
C	2600 ± 300	0.148 ± 0.01	1179 K (906 °C)
D	460 ± 50	0.277 ± 0.018	1291 K (1018 °C)