THE ANISOTROPIC THERMAL CREEP OF PRESSURIZED ZR-2.5NB TUBES

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Abstract – Anisotropic thermal creep of pressurized Zr-2.5Nb tubes is demonstrated. Anisotropic deformation of closed end, internally pressurized tubes (axial stress = _ transverse stress) is manifest by a change in length. The tests reported were carried out using an argon gas protection system designed to avoid the formation of a thick white oxide on the ends of the specimen near the closure weld and allow accurate length measurements. Tubes with three different combinations of microstructure and texture were tested. The results show that tubes with a predominantly radial texture exhibit a relatively high diametral creep strain, and a negative long term axial creep strain after a positive initial transient strain. Tubes with a predominantly transverse texture exhibit a relatively low diametral creep strain and a positive axial creep strain. The anisotropy demonstrated is qualitatively consistent with that exhibited during irradiation creep, and this supports the use of such tests to help understand the anisotropy of in-reactor creep.

1. Introduction

Zr-2.5Nb has been widely used as pressure-tube material in CANDU® reactors because it shows low neutron absorption and excellent resistance to corrosion. (CANDU[®] is the registered trademark of Atomic Energy of Canada Ltd.). In service, cold-worked Zr-2.5Nb pressure tubes are subject to irradiation by fast neutrons, and an internal pressure of about 10MPa which induces a hoop stress of about 130MPa at the operating temperature of about 300°C^[1]. Under these conditions, the pressure tubes undergo dimensional changes in the axial, transverse and radial directions. For example, they elongate several millimeters and their diameter also increases by a few 10^{ths} of a percent a year. These dimensional changes occur as a result of three processes: irradiation growth, which is the shape change at constant volume under no external stress; and thermal and irradiation creep, which lead to a shape change at constant volume due to an applied stress ^[2]. The creep deformation during service is anisotropic and it is related the texture of pressure tubes and the anisotropic HCP single crystal property of _-Zr. Thermal creep denotes the creep behavior in the absence of irradiation. It is intimately related to irradiation creep, and an understanding of irradiation creep in Zr necessitates a thorough understanding of the thermal creep behavior. The research on the thermal creep will provide an inexpensive way to understand the factors that affect the anisotropic deformation, and help determine how to model anisotropic irradiation creep.

Three types of Zr-2.5Nb tubes with different combinations of texture and microstructure were tested under biaxial stress in flowing argon, one type with a predominantly radial texture and two with predominantly transverse textures. The creep strains in both axial and transverse directions are reported.

2. Experimental materials

The experimental capsules were from three types of Zr-2.5Nb tubes which were fabricated by different procedures. The first type is experimental Zr-2.5Nb fuel sheathing (FS) made for use in the WR1 reactor. This material has a predominantly radial texture as shown in Figure 1(a) ^[3]. The other types of tubes were manufactured by a similar procedure to that of standard pressure tubes for CANDU reactors, but with smaller dimensions. They are designated micro-pressure tubes (MPT). Their textures are shown in Figure 1(b) and (c). The one made from a β -quenched billet (MPT 63) has a finer grain structure than the one made from a β -slow cooled billet (MPT 66) as in Figure 2(a) and (b).







(a) (b) Figure 2. TEM microstructure (a) MPT63 and (b) MPT66

3 Experimental procedures

3.1 Thermal creep in air

Prior to the start of creep testing short length of FS material was heated in air for up to 1700 h at 673K, and the thickness of the ZrO_2 layer formed on the outside surface was measured at

intervals by optical metallography, Figure 3. The maximum thickness of oxide formed (7 μ m) represents less than 2% of the wall thickness, and a contribution to apparent strain of 0.05% (diametral) and 0.01% (axial), representing a negligible contribution to either the stress in the capsule, or the measured strain. Creep tests of the FS material were therefore carried out in air (Ref. 4).



Figure 3. Heating time vs. oxidation percent at 673K in air heating atmosphere

In the experiment, a Lindberg tube furnace in Figure 4(a) was used to heat the capsules. As shown in Figure 4(b), the pressurized capsule was placed into the middle part of an open-ended stainless steel sample holder and a thermocouple was inserted into the wall of holder to monitor and record the test temperature. Then the holder with the capsule was put into a stainless tube, Figure 4(a), to be heated in the furnace.



Figure 4. Lindberg tube furnace and sample holder for air atmosphere heating

The creep capsules were heated for time intervals controlled to ensure that the creep strain increment did not exceed 0.2% at each interval. After heating, the capsules were cooled to room temperature by shutting off the furnace and measured using a Z-Mike 1102 Laser Dimensioning System, with an accuracy of 0.004mm in length and 0.001mm in diameter, respectively. The strains reported are relative to the dimensions of the pressurized capsules at room temperature, prior to the first heating cycle.

Because the creep capsule was exposed in air during heating, the surface exhibited a shiny black oxide as shown in Figure 5(a) after 4hrs heating at 623K. After about 38hrs, there were some white oxide appeared on the welded area between the Zr-2.5Nb tube and end caps in Figure 5(b). The amount of the white oxide increased with the exposed heating time in air at Figure 5(c). After 208hrs, the obvious thick white oxide (in Figure 5(d)) appeared on the end cap surface, which produced a significant apparent strain in the axial direction. The remainder of the capsule remained black.



(c) 50hrs (d) 208hrs Figure 5. The oxidation of creep capsules heated in air at 623K (hoop stress=300MPa)

3.2 Thermal creep with argon protection

To get accurate axial creep data, the sample holder was redesigned so the specimens could be exposed to flowing argon gas during testing. The new sample holder design is shown in Figure 6. At one end of the sample holder, a Swagelok fitting is connected, and at the other end a threaded plug allowed the argon to escape. The entire assembly is shown in Figure 7 as the specimen holder is being inserted into the furnace tube. After insertion, the thermocouple A+ and A- would be connected to monitor the temperature and the Swagelok fittings B+ and B- would be connected to transfer the argon gas.



Figure 6. The creep sample holder for argon gas protection



Figure 7. Argon gas protection assembly

The argon gas protect system was set up for six Lindberg tube furnaces and they were connected by the gas transfer stainless steel tube. Hence, six samples could be tested simultaneously. The valve (in Figure 7) controls the gas flow for each furnace. An overview is shown in the Figure 8.



Figure 8. Overview argon gas protect system for thermal creep

The creep experiments were carried out under argon protection using the method described in section 3.1 for the tests in air. Figure 9 shows that a shiny black oxide is maintained at the ends of the specimens and in the weld regions for up to 208h at 623K under argon protection. Figure 10 shows effect of the end-cap oxide on the axial creep strain. Initially there is little difference, but after about 30 hours heating it is clear that the oxidation in air contributes a significant positive contribution to the measured length change.



(c) 156hrs (d) 204hrs Figure 9. The oxidation of creep capsules heated in argon at 623K (hoop stress=300MPa)



Figure 10. Axial creep strains vs. time under the hoop stress of 300MPa at 623K in Argon /Air

4. Results and discussion.

The measured hoop(diametral) and axial creep strains at 623K and 300MPa hoop stress are plotted as functions of creep time in Figure 11(a) and (b) for Fuel Sheathing, MPT63 and MPT66 capsules, respectively. In the hoop direction, the FS capsule shows highest strain, and MPT66 with the coarse grains has the lowest values at all the test periods. In the axial direction, the FS specimen exhibits a positive transient strain, but subsequently a negative long term strain rate. The MPT specimens both exhibit a positive axial transient strain as well as a positive long term rate. However, in truly isotropic materials, the axial strain would be zero for axial stress =1/2 hoop stress ^[5]. Therefore, the experimental results demonstrate that the creep for the tested capsules is anisotropic and that the materials with a radial texture behave differently from the ones with a transverse texture.

The MPT specimens have strong transverse (0002) texture, suggesting that they should be more resistant to diametral (hoop) creep than the FS specimens which have a predominantly radial (0002) texture. However, the MPT63 (finer grains) exhibited substantially higher strain than MPT66 (coarser grains) in both directions in spite of a similar texture and grain morphology.

This suggests an effect of grain size. Although grain size is not normally thought to affect dislocation creep, the sub-micron grain sizes of the MPT materials represent a substantial boundary area to compete with dislocations as sinks and sources for vacancies. Therefore, the finer grain structure of MPT 63 could contribute to a higher creep rate.

Qualitatively the observed anisotropies for the two types of textures are similar to those observed during irradiation creep ^[6]. This supports the use of thermal creep tests to assist understanding the anisotropy of irradiation creep.



Figure 11. Creep time vs. strain (a) hoop (transverse) (b) axial at 623K

5. Conclusion

The design of argon gas protection during experimental thermal creep was described and there is no white oxide appeared on the end caps of pressurized Zr-2.5Nb capsules during the test. The accurate length measurement was obtained. Under the argon protection, the biaxial creep of Zr-2.5Nb tubes with three different combinations of microstructure and texture was investigated at 623K in the hoop stress of 300MPa. The creep strains in both hoop and axial directions demonstrated dependence on texture and microstructure. The anisotropy observed for the different textures is qualitatively the same as that observed during irradiation.

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Reference

[1] R.S.W. Shewfelt, L.W. Lyall and D.D. Godin, Journal of Nuclear Materials, Vol. 125, 1984, p.228.

[2] N. Christodoulou and N. Badie, Thermomechanical Processing of Steels, S. Yue and E. Es-Esadiqi, eds. Conference Proceedings, COM2000, Ottawa, Ontario, Canada, Aug. 2000, p.369.

[3] A.R. Causey, J.E. Elder, R.A. Holt and R.G. Fleck, Zirconium in the Nuclear Industry: 10th International Symposium, ASTM-STP-1245, American Society for Testing and Materials, Philadelphia, 1994, p.202.

[4] W. Li and R.A Holt, Twenty-Sixth Annual Canadian Nuclear Society Conference, Toronto, Ontario, Canada, Jun. 2005.

[5] K.L. Murty and B.L. Adams, Materials Science and Engineering, Vol. 70, 1985, p.169.

[6] R.A. Holt, N. Christodoulou and A.R. Causey, Journal of Nuclear Materials, Vol. 317, 2003, p. 256.