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Experimental observations of void growth in the Zr–2.5Nb pressure tube alloy

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Abstract

Voiding during tensile loading of Zr–2.5Nb pressure tube alloy has been experimentally observed at two temperatures. Tensile specimens cut from the wall of a pre-service pressure tube were tested to various degrees of strain and then sectioned to allow internal voids to be examined. A point counting procedure was used to determine the void volume fraction at various levels of strain. Scanning electron microscopy was completed to determine the degree of voiding, the number and size of voids and the void morphology. Observed voids generally remain small while the overall void fraction increased with increased strain. This suggests that the void formation process in this material is dominated by extensive void nucleation, with little growth of the nucleated voids before void coalescence and final fracture.

1. Introduction

The ductile fracture of metal occurs through the nucleation, growth and coalescence of small internal voids. Numerous studies on void nucleation and growth have been conducted for a variety of metals [1–7]. Thomason [8] proposed the plastic load limit model for ductile fracture. In this model voids are nucleated when the nucleation strain, ε^{n}, is reached. Void growth then occurs until a critical strain, ε^{F}, is reached, at which point void coalescence begins via the internal necking of the material between voids. Although in theory ductile fracture is a three-stage process some materials have been shown to skip, or have very short void growth stages. One example of this is 1090 steel, which was shown to have a very short void growth stage by Le Roy et al. [4]. An abbreviated or missing growth stage can occur when the particle–matrix interface is so strong that plastic flow, which occurs prior to void nucleation, brings the nucleation sites close enough together that once voids form the condition for load limit failure is instantly met with no growth of the voids being required [8]. Coalescence without void growth has also been discussed by Neumann [9], where he suggested that a large local strain field in the necking area between voids could cause the nucleation of a central void between the two previously existing voids, allowing coalescence to occur with little or no void growth.

This paper presents experimental observations of voiding during tensile loading of Zr–2.5Nb at room temperature and at 300 °C, which is the approximate service

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temperature of the pressure tubes. Very little void growth was observed in the specimens, with coalescence occurring a short time after void nucleation was initiated.

2. Material

Atomic Energy of Canada Ltd. (AECL) provided a section of pre-service CANDU pressure tubing material. The tensile specimens were fabricated directly from the wall of a pre-service pressure tube. The alloy has a fine microstructure, consisting of flat elongated α-grains (hcp lattice) surrounded by a boundary layer of the β-phase (bcc lattice) [10], a high density of <*c*>-component dislocations has also been observed in the microstructure of the material [11]. The microstructure is highly textured with the basal planes of the hcp lattice being perpendicular to the transverse direction of the pressure tube [12]. The Zr–2.5Nb alloy has the following chemical composition: Nb: 2.5–2.7 wt%, O: 1100–1200 ppm, Fe: ~1000 ppm, N: <55 ppm, and H: <16 ppm [11].

The tensile specimens had a rectangular cross-section with an area of 8 mm^2 and a gauge length of 12.00 mm. Fig. 1 shows the orientation of the specimens and their dimensions. The tensile axis of the specimen corresponds to the transverse direction of the pressure tube, and is therefore perpendicular to the basal planes of the hcp crystal lattice. The ultimate tensile strength and percentage elongation measured for the specimen geometry shown in Fig. 1 was 880 MPa and 21% at room temperature and 650 MPa and 18% at 300 °C.

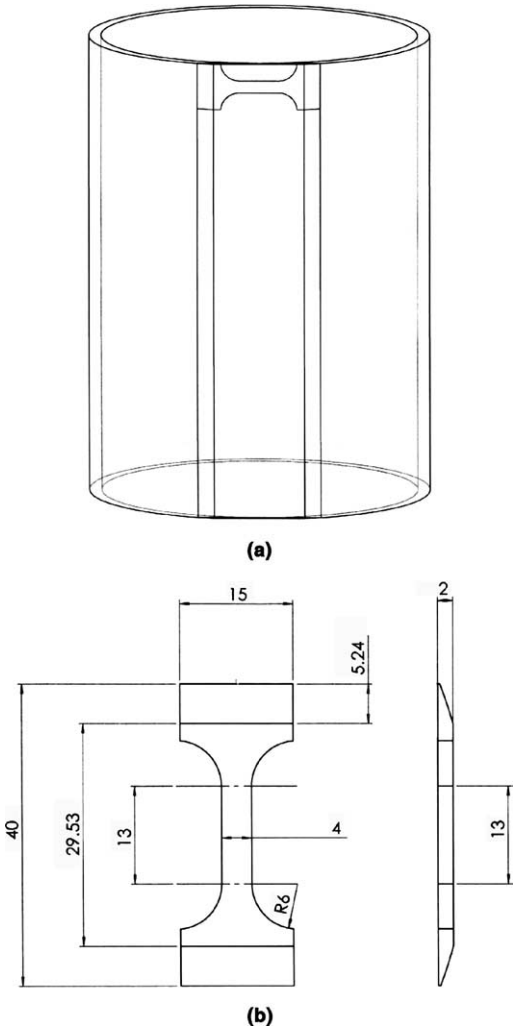

Fig. 1. (a) Specimen orientation on tube, (b) tensile specimen dimensions (mm).

3. Experimental procedure

Tensile testing was carried out on an instron 1137 materials testing machine at a constant cross-head speed of 0.5 mm/min, using a 15 kN load cell. The 300 °C tests were conducted inside a custom furnace attached to the instron machine.

Testing was interrupted prior to fracture, at various elongations in order to obtain specimens which had undergone different amounts of strain.

4. Metallography and stereology

After testing, each specimen was sectioned generally perpendicular to the tensile axis and carefully ground and polished for examination via a scanning electron microscope (SEM). To obtain a suitable finish the final polishing step was a mechanical–chemical attack polish, with chromium oxide powder as the abrasive and an acid solution of 0.5% hydrofluoric acid (48%) in distilled water as the attack agent. The use of an attack polish may have artificially increased the observed void size, however it was unavoidable if a proper finish for examination was to be obtained.

The true strain, ε_t, in each specimen at the plane of polish was determined by the following equation:

$$\varepsilon_t = \ln \frac{A_0}{A}, \tag{1}$$

where A_0 is the original cross-sectional area measured prior to tensile testing and A is the cross-sectional area of the plane of polish after testing.

The void volume fraction, V_v, was determined via a standard point counting procedure [13] for each specimen. The average size of each void observed was determined by measuring the void diameters in two

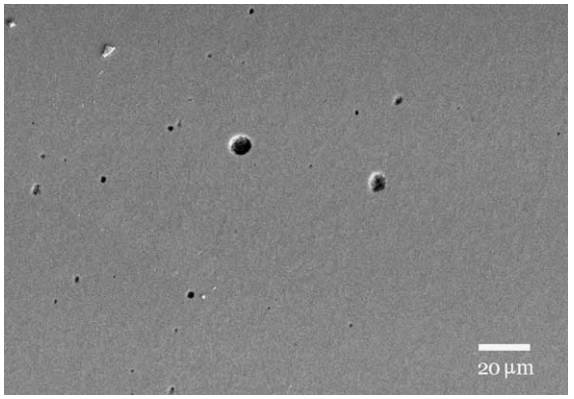

Fig. 2. Typical observation, indicating void size, morphology and density. 300 °C, true strain of 1.111.

directions, generally perpendicular to each other. The average void diameters were used to calculate the geometric mean diameters in each specimen. A geometric mean diameter was chosen because in a particle size analysis it is a better indication of the true average size than an arithmetic mean [13]. A Schwartz–Saltykov diameter analysis [13,14] was also completed to determine the void size distribution in the specimens. Fig. 2 shows a typical observation, indicating the size, morphology and density of voids in the test specimens.

5. Results and discussion

Void nucleation at room temperature only occurred in two specimens, with both of them displaying only a few voids. Neither void volume fraction, nor mean section diameter was determined for either specimen due to a lack of sufficient void numbers.

Voids were observed in six specimens tested at 300 °C, with strains ranging from 0.889 to 1.247. The information from these specimens was used to determine the change in void volume fraction with strain, the void nucleation strain and the void size distribution. Fig. 3 is

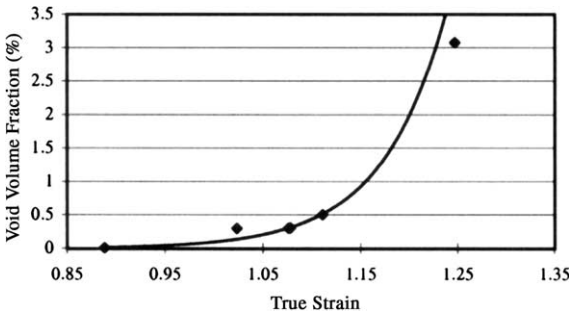

Fig. 3. Void volume fraction in Zr–2.5Nb as a function of true strain at 300 °C.

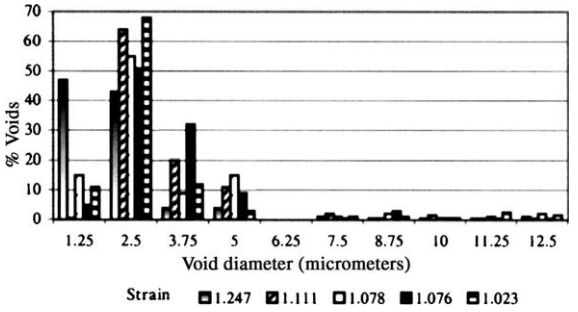

Fig. 4. Void size distribution at various strain levels. Zr–2.5Nb tensile specimens, 300 °C.

a plot of void volume fraction as a function of strain at 300 °C. The volume fraction increases slowly at first, then very rapidly when the specimen approaches the fracture strain. The rapid increase in volume fraction is due to the coalescence of voids [2]. An exponential curve was fitted to the experimental data as a guide to the eye; there is no theoretical justification. The void nucleation strain at 300 °C was determined by an interpolation procedure on Fig. 3. A value of 0.1% for void volume fraction is typically used to determine the approximate nucleation strain [3,4] in tensile tests. A void volume fraction of 0.1% occurs at a true strain of approximately 0.97 for the 300 °C tests, as determined by the exponential curve fit.

As shown in Fig. 3, the range of strain from void nucleation to void coalescence is fairly small, with a range of true strain from 0.97 to approximately 1.25. A nucleation strain of 0.97 is high when compared to such materials as spheriodized steels [4], indicating that the bond strength between the zirconium matrix and the β-phase precipitates is high. The high bond strength leads to a very short void growth stage in the ductile fracture of the material.

The diameter analysis provided more evidence of the limited void growth. Fig. 4 shows a histogram of void diameters calculated by the Schwartz–Saltykov diameter analysis, for various levels of strain. At the strain levels examined, the vast majority of voids had an approximate diameter of 2.5 μm. It is interesting to note that the specimen with the largest strain also had the largest percentage of small voids (diameters of 1.25 μm). This is most likely due to the fact that in the diameter analysis a large central void was not included, as it appeared to be the result of the coalescence of many smaller voids.

Acknowledgements

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Lockley of AECL demonstrated specimen preparation techniques for Zr–2.5Nb.

References

[1] M.J. Worswick, R.J. Pick, J. Appl. Mech. 58 (1991) 631.
[2] D. Kwon, R.J. Asaro, Metall. Trans. A 21 (1990) 117.
[3] J.B. Kosco, D.A. Koss, Metall. Trans. A 24 (1993) 681.
[4] G. Le Roy, J.D. Embury, G. Edward, M.F. Ashby, Acta Metall. 29 (1981) 1509.
[5] T. Pardoen, I. Doghri, F. Delannay, Acta Mater. 46 (2) (1998) 541.
[6] D. Watson, M.R. Warren, C.J. Beevers, Can. Metall. Quart. 11 (1) (1972) 53.
[7] J. Crepin, T. Bretheau, D. Caldemaison, Acta Mater. 44 (12) (1996) 4927.
[8] P.F. Thomason, Ductile Fracture of Metals, Pergamon, Toronto, Canada, 1990.
[9] P. Neumann, Mater. Sci. Eng. 25 (1976) 217.
[10] J.R. Theaker, R. Choubey, G.D. Moan, S.A. Aldridge, L. Davis, R.A. Graham, C.E. Coleman, ASTM STP 1245 (1994) 221.
[11] V. Perovic, A. Perovic, G.C. Weatherly, L.M. Brown, G.R. Purdy, R.G. Fleck, R.A. Holt, J. Nucl. Mater. 205 (1993) 251.
[12] D.D. Radford, M.J. Worswick, J. Phys. IV. 10 (2000) 293.
[13] E. Ervin, Underwood, Quantitative Stereology, Addison-Wesley Publishing Company, Reading Massachusetts, 1970.
[14] R.T. DeHoff, F.N. Rhines (Eds.), Quantitative Microscopy, McGraw-Hill Book Company, New York, 1968.