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Mechanisms of void formation during uniaxial tensile testing in a low-temperature-aged U-Nb alloy

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ABSTRACT

The microstructure and damage accumulation of a U-5.5Nb alloy aged at a low temperature were analysed as a function of strain. Four tensile tests were performed and interrupted at different strain levels to investigate the mechanisms of void nucleation, growth and coalescence via optical microscopy, scanning electron microscopy with energy dispersive X-ray spectroscopy, X-ray diffraction and nanoindentation measurements. The results show that voids first nucleate at U(N,C) inclusions at a low strain because they crack due to their low strength. Meanwhile, the higher strength of the Nb_2C inclusion than that of matrix does not lead to the fracturing of Nb_2C inclusions. However, with the further increasing strain, voids nucleate via the decohesion of the Nb_2C-matrix interface because of the mismatch between the strengths of the Nb_2C inclusion and matrix. In clusters comprising both Nb_2C and U(N,C) inclusions, void formation mechanisms are determined by the Nb_2C and U(N,C) inclusions. In addition, in clusters, the weak adhesion between Nb_2C and U(N,C) inclusions causes separation. Based on experimental observations, an illustration of failure mechanisms is presented for a low-temperature-aged U-5.5Nb alloy with a single α″ phase.

1. Introduction

Uranium is widely used in the nuclear industry, especially in nuclear power plants, because of its high density and unique nuclear properties. However, its poor corrosion resistance limits its further application. Adding alloy elements to uranium is an effective way to increase its corrosion resistance [1,2]. In industrial applications, Nb is always used, as U-Nb alloys can provide a good combination of strength, ductility and corrosion resistance when an alloy with a homogeneous distribution of Nb is quenched from the high-temperature γ-phase zone [3,4]. To further enhance the yield strength with only a slight loss in ductility and almost no effects on corrosion resistance, the as-quenched U-Nb alloy can be subject to low-temperature ageing (< 250 °C) [5,6]. For example, a low-temperature (200 °C for 100 min)-aged U-5.6Nb alloy [5] exhibited a yield strength of 312 MPa, ultimate strength of 827 MPa and fracture strain of 24.9% in contrast to corresponding values of the as-quenched alloy of 112 MPa, 747 MPa and 21%, respectively [5,7].

When the cooling rate is above 20 K/s [4], high-temperature γ-phase U-Nb alloys transform into different martensite phases (α′, α″ or γ°) depending on Nb contents [8]. As-quenched and subsequently low-temperature-aged U-6Nb alloys with α″ monoclinic phases have attracted much more attention for their excellent corrosion resistance and mechanical properties. α″ U-Nb alloys exhibit a very complex "double yield" deformation mechanism during uniaxial tensile testing. After elastic strain, the alloys begin to deform by twinning, followed by de-twinning, and finally fracturing via the dislocation-slip mechanism [9]. Several studies have discussed the twinning deformation mechanism [10–14]. Field et al. [13] found that during the twinning deformation stage with initial strain of 3%, the main deformation mechanism involves the motion of $\{\bar{1}72\}$ twins, which can move freely through finely twinned regions under low levels of twinning strain. At higher strains between 3% and 6%, fine twins associated with the martensitic transformation structure are no longer observed due to de-twinning, as defined by Sun and Bridges [9], and the proposed dominant mechanism involves the cooperative migration of fine $\{\bar{1}30\}$ and crossing $\{\bar{1}72\}$ twin boundaries to eliminate fine twins [13]. With a further increase in strain, a diffuse peak occurs between 6% and 10% strain, and work hardening via the dislocation-slip mechanism at strain levels of over 10% dominates final deformation until fracturing. However, most of the literature has focused on twinning deformation with strain of less than 7% [10,11,13,15,16] while little is known of irreversible deformation processes that follow twinning deformation and final fracturing, which account for more than 60% of the total strain.

The ductile fracturing process involves void nucleation, growth and

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coalescence [17,18]. Void nucleation mechanisms have been of particular interest as they can be applied to ductile failure models to predict damage behaviours. Voids can nucleate at second-phase particles through the decohesion of the particle-matrix interface or particle fracturing [19,20]. In addition, although as-quenched and low-temperature-aged U-Nb alloys show a single-phase matrix, inclusions, which cannot be avoided during high-temperature metallurgical processes, can act as void nucleation sites [21]. Carbon is the predominant impurity found in U-Nb alloys, and primary carbides found in U-Nb alloys include U(N,C), Nb_2C and clusters derived from aggregates of U(N,C) and Nb_2C [22]. Inclusions, especially large ones, are known to decrease the elongation and fatigue strength of materials due to the presence of preferential sites for cracking [23]. In α-U [24] and U-Cr [25] alloys, tensile failure is initiated through the decohesion of the UC-matrix interface or the prior cracking of UC, followed by the propagation of cracks through the surrounding matrix. In U-Nb alloys [3,5], typical ductile fractures initiate void nucleation at the inclusion sites (UC or Nb_2C), which can be observed after fracturing in the dimples. Furthermore, predominant sites of fatigue crack initiation in U-6Nb alloys have been identified as clusters of niobium carbide and uranium oxide inclusions [26]. Although the primary inclusion types in U-Nb alloy are clear, the different types of void nucleation and the subsequent void evolution mechanisms have not been sufficiently investigated. As concluded by other investigators [17], void nucleation depends on the shape and size of particles, the stress state and the distribution and mechanical properties of the inclusion itself. In our previous study [21,27] the mechanical properties of Nb_2C and U(N,C) inclusions and matrixes were determined from nanoindentation measurements. The results showed that Nb_2C inclusions were harder and stronger than U(N,C) and the matrix, which generates voids due to debonding at the interface with the matrix. Meanwhile, U(N,C) inclusions are softer and weaker but present high plasticity; thus, they are inclined to produce voids through cracking. However, void formation and damage evolution mechanisms of different deformation stages in U-Nb alloys with complex “double yield” deformation effects are poorly understood thus far.

The present study investigates the process of failure in low-temperature-aged (200 °C) U-5.5Nb alloys and observes the evolution of failure during different deformation stages of the alloy with a particular focus on void nucleation mechanisms. Therefore, to observe damage patterns at different strain levels, uniaxial tensile tests of the specimen were interrupted at specific deformation stages: (1) during twinning deformation; (2) after twinning deformation and before deformation controlled by dislocation slipping; (3) dislocation-slip deformation; and (4) after failure. In addition, we attempted to illustrate the roles of different inclusions on damage nucleation and development. Damage accumulation is characterized by a metallographic analysis and scanning electron microscopy (SEM). Nanoindentation was also performed to interpret probable reasons underlying different void nucleation mechanisms due to differences between the mechanical properties of the inclusion and matrix.

In situ SEM images are always recorded in studies on microstructural behaviour [28,29]. The results obtained from a small aggregate are extended to the entire structure and thus suffer from limited statistical representation [30]. In this study we focus on statistical results, which represent the entire aggregate. Therefore, in situ SEM images were not recorded during our investigation of damage accumulation trends.

2. Experimental

An alloy ingot of the U-5.5Nb alloy with the nominal composition (by weight percent) was prepared by vacuum-induced melting. The chemical composition of the ingot is listed in Table 1. Carbon and nitrogen were the primary impurity elements, which were taken up by the molten alloy during high-temperature metallurgical processes. The ingot was heat treated at 1100 °C for 10 h in a vacuum environment to homogeneously distribute Nb, followed by furnace cooling. The material was then held at 850 °C for 2 h under vacuum prior to water quenching. The as-quenched sample was directly aged at 200 °C for 2 h to increase the yield strength. Tensile specimens were machined from the aged samples with a gauge length of 25 mm and diameter of 5 mm.

Table 1
Chemical composition of the investigated U-5.5Nb alloy (in wt%).

C	N	B	S	Mg	Ti	Cr	Ca	Zn	Nb
150	60	≤ 20	≤ 20	≤ 20	≤ 20	≤ 20	≤ 20	≤ 20	5.46

A uniaxial tensile test was performed at a cross-head speed of 1.5 mm/min at room temperature on a servo-hydraulic MTS machine with a 100 kN load-cell capacity. To investigate processes of void nucleation and growth in greater detail, three tensile tests were interrupted at different engineering strains, i.e., ε = 2%, 6.6% and 16%, and one tensile test specimen was strained to the point of fracturing. During the tensile process, an extensometer set to a gauge length of 25 mm was used to measure the strain and to enable interrupting the test at different strains corresponding to different deformation stages.

As-received samples were cold mounted and roughly ground using SiC waterproof abrasive paper of an increasingly fine grade to a P2000 grit. The samples were then polished using a 1.5 mm diamond abrasive attached to a fine-polishing cloth. Microstructural morphologies were imaged by Olympus (DSX-500) optical microscopy (OM) after electroetching with 10% oxalic water solution at 2 V for 8 s. A mean linear intercept method was used to measure the grain size, with each sample including at least 200 grains. The phase composition of the aged sample was analysed via X-ray diffraction (XRD) (Empyrean, PANalytical, Holland) with Cu Kα radiation (λ = 0.1540598 nm) at 40 kV. Scans were collected over a 2θ range of 30–70° with a step of 0.01°.

A metallographic analysis of damage accumulation along the gauge length was performed on interrupted and fractured samples. All samples were machined along the longitudinal cross-section using wire electrical discharge machining to preserve damage. The amount of damage was recorded by OM in both polished and electroetched conditions using the same sample treatment method described above. More information on small voids and void nucleation sites of the etched samples were verified by scanning electron microscopy (SEM) (Helios Nanolab 600i) and energy dispersion spectroscopy (EDS). In addition, micro- and macroscale morphologies of the fracture surface of the failed samples were observed with SEM (model KYKY-EM3200).

The voids after fracturing were quantitatively characterized based on void area fraction, number density, size and aspect ratio. First, a low-magnification optical image of the fractured sample was recorded and imported into the image analysis software (Image Pro Plus). The initial diameter of the un-deformed gauge section was measured using a Vernier calliper and used to calibrate the image of the necked region. Then, the diameters of the fractured sample were measured as a function of the distance away from the fractured surface and used to calculate the strain profile in the neck, as suggested by Poruks et al. [20,31] (Fig. 1). The true strain (ε_{eq}) was calculated at approximately 500 μm intervals from the fracture surface from the measured sample diameter based on Eq. (1) [32]:

$$\varepsilon_{eq} = -2 \ln \frac{D_f}{D_i} \tag{1}$$

where D_i and D_f are the initial and final sample diameters, respectively, as schematically shown in Fig. 1. The void area fraction, number density, size and aspect ratio are correlated with the local ε_{eq}.

Mechanical properties of the inclusions and matrix were measured by nanoindentation to interpret inconsistent void nucleation mechanisms likely present at the sites of different inclusions. A nano-mechanical test instrument (Hysitron TI-950 US) equipped with Berkovich tip