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Micro-mechanical characterisation of uranium

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A B S T R A C T

This paper describes a study measuring the mechanical properties of cast uranium (U) and wrought U–6Nb using micro-indentation. Load-depth curves were generated, which were used to obtain data such as elastic modulus as well as hardness. This work has shown reasonable agreement between the experimental results acquired as part of this study and experimentally-derived mechanical properties obtained via other mechanical tests. However, a wide scatter in the data is evident, thereby necessitating a large number of indents on each specimen in order to ensure confidence in the values obtained.

1. Introduction

The development of depth-sensing indenters over the last two decades has enabled other mechanical properties in addition to hardness to be measured, for example elastic modulus. This has enabled the measurement of mechanical properties of components that cannot be tested using more conventional procedures owing to their size or shape. These depth-sensing indentation tests have been used on both the micro- and nano-scale to measure the properties of both bulk materials and coatings [1–6]. Another advantage of this test is that it enables the determination of the elastic modulus of materials that exhibit unconventional stress–strain behaviour. Two such materials that fall into this category are uranium (U) and uranium–niobium (U–Nb) alloys.

Unalloyed uranium has a room temperature strength that is similar to mild steel, albeit with considerably lower ductility and toughness [7]. However, the measurement of its elastic modulus can be problematic owing to its poorly defined proportional limit and apparent curvature of the stress–strain curve at very low stresses [8]. In the case of uranium–niobium (U–Nb) alloys, the addition of Nb results in a lower elastic modulus, together with a concomitant increase in ductility relative to unalloyed uranium [9] as well as increasing its oxidation and corrosion resistance. Nb is highly soluble in the high temperature gamma phase, although this is not maintained in the alpha phase at room temperature. In order to overcome this, the alloy can be subjected to a water quench resulting in a martensitic structure, which consists of packets of twin-related parallel plates with each individual plate also containing a profusion of internal twins. This structure, known as 'banded martensite' [7], confers on the alloy a softer and more ductile character than its unalloyed counterpart. For U–6 wt% Nb, the effect of the martensite on the structure is to distort, by 2–3°, the lattice angle from 90°, causing a slight crystal structure change from orthorhombic to monoclinic, which is designated α'' [9]. U–6Nb is metastable and hardens with age as the softer supersaturated α'' phase decomposes to a more thermodynamically stable structure, eventually reaching a final $\alpha + \gamma_2$ equilibrium. Indentation has been used to evaluate the increases in hardness with ageing time and temperature of U–6Nb [10]. Like unalloyed uranium, U–6Nb also exhibits unusual stress–strain behaviour, which is manifested in the form of double yielding [11].

Although the properties of uranium have been well characterised in the past, it is instructive to repeat these measurements using new test techniques as they become available. Moreover, changes to the manufacturing processes may result in different property values, which may render data obtained in the past unrepresentative of current material. For these reasons, the present study was undertaken to measure the mechanical properties of uranium and U–6Nb using micro-indentation.

2. Experimental details

The materials tested were cast uranium and wrought U–6 wt% Nb; micrographs of both materials can be seen in Fig. 1. The U specimens contained no significant alloying elements, the most abundant impurity being carbon, the concentration of which was 390 ppm. The wrought U–6Nb specimens were water-quenched and aged at ambient temperature for approximately 20 years prior to the indentation tests. Uranium specimens were free-standing with a ground surface finish while the U–6Nb specimens were in the form of polished metallographic specimens.

The indentation tests were carried out using a CSM MHT micro-indenter, which was equipped with a Vickers (four-sided diamond pyramid) indenter. The maximum loads were between 5 and 20 N and were applied at a rate of between 20 and 40 N min^{-1}; in each test the maximum load was maintained for 15 s before being

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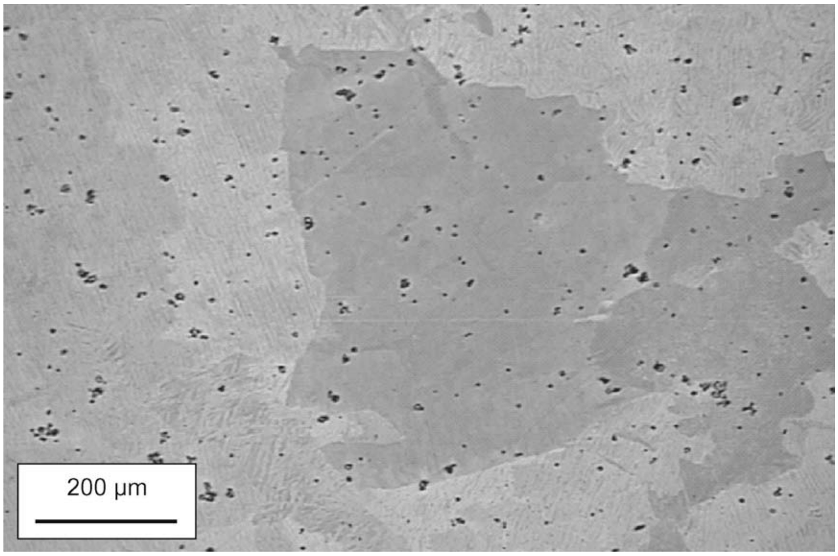

Fig. 1a. Optical micrograph of cast uranium. The black particulate features are uranium carbides.

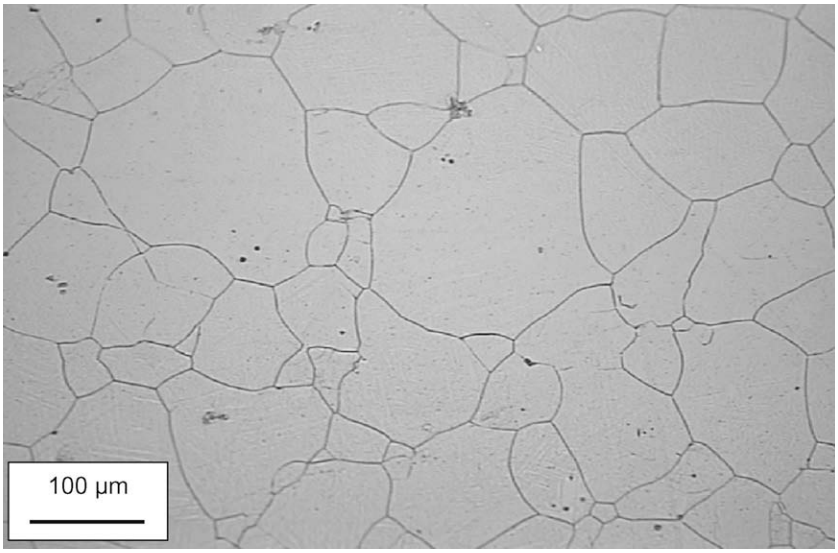

Fig. 1b. Optical micrograph of wrought U–6Nb.

released at the same rate. During the test, the load and depth were continuously recorded to generate loading and unloading curves. The indents were made in lines and were typically spaced 500 μm apart. This distance far exceeds the minimum indent spacing (generally three times the indent diagonal) required to ensure no influence on hardness values from adjacent indents.

The resultant load-depth curves were used to obtain the hardness (H) and reduced, or contact, modulus (E_r) by employing the procedure of Oliver and Pharr [12]. E_r was then used, together with the Poisson's ratio (v) to calculate the elastic modulus (E) in the following way:

$$\frac{1}{E_r} = \frac{1 - v^2}{E} + \frac{1 - v_i^2}{E_i}. \tag{1}$$

In the present study, the following values were used for the diamond indenter: $E_i = 1140$ GPa and $v_i = 0.07$. The Poisson's ratios used for U and U–6Nb were 0.21 and 0.35, respectively. In the case of unalloyed U, the Poisson's ratio is known to be dependent on crystallographic orientation, which may affect the values of elastic modulus when converting from reduced modulus. In the present study, the aggregate value of 0.21 [13], the figure for randomly oriented polycrystalline material, has been used.

The elastic modulus values obtained from the indentation tests were compared with data obtained from tensile testing of specimens machined from the same batches of material. The specimens were tested using a Zwick Z050 tensile test machine in accordance with the standard BS EN 10002-1 (2001). The specimen extension was measured using an Epsilon Static Extensometer with a gauge length of 10 mm.

3. Results and discussion

3.1. Hardness and elastic modulus

The results of the indentation measurements are summarised in Table 1; to aid comparison, the mean and standard deviations of elastic modulus values obtained from tensile tests of the same materials are also listed. Typical load-depth curves of both

Table 1
Mechanical properties ±1 standard deviation obtained from indentation measurements of U and U–6Nb using a maximum load of 10 N and a dwell time at maximum load of 15 s. The range of elastic modulus values obtained from tensile tests of the same material are also listed for comparison.

Mechanical property	U	U–6Nb
Mean hardness, HV	289 ± 31	205 ± 30
Mean contact modulus E_r (GPa)	190 ± 76	54 ± 9
Mean elastic modulus, E (GPa)	226 ± 108	51 ± 10
Elastic modulus from tensile test (GPa)	215 ± 41	62 ± 9

materials are shown in Fig. 2. It can be seen that the responses of the materials are predominantly plastic, although some elastic recovery on release of the load can also be seen. In both figures a small plateau can be seen at maximum load, which is indicative of creep of the material. Such behaviour is often observed in ambient temperature indentation tests and has been studied in detail by other workers [14,15]. In the present study, the indentation creep, C_{IT}, which is the relative change of the indentation depth at constant load, was quantified using the following expression:

$$C_{IT} = \frac{h_2 - h_1}{h_1} \times 100, \qquad (2)$$

where h_1 is the depth when the maximum load is first reached and h_2 the depth at the end of the dwell time (i.e. just prior to release of the load). C_{IT} is expressed as a percentage and enables the indentation creep behaviour of uranium to be compared with other materials tested under the same conditions. Using Eq. (2) the C_{IT} values for uranium were between 2.98 and 3.49%, which is approximately comparable to other metals such as tantalum (2.34–4.07%) and zirconium (2.56–3.29%). However, further work is required to examine this behaviour in more detail.

For both uranium and U–6Nb the results show a large scatter, which is thought to be due to the highly anisotropic nature of uranium, which results in its mechanical properties being markedly affected by orientation of individual grains. Nevertheless, the mean values are within the same region as those quoted by other workers. This finding underscores the importance of conducting a large number of tests in order to ensure high statistical confidence in the data. This is particularly pertinent in indentation tests, owing to the localised nature of the stress field associated with individual indents.

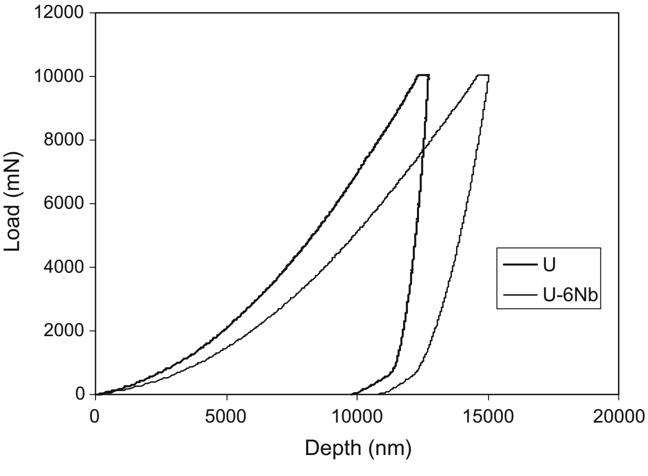

Fig. 2. Load-depth curves from the indentation of cast uranium and wrought U–6Nb. The hardness of the cast uranium was 266 HV and the indentation modulus (E_r) was 184 GPa, which corresponds to an elastic modulus (E) of 211 GPa. The hardness of U–6Nb was 213 HV and the indentation modulus (E_r) was 62 GPa, which corresponds to an elastic modulus (E) of 60 GPa.

In the case of the unalloyed uranium the mean hardness (289 HV) is close to other values given in the literature for cast uranium, for example [16], which quotes a hardness of 280 HV. Less agreement was observed in the elastic modulus values, where the mean value from the present study (226 GPa) is significantly higher than [16], in which a figure of 176 GPa was quoted. However, in tensile tests of material taken from the same batch as used in the present study the elastic modulus values recorded were between 191 and 263 GPa. Furthermore, no details were given in [16] as to the history of the specimen from which the figure of 176 GPa was obtained. Such information is particularly important in the case of uranium as its mechanical properties are dependent on factors such as grain size, grain orientation, processing conditions and impurity content [8]. As an example, the tensile strength of as-cast uranium has been observed to increase from 358 to 510 MPa as the carbon content is increased from 60 to 1250 ppm [17]. In the case of the present cast uranium material tested, the grain size was between 500 and 750 μm, while the carbon content was 390 ppm. In addition to carbon content, the cooling rate during solidification also exerts an influence on the mechanical properties. In castings subjected to fast cooling the carbon is retained in solid solution; however, slow cooling leads to formation of carbides (which can be seen in Fig. 1(a)) and loss of carbon from solid solution. These factors are thought to contribute to the higher values of hardness measured in the present study, some of which were as high as 320 HV. The effect of carbon content and grain size on elastic modulus is less clear. Nevertheless, despite the difficulties in measuring the elastic modulus caused by the indistinct elastic region of the stress–strain curves of many tensile tests of uranium, it is encouraging that the figures obtained via indentation are within the same range as those obtained by more conventional mechanical tests.

In tests of U–6Nb, the hardness values were close to other values recorded for this material, although care is necessary in such comparisons owing to the age hardening behaviour of this material. The elastic modulus values obtained from the indentation tests ranged from 48 to 61 GPa with an average of 51 GPa, which are within the same range as those measured from tensile tests (41–83 GPa). An example of a stress–strain curve from the same batch of material can be seen in Fig. 3. In that particular test an elastic modulus of 62 GPa was recorded, which was obtained from the straight-line portion of the curve prior to the first yield point, which occurred at a stress of approximately 120 MPa.

3.2. Variation of properties with depth

Another methodology that can be carried out using depth-sensing indentation is the load/partial unload technique. This involves

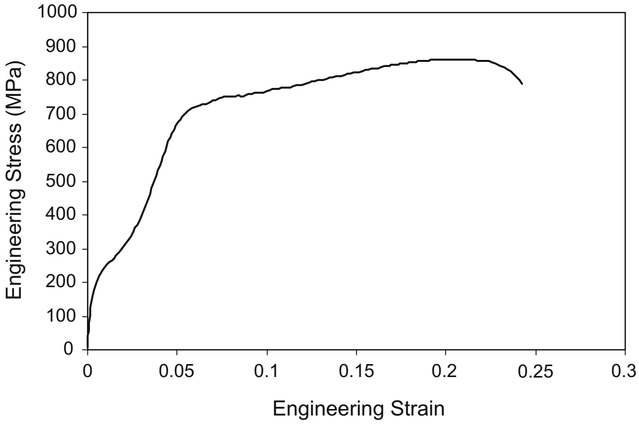

Fig. 3. Typical stress–strain curve from the tensile test of wrought U–6Nb. The elastic modulus from this test was 62 GPa.

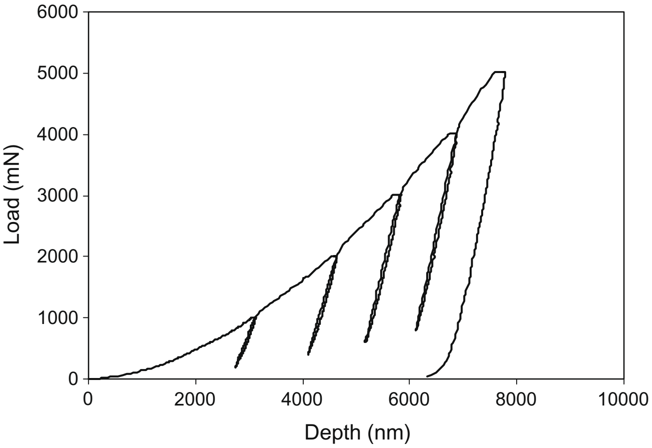

Fig. 4. Load-depth curve for the load/partial unload indentation test of cast uranium.

Table 2
Variation in mechanical properties with depth measured from the load/partial unload indentation of cast uranium.

Load (mN)	Depth (nm)	Hardness (HV)	E_r (GPa)	E (GPa)
1003	3120	486	179	203
2005	4630	442	165	184
3006	5837	424	150	165
4018	6882	400	149	164
5021	7796	386	145	159

the loading of the indenter and partially unloading it before reloading at the same location to a greater depth. This procedure can be repeated several times (the exact number is stipulated by the operator) to obtain a depth profile of the elastic properties of the material in the near-surface region.

Fig. 4 shows a load-depth graph for a multi-cycle indentation of unalloyed uranium. In this test the specimen was subjected to five cycles to loads of between 1 and 5 N. During each cycle, the load was held for 15 s before release to 20% of the load in order to measure the hardness and elastic modulus from the unloading curves. The resultant data is listed in Table 2 to show the hardness and elastic modulus as a function of maximum depth attained during each indentation cycle. It shows that the hardness values at the smallest depths are significantly higher than those recorded above. This was attributed to the presence of an oxide layer on the surface of the specimen. Indeed, at the shallowest depth (3120 nm) the hardness recorded (486 HV) is of a similar order to those recorded by Yamada et al. [18], the mean values of which were approximately 640 HV (25 g load) and 460 HV (1 kg load) for UO_2 specimens with porosities of 0% and 14%, respectively.

4. Conclusions

This study has shown that depth-sensing micro-indentation can be used to measure the mechanical properties (hardness and elastic modulus) of U and U–6Nb. The hardness values measured are within the range previously measured using other tests. Moreover, the elastic modulus values of the U and U–6Nb obtained from the unloading curves agree well with other values from the literature as well as those measured from tensile tests.

Depth-sensing micro-indentation enables mechanical properties such as elastic modulus to be obtained without the need for time-consuming machining operations to produce tensile test specimens. It also assists in determining the elastic modulus for materials that exhibit unconventional mechanical behaviour, in particular those where the elastic region of the stress–strain curve may be hard to define.

In analysing the results, it should be remembered that the stress fields in indentation tests are more localised than more conventional mechanical tests. For this reason the variation in properties must be properly understood: this is particularly important for materials such as uranium, in which the properties are highly dependent on crystallographic orientation.

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