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Effects of heat treatment on grain refinement in cast uranium–0.25 wt% vanadium alloy

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Abstract

The feasibility of refining the grain size of large castings of uranium–0.25 wt% vanadium alloy has been successfully demonstrated using a helium-mediated 'isothermal' heat treatment in a large commercial vacuum furnace. Using this method, the grains of 10–20 kg hemispherical castings were refined from 180 μm diameter to 35 μm, a reduction similar to that achieved in both laboratory-scale isothermally transformed material and carefully controlled wrought-processed uranium. The results of compression and tension tests on the cast alloy and the wrought uranium at a strain rate of 10^{-3} s^{-1} indicate that up to a strain of 30%, the cast and heat-treated uranium–0.25% vanadium alloy exhibits a higher compressive flow stress than that of comparable wrought uranium. In tension, the cast and heat treated alloy continues to exhibit high strengths, but displays a lower ductility.

1. Introduction

Wrought uranium (U) is frequently used in applications that require finer grain size, greater ductility and higher strength than can be obtained with cast materials. The fine (20 μm), equiaxed grains characteristic of wrought material are produced by repeated mechanical working (usually rolling) and recrystallization heat treatments. In addition to this processing, additional fabrication steps are needed to produce a finished part. The direct casting of near-net-shape pats is an alternative fabrication process that has potential to reduce the high costs and waste associated with the preparation and finishing of wrought U. Castings, however, typically have very large grains (hundreds of microns to several millimeters in size), which can reduce their strength and ductility relative to the wrought material and can lead to inhomogeneous deformation. To take advantage of near-net-shape casting as a substitute for wrought processing, a means of controlling the grain size through heat-treatment and alloying is therefore necessary.

For the grain refinement of cast unalloyed U, one well-documented approach is the beta (β) quench-alpha (α) anneal heat treatment [1,2]. During a rapid quench from the β phase to the α phase, adequate residual strain can be induced in α-U to cause recrystallization when the material is subsequently reheated to a range of temperatures between 580–640°C. This residual strain has been attributed to a very large anisotropy in the linear thermal expansion coefficient at high temperatures [3] and to a martensitic-type transformation [4]. Its effect is typically seen in extensively twinned α grains. The grains produced by β quench-α anneal treatment tend to be finer and more equiaxed than cast material. By executing this procedure twice (a 'double β heat treatment'), a final grain size of 120 μm can be achieved. Although this is still significantly larger than that of a wrought material, it is much smaller than that of as-cast unalloyed U, which can have grains as large as 6 mm.

An alternative approach makes use of vanadium (V) as a grain refiner which is added in very small amounts to cast uranium. Reisse et al. [5], using U–0.2 wt% V alloy, correlated the effects of cooling rates and isothermal transformation temperatures with the microstructure. They equilibrated small cylindrical specimens at 720°C, and then quenched them to an isothermal annealing temperature by plunging them into a bath of liquid lead–bismuth. From this experiment, they derived a time–temperature–transformation (TTT) diagram for

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the U–0.2 wt% V alloy (Fig. 1), and found that the nose of the transformation-time curve (point of maximum transformation rate) occurs at 560°C and 15 s. Jepson [6] also showed that by increasing the vanadium content to 0.43 wt%, the nose of the transformation curve shifts to about 575°C and 25 s, thereby allowing adequate time for cooling the alloy to a lower isothermal transformation temperature. The vanadium in solid solution apparently delays the β-to-α transformation, relative to that of the unalloyed uranium [7].

For the U–0.2 wt% V alloy, the best grain refinement occurred when the specimens were cooled from 720°C to 516°C within 15 s, and held there for about 3 h. This treatment places the material into a predominantly nucleation-driven region of the TTT diagram: many α nuclei are produced and their subsequent growth produces very fine grains. The resulting grain size after the isothermal transformation was about 20 μm, close to that of wrought processed U.

While these studies clearly prove the feasibility of using cast and isothermally heat treated U–V alloys to match the grain size of wrought U, a drawback to this method is that a scaling-up to heat treating large parts (hundreds of mm in size) is difficult. Rapidly moving a large part from one zone of a furnace at 720°C (the β region) to α second zone at the isothermal transformation temperature of about 520°C (the α region) is cumbersome. It is therefore very difficult to execute a true ‘quench’ from one temperature to the other. As a result, Reisse [5] and Wood [7] attempted to refine the grains of cast U–V alloys by using a continuous, controlled-cool argon-mediated gas ‘quench’. They found in small samples that a cooling rate of 10°C/min was sufficient to produce 70–90 μm grains.

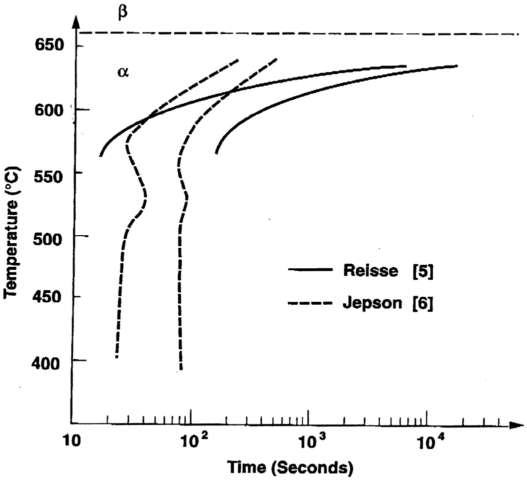

Fig. 1. A time–temperature-transformation diagram for the U–0.2 wt% V alloy containing 70 wppm carbon.

In order to maximize the grain refinement potential of V in large cast parts, two aspects of the isothermal heat treatment are critical. One is the cooling rate; the other is the stabilizing isothermal temperature. Heat transfer calculations and heat treatment studies with a 2-mm thick uranium–6% niobium alloy part showed that a cooling rate of about 160°C/min can be achieved by using helium-gas, rather than argon, as the cooling medium in a conventional vacuum furnace [8]. Although this binary alloy part is much thinner than the cast parts, the high thermal conductivity of helium relative to argon produces more rapid cooling. As a result, helium can be used more effectively than argon in cooling a large part from 720°C to a temperature close to the isothermal annealing temperature. If sufficient grain refinement could be achieved, this scheme could be employed to heat-treat large castings in typical industrial heat-treatment furnaces. A caveat to a modified isothermal heat-treatment, such as we purpose, is being able to prevent undercooling below 510°C. As reported by Reisse [5], cast U–0.2 wt% V coupons that were quenched below 510°C reveal an undesirable acicular structure, attributed to a martensitic transformation. Although addition of 0.25 wt% V rather than 0.2 wt% may lower the martensitic transformation start (M_s) temperature slightly, a minimum undercooling temperature of 510°C was selected to avoid this transformation.

The purposes of the present study are: (1) to demonstrate the feasibility of refining the grain size of 10–20 kg castings of a U–0.25 wt% V alloy using a helium-mediated ‘isothermal’ heat treatment in a large commercial vacuum furnace, and (2) to compare the microstructures and mechanical properties of the cast material heat-treated in helium to those of the wrought material, and to cast materials that have been isothermally heat-treated and/or heat-treated in an argon-atmosphere.

2. Experimental procedures

Master alloy ingots containing about 4.5 wt% V (the eutectic composition) were prepared in an electron-beam furnace. A suitable amount of this master alloy was then added to the uranium charge material to yield a composition of about 0.25 wt% in the final casting. The charge was melted in an yttrium-oxide coated, bottom-pour graphite crucible and held at (or above) 1390°C for approximately 10 min before pouring in order to allow slag particles to float to the top of the melt. The liquid alloy was poured into an yttrium-oxide coated graphite mold, which had been pre-heated to 1000°C at its top and about 850°C at its bottom. The vacuum level of the casting was 1 Pa or better at pour time.

Two castings were prepared for this study. The first casting was a 20 kg, thick-walled (30 mm) hemisphere, which provided material for heat-treatment coupons used to develop the heat-treatment method. These heat-treated coupons were subsequently used for a welding study [9]. The second casting was a 16 kg, thin-walled (10 mm) hemisphere, which was directly heat-treated. Hemispherical castings were selected for two reasons: (1) they are easy to handle in the foundry and the heat-treatment furnace and (2) they are known to produce sound castings. These hemispherical ingots were machined to remove surface defects before their heat treatment. The vanadium, carbon and hydrogen contents of the castings are given in Table 1.

Fig. 2 shows a schematic sketch of the experimental set-up, arranged inside a large commercial vacuum heat-treatment furnace. To position the samples at the center of the vacuum furnace, a 350-mm diameter, 610-mm long, and 6.35-mm thick stainless steel cylinder, open on both ends, was vertically placed on the furnace floor. A 360-mm diameter copper hemisphere, placed 50 mm from the uranium, provided isothermal temperature control and uniform cooling rates for the large castings. A small perforated stainless steel tube and a tantalum cup increased heat dissipation at the pole region of the hemisphere during cooling. The cast part was placed on the tantalum cup and inside the copper hemisphere. Chromel–alumel thermocouples were embedded in the samples to monitor the temperatures and cooling rates at the pole, 45° position and the waist. Four small coupons (each 32 mm × 9.5 mm × 5 mm) were cut from the thick-walled casting for use in experiments designed to establish the parameters for heat-treating the larger casting. These coupons were placed inside a uranium hemishell during heat-treatment so that their thermal histories would be approximately the same as the pole of an actual casting.

Test coupons were heated to 720°C at the rate of 5°C/min. After holding for 2 h at 720°C in a 1.33×10^{-3} Pa vacuum, helium at 170 MPa was added to the chamber until the pressure reached 16.6 kPa. After reaching a target temperature between 570°C and 575°C, the chamber was evacuated with a mechanical pump. Before the heater was turned on to start the annealing cycle, a pressure of $\leqslant 0.133$ Pa was desirable. However, the size of the chamber restricted the rate of evacuation. As a result, there was about a 2-min delay before the start of the annealing cycle. During this time, the temperature of the coupon could increase to 600°C from residual heat in the furnace. To prevent such reheating, and to cool the inside of the furnace, the helium back-fill and pump-down cycle was executed twice. During the second pump-down cycle, the temperature of the coupons slowly decreased to about 510°C, and finally stabilized at about 520°C. After annealing for 3 h at 520°C, the chamber was back-filled with helium to rapidly cool the coupons to room temperature.

Table 1
Analyzed chemical composition of castings

	V (wt%)	C (wppm)	H (wppm)
Thin-walled, as-cast	0.244 ± 0.003	160–245	– [a]
Thick-walled, as-cast	0.259 ± 0.004	70–90	2.1

[a] Not determined, assumed same as thick-wall casting.

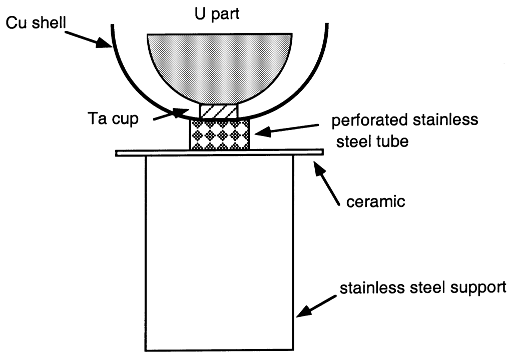

Fig. 2. A schematic illustration of the heat treatment set-up. The thermocouples located on the pole, 45°, and the waist are not shown.

For comparison, a limited number of small coupons were heat-treated in a ‘true isothermal’ fashion using the type of two-zone furnace described by Wood [7]. Each specimen was held at the equilibration temperature (720°C) in one zone of the furnace and then dropped into a second annealing temperature (516°C) zone. Quenching to the isothermal annealing temperature was effected by clamping the small specimen between two pre-heated copper ‘quench blocks’.

The research grade helium-gas used for the isothermal heat treatment study was supplied from a four-bottle manifold connected to the furnace with a 12-mm copper line. The helium was nominally 99.998% pure and contained less than 0.5 ppm water vapor. During cooling the chamber’s interior was pressurized from 48 evenly distributed gas-inlet plots. A cooling turbine was used to recirculate the helium through a water-cooled heat exchanger, thereby continuously cooling the coupons with cold helium.

Optical micrographs of heat-treated specimens were taken in polarized light. The vanadium content was determined with an inductively coupled plasma technique after dissolving the test piece in heated nitric acid. A combustion technique was used to determine the carbon content of the castings. The results of the chemical analyses are summarized in Table 1. Cylindrical specimens 5-mm in diameter and 5-mm high were tested in compression on a hydraulic test machine to a final strain of 30%, at a strain rate of 10^{-3}/s. Flat, dogbone-shaped sheet specimens 1-mm thick and 5-mm in gauge length were strain tested to failure at a strain rate of 10^{-3}/s. All tests were done at room temperature and ambient relative humidity. Two samples were tested for each heat treatment condition.

3. Results and discussion

3.1. *Effect of heat treatment on grain refinement*

The cooling profiles for a helium-mediated cooling cycle of the U–0.25% V coupons are shown in Fig. 3. The transition from the β- to the α-temperatures was not as smooth as a true isothermal transformation between two furnace zones set at 720°C and 516°C. Nevertheless, the calculated cooling rates between 700°C and 600°C were substantial, about 100°C/min. The most difficult aspect of this heat treatment was determining a target temperature that would prevent both reheating and undercooling. Once the coupon started to cool below 560°C, it was extremely difficult to prevent undercooling. Coupon A (Fig. 3) cooled to 575°C but reheated to 600°C and then undercooled to nearly 500°C. The resulting microstructure of coupon A (shown in Fig. 4(a)) has a bimodal distribution of equiaxed grains, with an average grain size of 65 μm.

A more satisfactory cooling history was attained for coupon D (Fig. 3). The benefit of performing the helium back-fill and pump-down cycle twice is seen in the cooling profile of coupon D. It was quenched to 575°C with limited reheating, and then cooled to 510°C. This cooling history produced a uniform distribution of 35 μm average diamete r grains, as shown in Fig. 4(b). since one of the concerns was to prevent undercooling below 510°C to avoid a martensitic transformation, the figures further indicate that coupons cooled close to 500°C can retain an equiaxed grain morphology. This may be a result of the slightly greater amount of vanadium, carbon or other impurities in our materials relative to samples studied by Reisse [5] (Section 3.5).

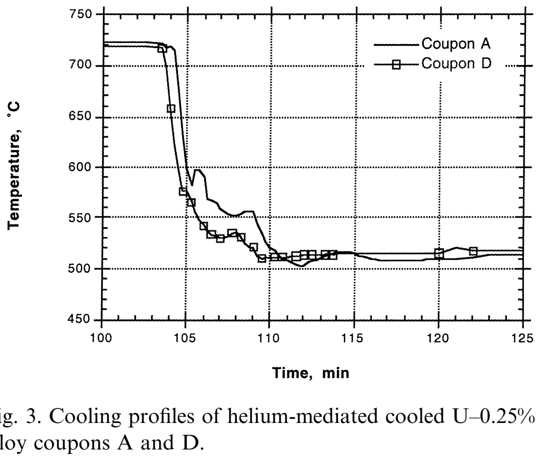

Fig. 3. Cooling profiles of helium-mediated cooled U–0.25% V alloy coupons A and D.

The grain refinement achieved using the helium-mediated heat treatment is more apparent when the microstructure is compared with that of the as-cast, argon-mediated cooled, true isothermally transformed U–0.25% V, and wrought U. The as-cast material, with a cooling rate of roughly 33°C/min during solidification, produces 150 μm grains (Fig. 5(a)). Thermally induced twinning is apparent in the as-cast a grains. Argon-mediated cooling reduces the grain size from 150 to 70 μm (Fig. 5(b)). However, true isothermal heat-treatment reduces the grain size further to 35 μm (Fig. 5(a) and (c)). Note that the size and morphology of the true- and helium-isothermally transformed coupons are comparable (Fig. 5(c) and 4(b)). In the wrought uranium, the effect of cold work is completely eradicated during recrystallization, and the microstructure consists of very fine (20 μm), uniformly equiaxed grains (Fig. 5(d)).

The cooling curve for the thin-walled casting is shown in Fig. 6(a). Because of the greater mass of the casting compared to that of the coupons, it did not cool as rapidly at the lower temperature region of the curve. Instead, there was some reheating (to 570°C), before it stabilized at 516°C. The microstructure of the heat treated casting, sampled at the pole, mid-position, and waist (Fig. 6(b)), consists of a uniform, equiaxed 35 μm grain. The morphology of the grains appears more equiaxed than that of coupon D and the true isothermally transformed material.

3.2. *Compressive behavior*

The results of quasi-static compression tests done (at the rate of 10^{-3}/s) on the as-cast, the cast-and-heat-treated U–0.25% V, and the wrought U are shown in Fig. 7. At this strain rate, no signs of premature cracking were observed. The compressive behavior of as-cast U–0.25% V was nearly identical to that of the wrought U, even though the grain size of the as-cast material was an order-of-magnitude larger. Cast-and-heat-treated U–0.25% V specimens display similar strain hardening characteristics, but at a higher stress level than the wrought specimens. Isothermally heat treated cast U–V alloy with a grain size similar to wrought has a higher flow stress, an effect that might be the result of either solid solution or precipitation hardening associated with the vanadium. Among the cast-and-heat-treated materials, the effect of grain refinement on mechanical response is apparent. There are substantial differences in compressive strengths among the as-cast, argon-mediated cooled, and true and helium-isothermally transformed specimens. The latter two heat treatments yield significantly higher compressive flow stresses. It is interesting to note that the stress–strain curves of true- and helium-isothermally transformed specimens are practically indistinguishable.

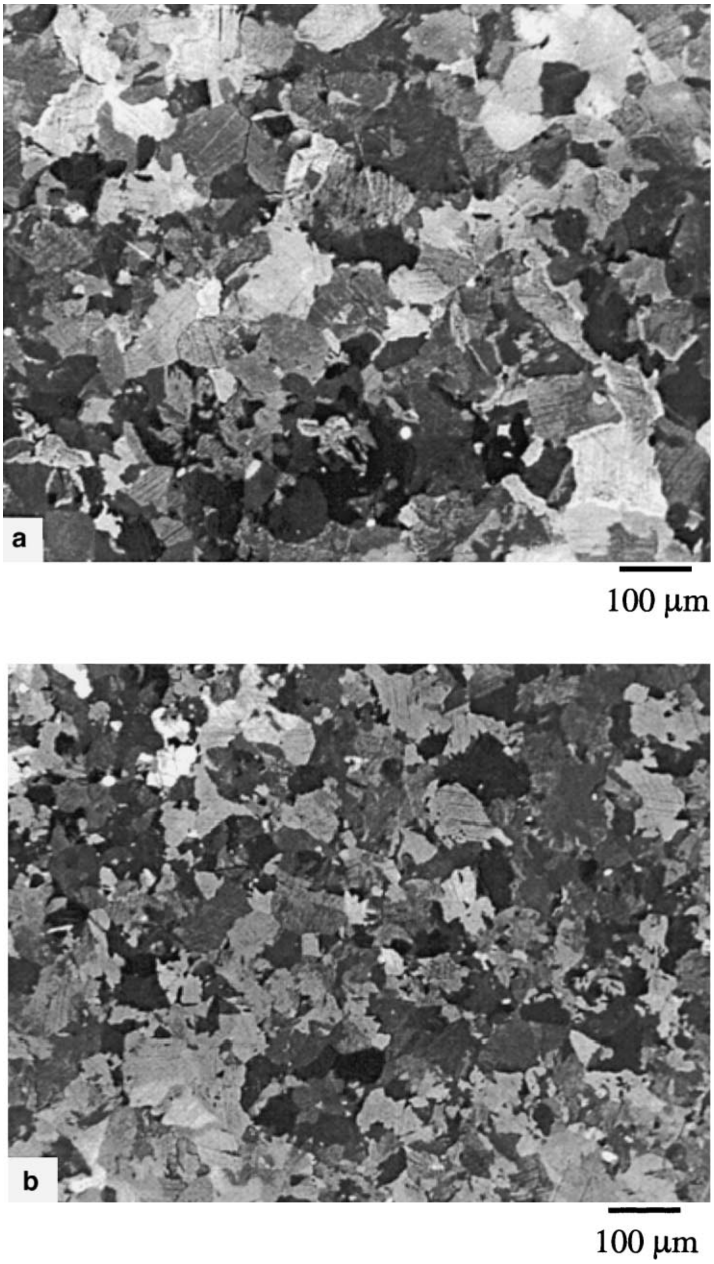

Fig. 4. Optical micrographs of helium-mediated cool U–0.25% V alloy coupons: (a) coupon A; (b) coupon D.

The physical appearance of the compressed specimens varied. Wrought and fine grained cast-and-heat-treated specimens have a uniform, slightly barreled shape, while the as-cast specimens developed an orange peel appearance on their surfaces, which is a common characteristic of coarse grained materials. The microstructural characterization of compressed specimens revealed the effects of 30% compressive strain on the cast-and-heat-treated U–0.25% V specimens. Compared to the isothermally transformed material, the coarser grained argon-controlled cooled specimens show more extensive deformation-induced twinning and a distorted grain morphology (Fig. 8). This clearly indicates that the major mode of deformation at the ambient temperature is by twinning, and that twinning is more difficult in fine grained U [10]. A similar observation was made by Hull [11] for an iron–3% silicon alloy. He found that the twinning behavior of that alloy is related to the in-

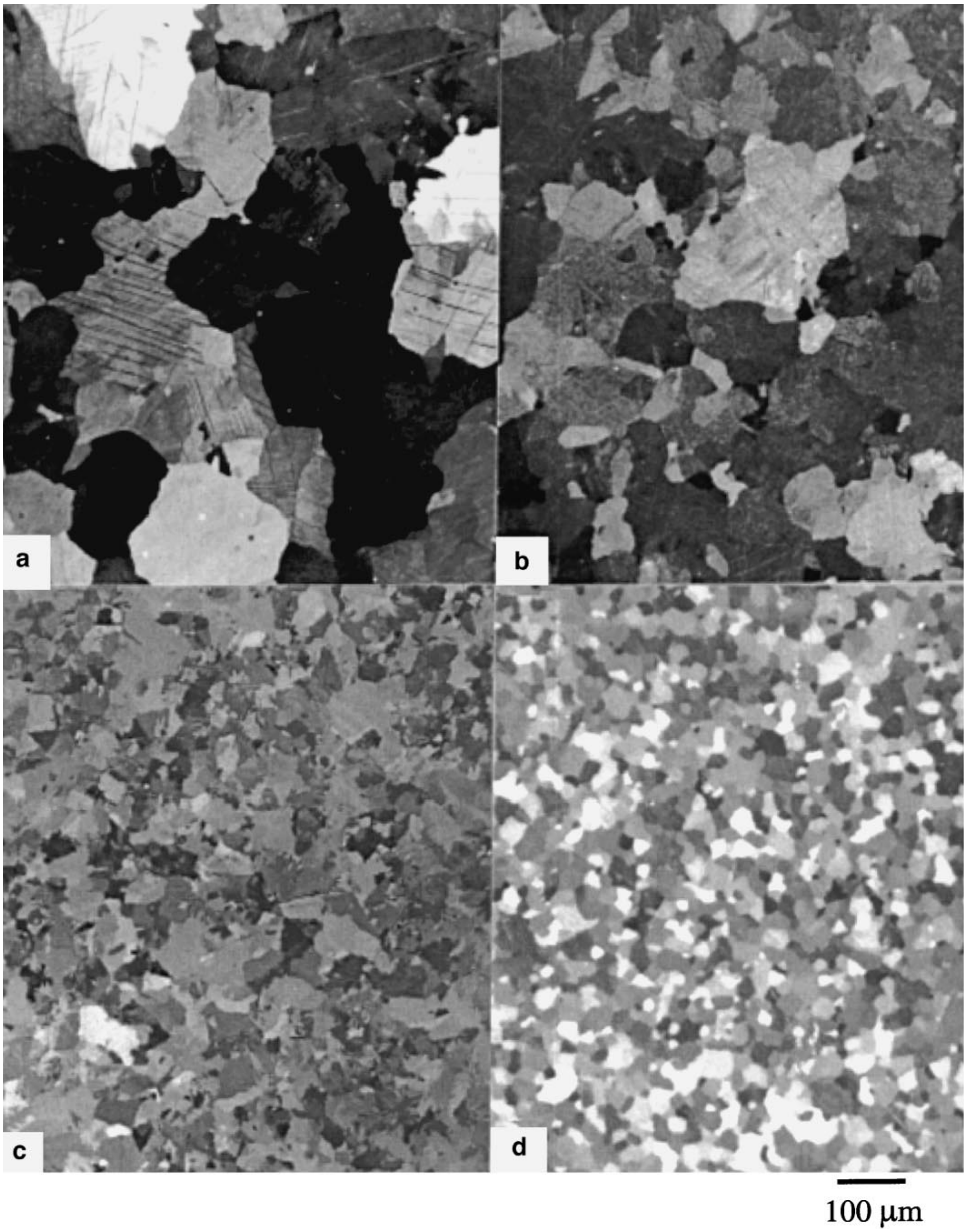

Fig. 5. Effects of heat treatment on grain refinement in cast U–0.25% V alloy and a comparison to wrought U: (a) as-cast; (b) argon-mediated cool; (c) true-isothermal transformation; and (d) wrought U.

verse square root of the grain size. As a result, the grain size difference had a significant influence on compressive deformation behavior by a factor of 2.

3.3. Tensile behavior

Tensile behavior of the helium-mediated cooled, thinner-walled casting was characterized from the pole to the waist and in the tangential and axial directions (Fig. 9(a)). The stress–strain behavior of cast-and-heat-treated U–0.25% V alloy does not vary significantly with position in the casting. Fig. 9(a) further suggests that the cast tensile specimens tend to break near the maximum of the engineering stress–strain curve at the onset of plastic instability. Nevertheless, subsequent isothermal heat treatment of the cast material provided a substan-

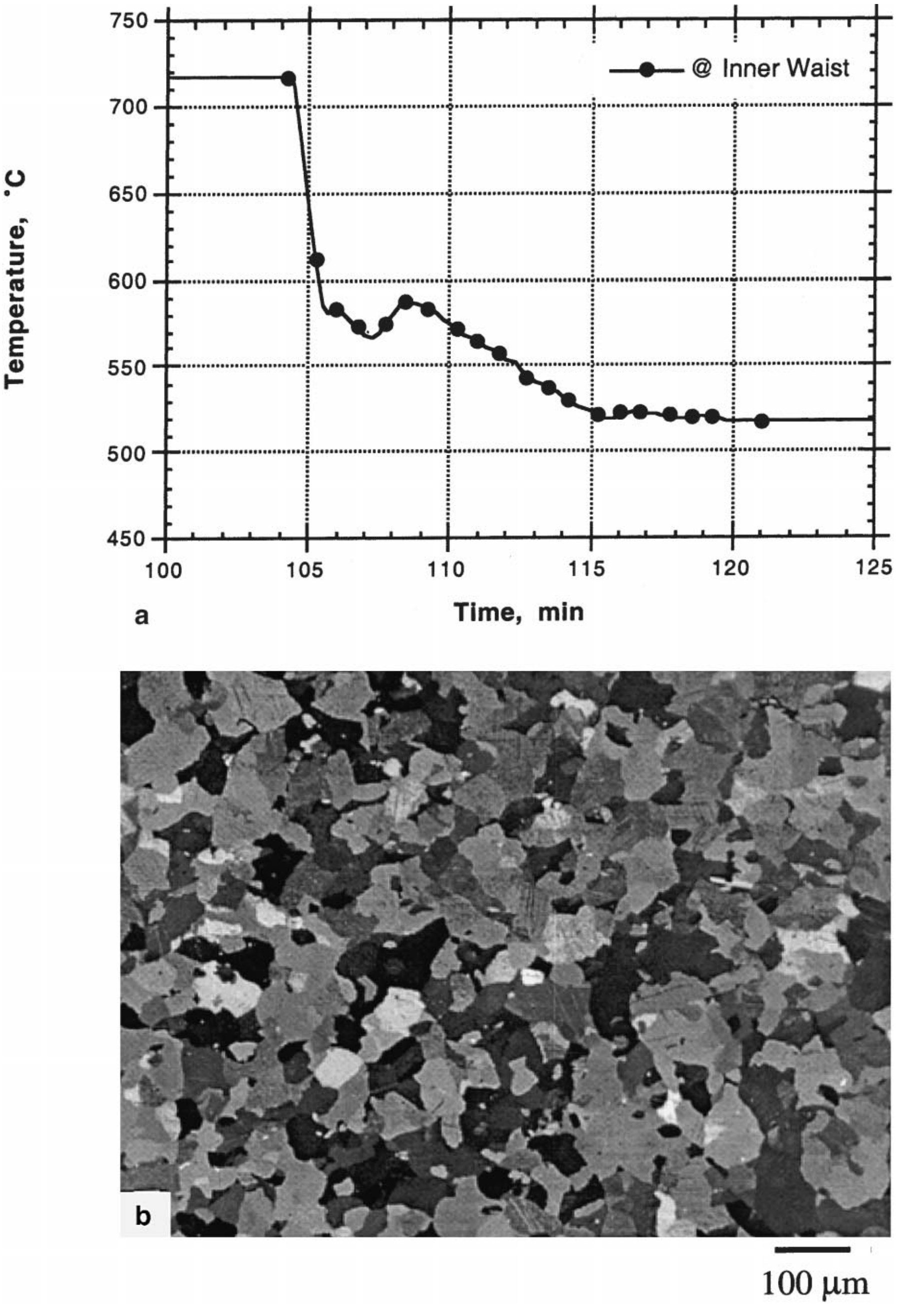

Fig. 6. (a) A cooling curve of a helium-mediated cooled 16-kg casting; (b) optical micrograph of the waist region.

tial improvement in the tensile ductility, from less than the 1% seen in the as-cast material [8] to a 15% elongation in the cast-and-heat-treated material. The reduction in area of the broken specimens was measured at about 17%, very close to the elongation values. The strengths of cast-and-heat-treated material increased accordingly with grain refinement.

Fig. 9(b) compares the stress–strain behavior of the heat treated cast U–0.25% V alloy with the vacuum annealed wrought U. Under the same laboratory test conditions, the helium-mediated cooled U–0.25% V alloy exhibits equal, or superior yield and ultimate strength, but the vacuum annealed wrought U exhibits exceptionally high ductility. However, the ductility of the wrought

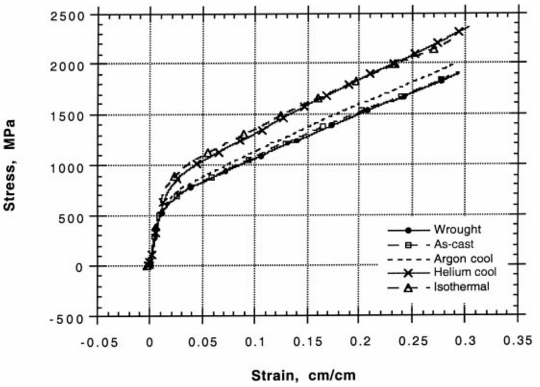

Fig. 7. Quasi-static compression stress–strain curves of cast U–0.25% V alloy and wrought U.

U can also vary substantially with annealing. The wrought U that underwent a salt-bath anneal can display ductility as low as 4%, without adversely affecting its strength. The higher strength of the U–0.25% V alloy can be attributed to the effect of the vanadium solute, while the lower ductility can be attributed to the difference in grain size. Another factor is the presence of hydrogen in the helium-mediated cooled U–0.25% V alloy specimen. It is well documented that small amounts (as low as a fractional ppm concentration level) of hydrogen in uranium leads to embrittlement [12]. The hydrogen content of one of our castings was determined to be about 2.1 wppm. Since the castings were prepared under similar conditions, the hydrogen content of the second casting should be comparable. This could account in part for the low ductility of the cast material relative to wrought. Although the ductility values for the helium-mediated cooled specimen is low compared to the vacuum annealed wrought U, the tensile properties of this material are equivalent or better than tensile property values of other cast-and-heat-treated U–0.25% V alloys [5,13].

3.4. Fractography

The effect of the two different processing methods on U is clearly seen in the fractographs. The fracture surface of the helium-mediated cooled specimen reveals a predominantly quasi-cleavage fracture (Fig. 10(a)). A vacuum annealed wrought U tensile specimen displays a predominantly ductile dimple fracture, with some cleavage (Fig. 10(b)). The grain size influences the deformation behavior. For a larger grained U material, the governing deformation mechanism is twinning.

3.5. Effects of vanadium and carbon in the cast uranium

In addition to its benefits as a grain refiner, vanadium might also function as a hardening agent, presumably either in the form of precipitates, or as a solid solution strengthener in α-U. The solubility of V in U decreases from 0.43 wt% in β-U at 727°C to 0.32 wt% in α-U at 652°C, to nearly zero at room temperature [5]; thus it is possible that V could have precipitated during the aging at 520°C for 3 h. It is also possible that since the coupons were helium-quenched from 516°C to 25°C, some V could have remained in solid solution in the supersaturated α-U matrix to produce strain-induced hardening.

Carbon is of concern in cast U–V alloys. Among other effects, the formation of the carbide V_2C can deplete the vanadium needed to refine the cast microstructure. Collot et al. [14] found that grain refinement no longer occurred when the carbon content exceeded about 150 ppm in U–0.2% V alloy. However, a study by Collot and Reisse [15] showed that for a material containing a very high concentration of carbon (up to 1000 wppm), increasing the V addition to 0.3% gave a grain size of about 50 μm for a controlled cooling at a 30°C/min rate. They therefore concluded that a V addition of 0.3% was insensitive to the action of carbon. Instead, they proposed that the carbon content directly influenced precipitation of the primary carbide, which caused the heterogeneous nucleation of the α phase, and indirectly influenced the kinetics of the β to α transformation [16]. Thus, it seems that the small amounts of carbon that were present in the present casting did not adversely affect the as-cast, or the cast and heat treated structures in U–0.25% V alloy.

The beneficial effect of carbon might be that, coupled with other alloying elements, it lowers the martensitic transformation start (M_s) temperature [17]. In steel containing 1.0 wt% carbon, increasing the manganese content from 4 to 6 wt% causes the M_s temperature to rapidly decline from 100 to 0°C. This inverse relationship between the solute content and the M_s temperature is similarly observed in U alloys [16]. In the cast U, the effect on lowering the M_s temperature of a 0.05% increase in vanadium concentration could be very subtle. However, vanadium combined with carbon could contribute to a lower M_s temperature [18].

The M_s temperature for the U–0.2% V alloy containing 70 wppm carbon was about 510°C, while the M_s temperature for the U–0.25% V alloy containing about 200 wppm carbon may be about 500°C. This would then explain the microstructure seen in the cast-and-heat-treated coupons: Because of a lowered transformation temperature, coupons that were undercooled to 510°C did not transform to the martensitic structure.

4. Conclusions

From this work, the following conclusions are drawn:

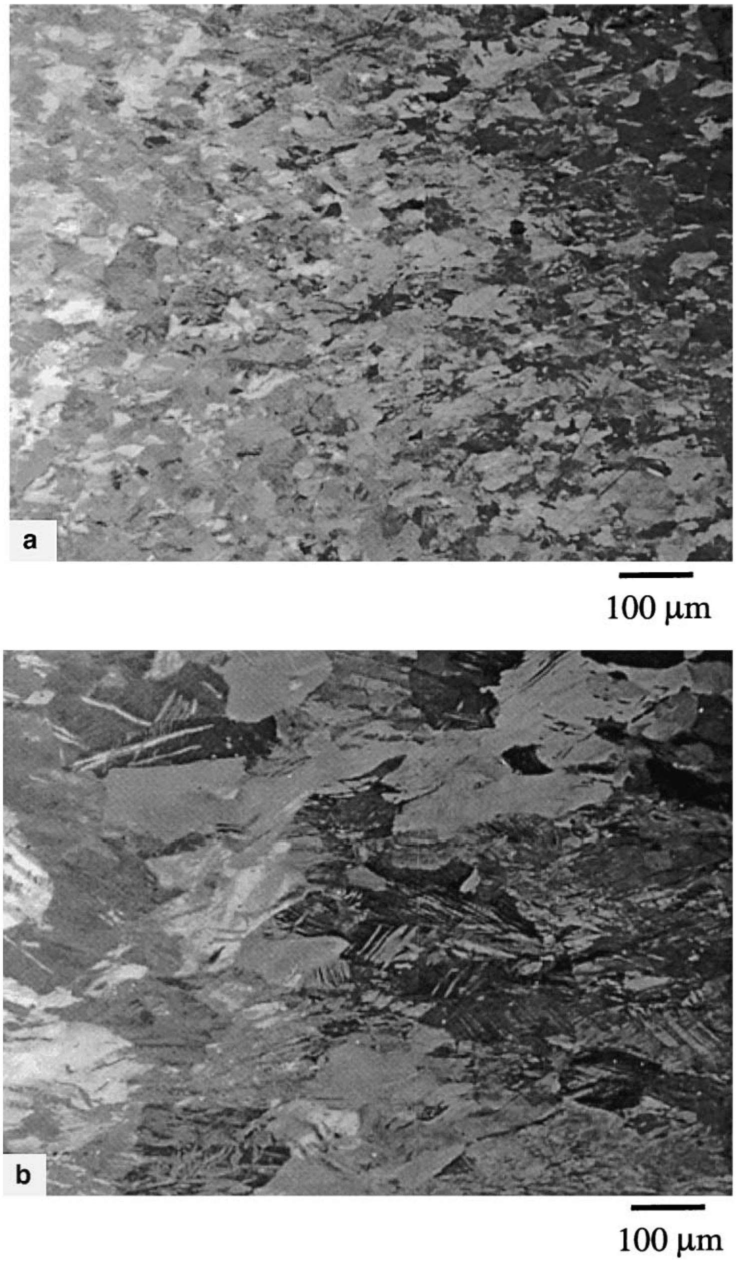

Fig. 8. Cross-sectional view of cast and heat treated U–0.25% V alloy specimens strained to 30% compression: (a) isothermal transformed; (b) argon-mediated cooled.

1. Large (10–20 kg) castings of U–0.25% V can be successfully heat-treated to refine their grain size by using a helium mediated ‘quench’-and-anneal procedure suitable for large commercial vacuum furnaces. Using this method, the grain size can be reduced from 180 μm in diameter to as small as 35 μm.
2. The yield and flow stress behavior of the as-cast U–0.25% V alloy is comparable to that of the wrought uranium, despite the large difference in grain size (150 μm versus 20 μm). The vanadium addition produces solid solution or precipitation strengthening that offsets the effect of this difference in grain size between cast and wrought.
3. Finer grain size in the cast-and-heat-treated U–0.25% V alloy promotes larger yield and flow stress.
4. Vanadium additions combined with carbon might slightly depress the transformation temperatures in uranium, thereby allowing a fine, equiaxed heat-treat-

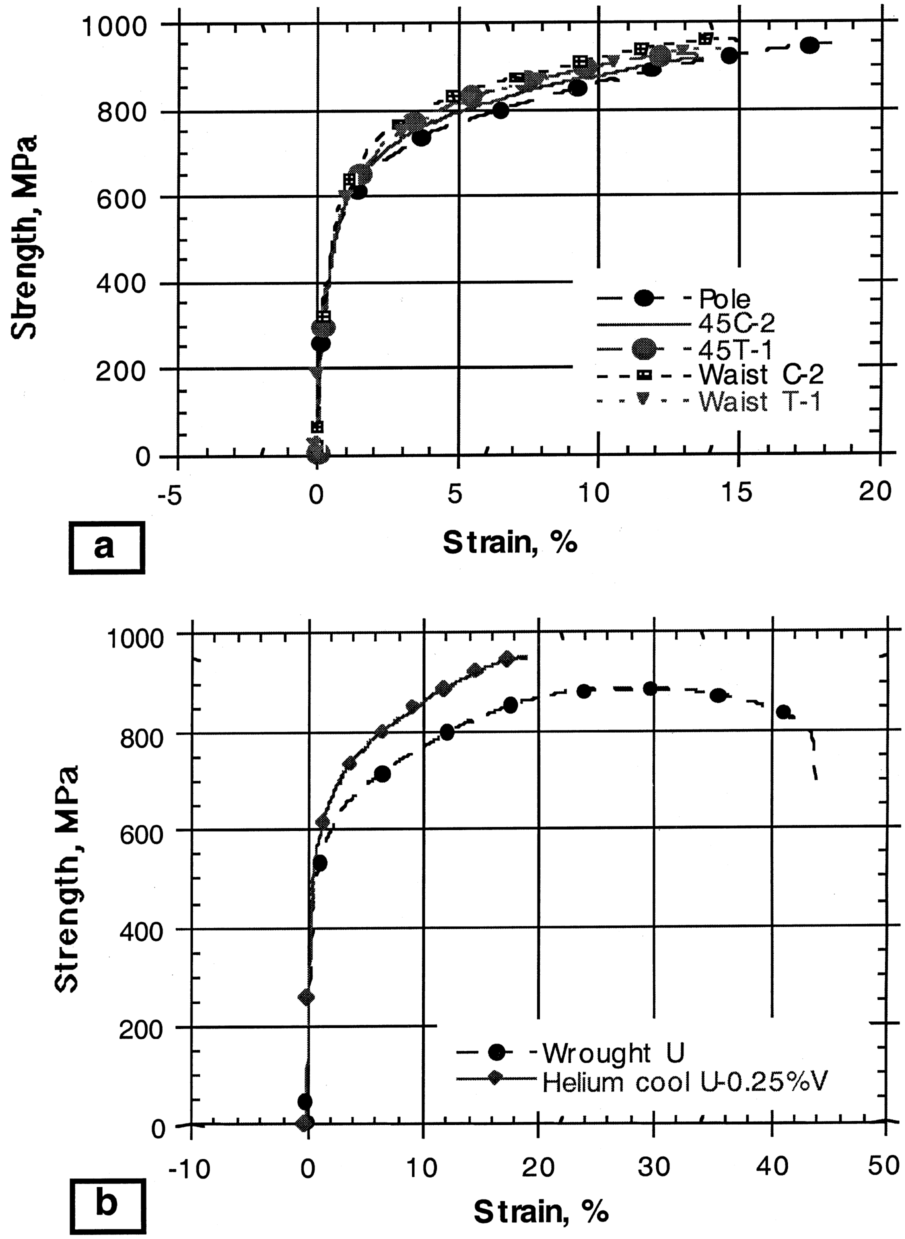

Fig. 9. (a) Quasi-static tension, engineering stress–strain curves of a helium-mediated cooled U–0.25% V hemisphere, from pole to waist (in the tangential (T) and circumferential (C) directions); (b) comparison to a vacuum annealed wrought U.

ed microstructure to be retained at annealing temperatures as low as 510°C.

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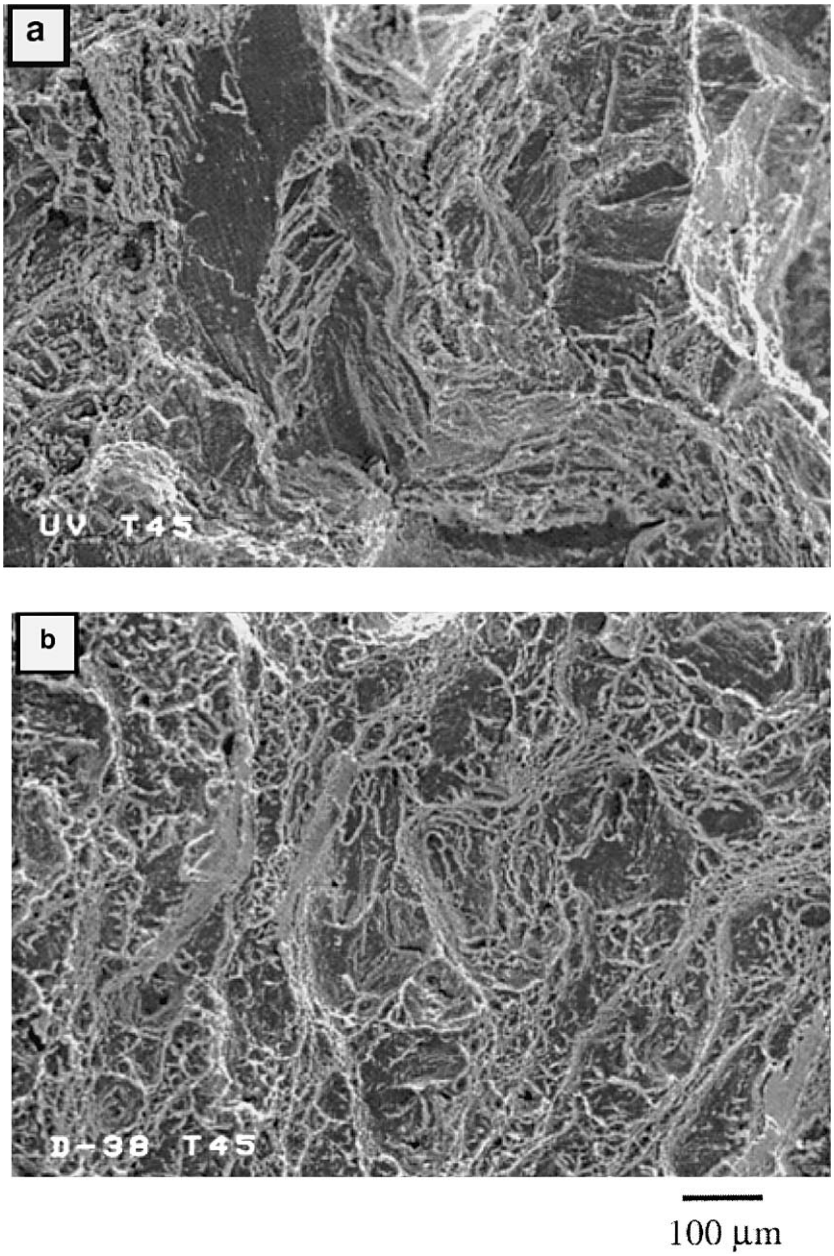

Fig. 10. Fractographs of broken tensile specimens in the 45° circumferential direction: (a) a helium-mediated cooled U–0.25% V hemisphere; (b) a vacuum annealed wrought U hemisphere.

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