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# Achieving superior grain refinement and mechanical properties in vanadium through high-pressure torsion and subsequent short-term annealing

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ABSTRACT

Commercial purity vanadium with an initial grain size of ~27 μm and a Vickers microhardness of Hv ≈ 85 was processed by high-pressure torsion (HPT) under a pressure of 6.0 GPa at room temperature through 1/2 to 10 turns. After processing through 10 turns, some samples were immediately subjected to a short-term annealing (15 min) at different temperatures from 773 to 1173 K. The microstructures developed in HPT and in HPT plus post-HPT annealing were characterized by electron backscatter diffraction (EBSD). Processing by HPT for 10 turns gave a refined grain size of ~410 nm and an increased hardness of Hv ≈ 240. Post-HPT annealing demonstrated that the ultrafine grained vanadium has good thermal stability up to at least 873 K. Tensile testing at room temperature gave an ultimate tensile strength of ~920 MPa after 10 turns of HPT with an elongation of ~29%. These results show HPT processing produces superior mechanical properties in vanadium by comparison with processing by ECAP or ECAP plus cryorolling.



## 1. Introduction

Vanadium is a refractory metal having a body-centred cubic (bcc) structure with a high melting temperature (2163 K). It is soft and ductile and has good forming properties at low temperatures [1]. It also has a high strength at high temperatures with a brittle-to-ductile transition temperature which is low compared with other refractory metals [2]. Vanadium is used mostly in industry as an additive to steels or titanium alloys in order to increase the strength, hardness and high temperature stability.

Vanadium has received considerable attention in nuclear applications because it has the lowest capture cross-section for high energy neutrons of all refractory metals (~1.5 m barns for 1 MeV neutrons) combined with good structural strength and a high melting point [3]. Thus, vanadium is a leading candidate material for first-wall and blanket structures in fusion reactors [4–8]. It is well known that vanadium is chemically reactive with gaseous interstitial impurities, such as oxygen and nitrogen, and this would produce interstitial embrittlement with an increase in yield strength and hardness and an accompanying loss in ductility and formability [7,9–10].

In practice, interstitial embrittlement is a major deterrent for using vanadium in nuclear reactor applications and it is generally attributed to an overall weakness of the grain boundary bonding strength [11]. Furthermore, it is anticipated that irradiation by fast neutrons, at temperatures between ~0.3 and $0.5T_m$ where $T_m$ is the absolute melting temperature, may also produce swelling by void formation and growth processes depending upon the fluence, temperature, gas content and microstructure. It is generally recognised that a refinement of the grain size will reduce the irradiation-produced vacancy supersaturation which is required for void nucleation and growth [3]. Thus, it is reasonable to anticipate that the development of vanadium with an ultrafine grain size will be beneficial in reducing the interstitial embrittlement and swelling during radiation and thereby it will significantly enhance the potential for nuclear applications.

Attempts were reported for achieving a fine-grained structure in Y-containing vanadium alloys through mechanical alloying and hot isostatic pressing (HIP) followed by annealing [8–10] and in pure vanadium via HIP and annealing [12]. However, it is recognised that these powder metallurgy products may suffer from contamination and residual porosity which would hinder their application in nuclear reactors.

It is well known that significant grain refinement may be

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introduced in bulk polycrystalline materials through the application of severe plastic deformation (SPD) [13–15], with the processed grain sizes typically lying within the submicrometer or even the nanometer range. Among various SPD techniques, the most common procedures are equal-channel angular pressing (ECAP) [16] and high-pressure torsion (HPT) [17] where ECAP requires repetitive pressings in order to achieve large strains whereas HPT leads to exceptionally high strains in a single processing operation. In practice, HPT is especially attractive because, by comparison with ECAP, it produces both smaller grains [18,19] and a higher fraction of grain boundaries having high angles of misorientation [20].

To date, there are only a limited number of reports describing the processing of vanadium to produce microstructural refinement using SPD techniques [11,21–24]. Earlier experiments showed that the grain refinement in vanadium saturated after eight passes of ECAP processing [21] and therefore cryogenic rolling was introduced after ECAP in order to achieve additional refinement by imposing a further 60% strain by cryorolling [11,22]. These materials showed increases in the ultimate tensile strength (UTS) from ~750 MPa after ECAP to ~920 MPa after ECAP plus cryorolling but this was accompanied by a ductility loss so that the total elongations to failure for these two processing routes were reduced from ~14% to ~8.5% [11]. Very recently there have been two reports describing grain refinement of pure vanadium processed by HPT [23,24] and these results show that the material exhibits little or no ductility.

Considering the limited ductility attained in the ECAP plus cryorolled condition and the lack of any systematic evaluation of microstructural evolution during HPT processing, the present research was initiated to study the modifications that may be introduced in pure vanadium by combining HPT processing and subsequent short-term annealing. The overall objective of this research was to establish a material processing procedure that may be used to refine the microstructure and enhance the properties in order to achieve a high strength vanadium with improved ductility

## 2. Experimental material and procedures

Vanadium of 99.8% purity was supplied by Goodfellow Ltd. (Cambridge, UK) with typical trace elements (in ppm) of Ag 1, Al 2, Ca < 1, Cr 15, Cu < 1, Fe 70, Mg < 1, Mn 1, Si 300. The as-received vanadium was in a rolled state in the form of rods having diameters of 11 mm. These as-received rods were annealed at 1173 K for 1 h in a vacuum furnace and this is henceforth designated the as-annealed state. The rod was then machined to a diameter of 9.9 mm, sliced into discs with thicknesses of ~1.2 mm and these discs were ground with abrasive papers to final thicknesses of ~0.8 mm.

All discs were processed by HPT at room temperature through total numbers of turns, $N$, of 1/2, 1, 5 and 10 using an imposed pressure of 6.0 GPa and a rotational speed for the lower anvil of 1 rpm. The HPT was conducted under quasi-constrained conditions where there is a small outflow of material around the periphery of the disc during the processing operation [25,26],

To investigate the thermo-stability of the HPT-processed discs, samples processed through 10 turns were removed from the HPT facility and then immediately annealed for short times of 15 min at temperatures of 773, 873, 973, 1073 or 1173 K. These annealing temperatures were selected based on an earlier report presenting microstructural analysis as a function of annealing temperature for vanadium processed by ECAP where recrystallisation was almost complete at 973 K after annealing for 1 h [21]. Considering the small size of the HPT discs, and in order to avoid recrystallisation which may cause strength loss and grain growth, the HPT-processed samples were subjected to only very short-term anneals of 15 min. This is henceforth designated the post-HPT annealed condition.

For microstructural analysis, discs were examined in the as-annealed condition, in the HPT-processed condition and in the post-HPT annealed condition. These discs were hot-mounted in bakelite, ground with abrasive papers and then a final polish was performed using a colloidal silica solution in order to produce a mirror-like surface. For the as-annealed sample, the polished surface was etched using a solution of 30 mL of 32% hydrochloric acid (HCl), 15 mL of 65% nitric acid ($HNO_3$) and 30 mL of hydrofluoric acid (HF) with an etching time of 10 to 20 s. The sample surface was then observed in dark field using an Olympus BX51 optical microscope in order to determine the initial grain structure. The average grain size was measured by the linear intercept method using Image J software and measurements of more than 300 grains. The grain structures after HPT processing and post-HPT annealing were examined by electron backscattered diffraction (EBSD) using a JSM6500F thermal field emission scanning electron microscope (SEM). The EBSD patterns were collected using a step size of 60 nm and a cleaning procedure was applied such that the total numbers of modified points were less than 10% of all points measured. High-angle grain boundaries (HAGBs) were defined as boundaries having misorientation differences between adjacent measuring points of more than 15° and low-angle grain boundaries (LAGBs) had misorientation differences of $2° - 15°$.

Hardness measurements were taken using an FM300 hardness tester equipped with a Vickers indenter with a load of 300 gf and a dwell time of 15 s. The hardness values on the HPT-processed samples were measured after $N$=0.5, 1, 5 and 10 turns by mapping the values of Hv over the total surface of each disc. Specifically, the individual values of Hv were recorded following a rectilinear grid pattern with a separation of 0.3 mm between each adjacent point [27]. All of these values were then used to construct colour-coded contour maps that provided a clear visual presentation of the distributions in hardness across the surface of each disc. The hardness values on the as-annealed and post-HPT annealed samples were measured along disc diameters with a separation distance of 0.3 mm between neighbour points.

Tensile tests were conducted using the as-annealed sample, the HPT-processed samples and the post-HPT annealed samples. Following earlier practice [28], and in order to avoid any microstructural inhomogeneities in the centres of the discs, two tensile specimens were prepared from each disc using electro-discharge machining and with the specimens arranged symmetrically on either side of the disc centre. These miniature tensile specimens had gauge lengths and widths of 1 mm. The tensile specimens were pulled to failure at room temperature using a Zwick 30 KN Proline testing machine operating at a constant rate of cross-head displacement with an initial strain rate of $1.5 \times 10^{-3}\ s^{-1}$.

## 3. Experimental results

### 3.1. Microstructure evolution during HPT processing

In the initial as-annealed condition, the vanadium had a uniform equiaxed microstructure with an average grain size of ~27 μm and an average hardness of Hv ≈ 85.

The microstructural development after HPT processing is shown in Fig. 1 with the centre areas of the discs on the left, the edge areas on the right and images displayed in the order of 1/2, 1, 5 and 10 turns, respectively: the colours denote different grain misorientations as depicted in the unit triangle on the right, LAGBs (2–15°) are shown in yellow and HAGBs ( > 15°) in black. After 1/2