Development of tungsten oxides on \{1\ 1\ 1\} and \{0\ 0\ 1\} crystallographic planes in hot-rolled tungsten

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Abstract

Tungsten, a divertor material for future fusion reactors, may be exposed to air during abnormal operation or accidents. The oxidation mechanisms in tungsten are understudied. This work addresses the structural transformations in tungsten oxides linking them to crystallography of the base material. Annealing experiments in Ar–O\(_2\) have been conducted at a temperature \(T = 400\) °C under a varying oxygen partial pressure and oxidation time. A combination of EBSD, Raman spectroscopy and confocal microscopy shows preferential oxidation initially on \{1\ 1\ 1\} base material planes, which later reverses to \{0\ 0\ 1\} planes. A transition between the orthorhombic and the hexagonal tungsten trioxides has been identified.

Keywords: A. tungsten; B. Raman spectroscopy; B. EBSD; B. CLSM; C. oxidation

1. Introduction

Tungsten, a refractory BCC metal with high thermal conductivity [1] and low sputtering yield [2] [3] [4], is the current material of choice for the divertor component of International Thermonuclear Reactor (ITER) [5] [6] – and potentially for the DEMOnstration Power Station. There are two major issues with using tungsten as a divertor material. Firstly, the low oxidation resistance [7] [8] of ITER grade (99.94 wt. % fully sintered; forged and/or swaged, cold or/and hot-rolled and stress relieved [9]) tungsten involves certain risks during accidents and abnormal operation (such as air leaks [10]). A worst case loss-of-coolant accident (LOCA) scenario with simultaneous air ingress [11] [12] would result in a complete loss of the tungsten monoblock divertor by its conversion to volatile oxides [13]. In the absence of cooling, nuclear decay heat would gradually increase the divertor temperature beyond the point at which oxides start to volatilise. These oxides contain radioactive material formed during reactor operation [11]. The second issue pertaining to the use of tungsten in fusion applications is its high brittle-to-ductile transition temperature [2] [14], which may lead to cracking during high thermal loads in the event of plasma disruption.

Multiple attempts have been undertaken to promote oxidation resistance of tungsten-based materials by proposing several ternary (W–Cr–Y ‘smart alloys’ [15] [16] [17]) and quaternary (W–Mo–Cr–Pd [18] [19] [20]) tungsten alloys. Although some results look promising, the new material is still unlikely to withstand an extended LOCA event, which could last for up to three months. For instance, the passivating Cr\(_2\)O\(_3\) layer in a W-Cr-Y smart alloy is breached after 467 h oxidation at 1000 °C [21]. With increasing the complexity of composition, it becomes harder to predict how other properties of these alloys (e.g., thermal conductivity, fracture toughness) will evolve, and whether the original benefits of using tungsten are preserved. Another trend in tungsten alloy design places emphasis on improving mechanical properties of the base material and considers a completely separate set of alloying elements (potassium, rhenium, lanthanum oxide [22]). These two lines of thought may potentially be conflicting.

A systematic study of tungsten oxidation is still lacking, although many researchers have proposed different interesting approaches to tackle this phenomenon [23] [8] [24] [25]. For instance, analysis of tungsten exposed to short oxidation at small oxygen partial pressure has been previously reported in [23] [25]. Authors of [24] [25] have observed preferential oxidation in tungsten (Schlueter & Balden [24] report preferential oxidation on \{0\ 0\ 1\} grains) when the average thickness of the oxide layer is about a few micrometers – an effect known in zirconium alloys for fuel cladding materials in fission reactors [26] and some silver-based intermetallic compounds [27]. Cifuentes et al. [8] report different oxide phases found on the surface of tungsten oxidised at 600–800 °C for up to 100 h. Overall, the tungsten-oxygen system seems to be a very complex one. A variety of oxides may be formed on the surface of pure tungsten including [28] [29]: stable stoichiometric oxides formed by either W\(_{6+}\), W\(_{5+}\), or W\(_{6+}\) ions; non-stoichiometric oxides formed by a mix of different ion species; metastable oxides with intermediate compositions presented by W\(_n\)O\(_{3n-2}\) and W\(_n\)O\(_{3n-1}\) series, also known as the homologous Magneli phases. In addition, tungsten trioxide (WO\(_3\)), which is also the higher oxide in this system, comes in variety of polymorphous states, including: monoclinic, triclinic, tetragonal, hexagonal, and orthorhombic. This material attracts significant attention due to...
its unique semiconducting properties [30, 31].

The goal of this study is to examine how the crystallographic orientation of tungsten grains affects the oxidation process – both at the initial stage, when the oxide scale is very thin and oxidation is mainly controlled by adsorption, and at later stages, when the growth of the oxide is mostly diffusion-controlled.

2. Material and methods

2.1. Sample preparation

Cylindrical (Ø 3 mm × 0.5 mm) samples were cut by electrical discharge machining from standard quality, hot-rolled pickled 99.97 wt. % pure tungsten sheet (1 mm × 100 mm × 100 mm) supplied by Plansee.

Grinding and polishing was performed using an AutoMet™ 250 Grinder-Polisher. For this purpose, samples were mounted on stainless steel holders with Crystalbond™ 509. Wet grinding was done with SiC paper followed by fine polishing with 3 µm and 1 µm diamond suspension. The final stage of polishing involved using Buehler MasterMet 2 Non-Crystallizing Colloidal Silica. To ensure samples were free of scratches, examination on an Olympus MX51 optical microscope was performed between polishing stages. Samples were thoroughly rinsed with water to avoid colloidal silica stains. Crystal bond was removed with acetone in an ultra-sonic bath. Residues of acetone and carbon contamination were removed from the samples’ surface by rinsing in isopropanol and deionised water and scrubbing with PELCO optical lens tissue until no carbon particles could be observed with low-magnification optical microscopy.

Samples were subsequently mounted on double-sided conductive adhesive copper tape. For later imaging analysis, three fiducial markers (≈ 10 µm each) – to introduce asymmetry used for orienting the sample – were indented on each sample (Fig. 1). This was done using a Agilent Nanoindenter G200 in the indentation mode with a Berkovich tip.

Finally, before oxidation experiments, the copper tape was removed and samples were cleaned to remove residual adhesive following the cleaning procedure above. Cleaned or oxidised samples were kept in a desiccator system at near vacuum.

2.2. Characterisation methods

Prior to oxidation, samples were characterised with SEM and electron back scattered diffraction (EBSD). In addition, large-area (350 µm × 350 µm) scans were performed with confocal laser scanning microscopy (CLSM) to gather data on surface roughness. Raman spectroscopy and CLSM imaging were used to examine the oxidised layer.

2.2.1. SEM and EBSD

The SEM used in this work was TESCAN Mira3 XMH with a Schottky field emission gun and a NordlysNano EBSD detector. For EBSD mapping (Fig. 2) samples were placed on a 70° pre-tilted specimen holder. The following settings were used during imaging: high voltage (HV) 20 kV, DEPTH scanning mode and beam intensity ≈ 20 (corresponding to a spot size 60–70 nm). Samples were oriented so that indents were aligned vertically (as in Fig. 1). For EBSD, the scan region was centered exactly between the indents. After centering, the view field was set to 550 µm, and the size of the scan region inside that view field was set to 350 µm × 350 µm. A step size of 0.25–0.34 µm was chosen for EBSD imaging to allow detailed mapping. Due to the tilt and long scanning time (2 hours), samples experienced small drift, which was corrected for using the Aztec automatic drift correction. For Kikuchi pattern acquisition, we used a gain of 5–7, exposure time of 20–35 ms, and binning mode 4 × 4. The percentage of unresolved pixels was less than 10 %, which allowed effective noise elimination using HKL CHANNEL5 post-processing software (9 × 9 smoothing window). For grain boundary detection, the critical angle was set to 10° and minimum grain size to 10 pixels.
2.2.2. Raman spectroscopy and CLSM

Raman spectroscopy has been used recently to characterise structural changes in nanocrystalline WO$_3$ as a function of applied voltage [32], and to characterise thin (up to 200 nm) oxide layer formed on a tungsten metal before and after exposure to deuterium or helium plasma [33][34]. CLSM has been used previously as a tool to study topography in the range of 1–2 µm in oxidized and sputtered tungsten in [24][4].

In this work, the WITec alpha300 R confocal Raman imaging microscope was used to acquire Raman spectra in the raster mode and perform large area scans (Raman and CLSM maps). The TrueSurface feature enabled dynamic focusing of the confocal laser during scanning. The inter-quartile (IQR) range of CLSM data gathered from the 350 focal laser during scanning. The inter-quartile (IQR) range of CLSM data gathered from the 350 focal laser during scanning.

The latter could be achieved by adopting a gain of 230 and an integration time of 2 h per sample. The latter could be achieved by adopting a gain of 230 and an integration time of 2 h per sample.

For individual Raman spectra a conventional low-noise high-intensity detection mode was used (integration time was set to 0.5 s with 100–300 accumulations); whereas for the large area scans (75 µm x 75 µm) an electron multiplying charge coupled device (EMCCD) camera was selected which allowed fast acquisition (scanning time ≈ 1500 px x 1500 px) areas.

2.2.2.2. Raman spectroscopy and CLSM

In order to remove noise from the baseline Raman signal, an electron multiplying charge coupled device (EMCCD) camera was selected which allowed fast acquisition (scanning time ≈ 1500 px x 1500 px) areas.
taken. This fitting procedure relies on two parameters: $\lambda$ and $p$. $\lambda$ is the curvature penalisation term, and $p$ determines the asymmetry in the least squares iterations:

$$S = \sum_{i} w_i (y_i - z_i)^2 + \lambda \sum_{i} (\Delta^2 z_i)$$  \hspace{1cm} (2)

where $w_i = p$, if $y_i > z_i$, else $w_i = (1 - p)$. Typically $\lambda = 10^5 - 10^7$ and $p = 0.001 - 0.05$.

The AsLS is encoded in the weighting term $w_i$. If the calculated baseline intensity $z_i$ is higher than the measured signal, then the weighting term is equal to $(1 - p)$ and is large. A set of linear equations is solved iteratively in reference to [37]. Fig. 3c illustrates the baseline fitting procedure for a model spectrum.

### 2.3.3. Gaussian peak deconvolution

A gaussian peak deconvolution software package based on python’s lmfit module was used to deconvolute the peak positions for the baseline subtracted W oxide Raman signal. The Levenberg-Marquardt algorithm, a damped least-squares (DLS), was used in combination with a BIC statistic to determine the most likely number of gaussian peaks and their parameters (peak position, FWHM and amplitude) which form up the tested spectra. Gaussian functions were chosen to fit the underlying positions, FWHM and amplitude) which form up the tested spectra.

### 2.4. Oxidation procedure

In this work, oxidation at a relatively low temperature was considered to make the set-up easier: $T = 400 \ degree \ C$ – close to the conditions used by Schlueter & Balden [24]. Under these conditions, interaction between W and the crucible material can be ignored. Because oxidation is slow compared to the chamber filling time, the oxygen partial pressure or the oxidation time can be efficiently varied to model specific oxidation stages. In addition, $T = 400 \ degree \ C$ corresponds to the starting conditions of a W divertor monoblock during LOCA, as simulated in [39].

Each sample (a full list of samples with respective experimental conditions is given in Table [1]) was placed in Al$_2$O$_3$ crucibles with 0.12 ml capacity, polished side facing upwards. No lid was used to cover the samples. The crucibles were then placed on top of the Pt heating stage with Pt/Pt-Rh sensors of a Linseis STA PT1600 simultaneous thermal analyser. The chamber was then evacuated and samples heated up to 400 °C with an initial heating rate 20 deg./min which was changed halfway through the heating process to 10 deg./min (Fig. 5a) to decrease thermal inertia. After thermal equilibration, the instrument chamber was filled with Ar–20 % O$_2$ at a flow rate 38 l/h. Isothermal oxidation segments were designed differently to model.

(a) **Initiation of oxidation.** Chamber was filled up to different oxygen partial pressures below 1 bar. The oxidation time was kept at 20 mins (Fig. 5b), which is much longer than the transient period when the gas was flowing in or out. Different initial stages of oxidation could be observed depending on the controlled atmosphere inside the chamber;

(b) **Progression of oxidation.** Pressure was kept at 1 bar during the whole duration of oxidation. Oxidation time varied from 20 min to 72 h.

The chamber was then evacuated and the furnace cooled down to room temperature at 20 deg./min.

### 3. Results and Discussion

#### 3.1. Summary of reference spectra

A well-developed Raman peak database for characterisation of tungsten oxides doesn’t exist. Therefore, reference samples of WO$_3$ (Tungsten (VI) oxide) and WO$_2$ (Tungsten (IV) oxide) powders were purchased from Sigma-Aldrich and characterised with Raman scattering analysis. It was noticed that WO$_2$ was unstable at a laser power $W > 4 \ mW$. The instability also manifested itself as an irreversible change of color from black to blue-violet. Different polymorphous states of WO$_3$ which were not readily available for purchase were analysed by digitising published literature and subjecting them to the same

![Figure 5: Oxidation conditions in the STA: (a) time-temperature profile (measured values); (b) Ar–20%O$_2$ partial pressure profile (measured values).](image-url)
baseline subtraction, smoothing and peak fitting as described in Sect. 2.2.3. Individual positions of the peaks were additionally confirmed by a Fourier self-deconvolution procedure [40]. The resulting spectral shapes are provided in Fig. 5 and Table 2. All the peak positions arranged so that the reader is easily able to match the occurrence of a specific peak to particular oxide phases.

Referring to Fig. 6 a few discrepancies between digitised literature spectral data are observed and this might be attributed to different instrumental settings. Hence, where possible, measurement conditions were kept to as described in the experimental procedure outlined in Sect. 2.2.2. The low-wavenumber region of Table 2 shows that only few peaks can be used as markers of the oxide phase — especially when the signal/noise ratio is weak. In addition, bands below 200 cm\(^{-1}\) can be ascribed to lattice vibrations according to [44]. Therefore, the 600–1000 cm\(^{-1}\) region of the spectra is used for phase analysis, as the spacing between characteristic peaks for different oxides phases seems to be sufficient for robust peak deconvolution. All the analysed Raman spectra of W oxides exhibit a dominant peak in the range 770–820 cm\(^{-1}\). Because non-oxidized tungsten does not show any significant Raman signal (perhaps, only fluorescence, which contributes to the baseline), a sum filter in the range 750–850 cm\(^{-1}\) may be used as an indicator of the thickness of an oxide scale grown on W metal. Additional examination of the reference spectra shows that at the same experimental conditions (i.e., laser power, spectrometer configuration, etc.), the Raman signal of certain oxide phases is inherently weaker than of the others (Fig. 5). This is a key factor contributing to the occurrence of ‘tails’ and ‘humps’ on the measured Raman spectral peaks.

### 3.2. Initiation and growth: correlating EBSD and Raman scattering maps

First results were gathered for the variable \(p_{\text{Ar}-\text{O}_2}\) oxidation runs with a 20 min duration (Table 1). Then the samples were exposed to oxygen at normal pressure while the oxidation time was allowed to vary up to 72 h (Sect. 2.4).

The results of applying the sum spectral filter (770–820 cm\(^{-1}\)) to the Raman spectral data gathered for a 75 \(\mu\)m \(\times 75 \mu\)m region on the less-oxidised samples are shown in Fig. 7. A clear preference is observed for oxidation on \{1 1 1\} oriented W grains compared to \{0 0 1\} type orientations. This was noticed for all samples in the initiation stage \((p_{\text{Ar}-\text{O}_2} = 0.01–0.2 \text{ bar})\).

This reduction of intensity in the Raman signal for \{0 0 1\} and \{1 1 1\} type surface grain orientations, used as an indicator for oxide thickness, is shown in Fig. 5 and this presents direct evidence of a thinner oxide on \{0 0 1\} planes. Schlueter & Balden [24] observed that the \{1 1 1\} oriented W grains showed a lower oxidation rate compared to the \{0 0 1\} orientations, e.g. at 600 °C for 30 min or at 450 °C for 96 h. At first, this seems to contradict the results discussed above as the preference observed in this work is opposite.

For the next stage, the same spectral analysis was applied to the Raman data gathered from samples oxidised at normal pressure for 20 min, 1 h, 5 h, 10 h and 72 h. The results for two samples exposed to oxygen for 20 min and 72 h are shown

| \(\text{WO}_2\) | \(\text{tri-WO}_3\) | \(\text{m-WO}_3\) | \(\text{o-WO}_3\) | \(\text{h-WO}_3\) |
|---|---|---|---|---|
| this work | 41 | 650 K | 41 | RT |
| 75 | 70 | 73 |
| 87 | 82 | 84 |
| 102 | 118 | 116 |
| 128 | 125 | 123 | 126 | 134* | 137 |
| 165* | 171* | 176 |
| 186 | 186 | 184 |
| 185 | 199 | 200 |
| 218 | 216 | 217 |
| 245 | 247 |
| 263 | 263 | 265 | 275* |
| 285 | 278 | 274 | 301 |
| 330 | 324 | 329 | 325 | 336 |
| 345 | 352 | 354 |
| 377 | 373 |
| 423 | 423 | 429 |
| 473 | 479 |
| 512 | 574 |
| 595 | 606 |
| 615 | 640 | 646 | 653 |
| 696 | 680 | 690 | 692 |
| 708 | 718 | 704 | 708 |
| 730 |
| 777 | 805 | 808 | 791 | 807 | 820 | 810 |
| 834 |
| 950 |

* Multiple overlaid peaks
Figure 6: Raman spectra for a variety of crystalline tungsten oxides: hexagonal WO$_3$ (h–WO$_3$) [41, 42]; orthorhombic WO$_3$ (o–WO$_3$) [41, 43]; monoclinic WO$_3$ (m–WO$_3$); triclinic WO$_3$ (tri–WO$_3$) [41]; and monoclinic WO$_2$.

Figure 7: Exact overlays of the EBSD IPF z map (left column) and the intensity map of the principal $\nu$(O – W – O) Raman peak (right column) for short oxidation: (a) and (b) – Sample 4 ($p_{Ar-O_2}$ = 0.1 bar); (c) and (d) – Sample 8 ($p_{Ar-O_2}$ = 0.2 bar). In these cases, the preference on [1 1 1] type orientations grows weaker with longer oxidation time. After 72 h of oxidation, the oxide grown on the [0 0 1] type orientations appears to show a more intense Raman signal. This was the same result which was obtained by Schlueter & Balden [24]. The oxide growing on the [0 0 1] oriented grains is thicker, hence the oxide scale on the [0 0 1] oriented grains grows faster than for the [1 1 1] orientation – when the oxide is thicker than at short oxidation runs.

3.3. Tungsten oxide phase analysis

From the EBSD map for Sample 4 ($p_{Ar-O_2}$ = 0.1 bar) and Sample 8 ($p_{Ar-O_2}$ = 0.2 bar), 2 x 25 points were selected corresponding to original tungsten grains of [0 0 1] and [1 1 1] type orientation for subsequent Raman analysis. The statistical average of the Raman spectra corresponding to different orientations was calculated. The averaged spectra corresponding to each orientation are shown in Fig. 10. For oxides grown on W grains oriented on [0 0 1] at different partial pressures, a shift in the local maximum is observed. This shift in peak from 690 cm$^{-1}$ towards 710 cm$^{-1}$ is characteristic to a phase transformation from h–WO$_3$ to o–WO$_3$. Furthermore, the amplitude of the dominant 806 cm$^{-1}$ peak above the baseline for [0 0 1] type orientation increases by a much larger factor than for the [1 1 1] type orientation. The peak shift is not observed on [1 1 1] orientation either. Referring to Fig. 6, the amplitude of the o–WO$_3$ 806 cm$^{-1}$ peak is three times the amplitude of the corresponding h–WO$_3$ peak. For the [0 0 1] type orientation, the presence of the shift in local maxima combined with the pronounced increase in intensity of the Raman signal is a strong
The formation of metastable $h$–WO$_3$ on [001] type grains might explain the sluggish growth during the onset of oxidation. The Gibbs free energy cost of its formation relative to the thermodynamically stable $o$–WO$_3$ phase scales with the thickness of the oxide and acts to decrease the stability of the $h$–WO$_3$ during growth. It is possible that the $h$–WO$_3$ phase forms preferentially on W grains oriented on [001] because of a lower crystal structure mismatch and/or favourable bonding compared to $o$–WO$_3$, thereby reducing the effective interfacial energy of the $h$–WO$_3$ bulk relative to $o$–WO$_3$ bulk interface. However, this negative interfacial energy is independent of the oxide thickness and therefore, the initial reduction in free energy costs is outweighed as the oxide grows thicker. This might explain why the $h$–WO$_3$ grows unstable at a certain thickness, transforming to the stable $o$–WO$_3$ phase.

However, the $h$–WO$_3$ to $o$–WO$_3$ transformation does not explain the more rapid growth of the $o$–WO$_3$ phase on [001] grain orientations at the late oxidation stages. For oxidation runs longer than 1 h, a colour change from yellow to blue to purple was observed (Fig. 12). The moment of this colour change seems to coincide with the observed reversal in preferential oxidation. Therefore, it is inferred that this colour change is attributed to a separate phase transformation to an entirely different oxide, and these new coloured oxides might contain valuable information into the reason for the reversal of preferential oxidation.

### 3.4. Correlating EBSD and confocal topography maps

As outlined in Sect. 2.2.1 and shown in Fig. 2 preferential polishing of softer W grains corresponding to [111] type surface orientation generates a surface topography which matches well with EBSD mapping. However, it was observed that the variation in topography as characterised by the interquartile range (IQR) initially decreases. This indicates that valley regions in the topography, which correspond to [111] type orientations initially grow more rapidly. This is in agreement with Fig. 7. However, for longer oxidation times, the variation of topography begins to increase again. This indicates that oxidation on W grains protruding from the surface and showing [001] type orientations becomes dominant over the low-lying [111] regions. This is again in agreement with the transition in the...
Figure 10: Averaged Raman spectra over 25 selected regions corresponding to different tungsten surface grain orientations for Sample 4 ($p_{Ar-O_2} = 0.1$ bar) and Sample 8 ($p_{Ar-O_2} = 0.2$ bar): (a) – oxides on $\{100\}$ tungsten grains; (b) – oxides on $\{111\}$ tungsten grains. The intensity $I$ of the main peak and its area $A$ [cm$^{-1}$] are shown alongside the corresponding spectra.

Figure 11: De-convoluted peak positions for $\{001\}$ (left column) and $\{111\}$ (right column) type grain orientations sampled over a 350 – 350 µm region for: (a) and (b) – Sample 4 ($p_{Ar-O_2} = 0.1$ bar); (c) and (d) – Sample 8 ($p_{Ar-O_2} = 0.2$ bar).
A robust method for smoothing, baseline subtraction and deconvolution based on a statistical information criterion was adopted to analyse the Raman scattering response of these oxides. Using this method, the metastable $h$-WO$_3$ phase was shown to form specifically on {001} oriented tungsten grains just at the start of oxidation and is later either substituted or covered by an α-WO$_3$ oxide layer. Tungsten grains with a {111} surface orientation showed only the orthorhombic phase both at the initiation and progression stages of oxidation.

It is still currently unclear what drives the rapid growth of tungsten oxide on {001} tungsten planes. The reversal in preferential oxidation coincides with an oxide colour change from yellow to blue to purple, and it is expected that a second phase transformation holds the answer to why this happens. Further work will involve the analysis of the composition and structure of this coloured oxide.

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**Data availability**

The raw/processed data required to reproduce these findings is stored in a Dropbox repository and may be shared on request by contacting the corresponding author. Due to technical limitations, it is not possible to upload all data to a public repository. A software toolkit implementing the methodology described for data processing in this work is available at [https://github.com/gfulton96/W_Oxidation](https://github.com/gfulton96/W_Oxidation).

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