CONFERENCE PRE-PRINT

FEASIBILITY STUDY OF TUNGSTEN-WATER/AIR REACTION IN DEMO CONDITIONS

D. CAPOBIANCO
RINA Consulting - Centro Sviluppo Materiali S.p.A
Via di Castel Romano 100, 00128 Roma, Italy
Email: damiano.capobianco@rina.org

T. BEONE
RINA Consulting - Centro Sviluppo Materiali S.p.A
Via di Castel Romano 100, 00128 Roma, Italy
Email: teresa.beone@rina.org

E. SAINSUS
RINA Consulting - Centro Sviluppo Materiali S.p.A
Via di Castel Romano 100, 00128 Roma, Italy
Email: eugenia.sainsus@rina.org

D.N. DONGIOVANNI
ENEA Fusion Technology for Nuclear Safety and Security Department, CR Frascati
Via Enrico Fermi, 45, 00044 Frascati Roma, Italy
Email: danilo.dongiovanni@enea.it

Abstract

Oxidation of tungsten (W) in the presence of water vapor (H_2O) at high temperatures is a critical concern for nuclear fusion applications. In fusion reactors like DEMO, explosion risks pose a significant challenge, especially within the vacuum vessel particularly for plasma-facing components, where hot tungsten surfaces, including dust, may interact with air and steam. Recognizing the importance of addressing nuclear safety from the earliest stages of reactor design, this issue has become a key focus within the EUROfusion program, specifically under the Work Package for Safety and Environment (WP-SAE). Air may come from a LOVA (Loss of vacuum accident) accident scenario while water from LOCA (Loss of coolant accident) scenarios. Particularly LOCA scenario led to the generation of hydrogen that added to the inventory of tritium present in the vacuum chamber and an eventually succeeding LOVA represents a risk of explosion. Moreover, exposure to such conditions can lead to the formation of volatile tungsten oxides, notably tungsten trioxide (WO_3), which poses risks due to potential environmental release. The objective of this work is to assess the key parameters that influence hydrogen production from H_2O tungsten interaction in LOCA scenarios under DEMO conditions. This includes evaluating the effects of tungsten material temperature (bulk and dust) and affected surfaces as well as conducting experiments to complement the existing experimental data. It is well known that tungsten oxidation rate expressed in terms of H_2 production flow rate as during the oxidation of tungsten in presence of H_2O vapor hydrogen is produced. The study aims to further investigate and extend the knowledge of the oxidation of tungsten-based materials in the presence of H_2O. The research focuses on examining the effect of time, temperature, and sub-atmospheric pressure on the oxidation rate of pure tungsten within the 800˚-1200˚C temperature range.

1. INTRODUCTION

The paper presents a summary of the activities conducted within the EUROfusion program, specifically under the Work Package for Safety and Environment (WP-SAE). The primary objective is to assess the key parameters influencing hydrogen generation resulting from H_2O/O_2–tungsten interactions during Loss of Vacuum Accident (LOVA) and/or Loss of Coolant Accident (LOCA) scenarios under DEMO reactor conditions. The study focuses on the behaviour of Plasma Facing Components (PFCs), and generated dust during the machine normal operation and abnormal events, investigating their response under air/steam oxidation in sub-atmospheric pressure. Particular attention is given to extreme accident conditions, with surface temperatures ranging from 800°C to 1200°C, and to the role of specific material surface area in hydrogen production.The oxidation and volatilization of tungsten in steam at elevated temperatures is a complex phenomenon governed by multiple mechanisms, which vary depending on temperature, steam pressure, and steam velocity [1].

TABLE 1. REVIEW OF KINETICS CORRELATION DEVELOPED FOR H_2O AND TUNGSTEN REACTION [2]

Reference	Kinetic corelation[gW/(cm^{2}s)]	Temperature range[K]
Kilpatrick and Lott	$0.61\ e^{-\frac{48.9}{RT}P_{H_2O}}$	1323-1723
Greene and Finfrockt	$1.89\ e^{-\frac{47.2}{RT}P_{H_2O}}$	1073-1623
Unal et al.	$10\ e^{-\frac{48.02}{RT}P_{H_2O}}$	1073-1973

The tungsten oxidation rate is commonly expressed in terms of hydrogen production flow rate, assuming complete oxidation of the reactants:

1) $\boldsymbol{W(s) + 3H_2O \rightarrow WO_3(s) + 3H_2(g)}$

the tungsten oxidation rate was previously studied and published by Sabourin [2] as reported in the TABLE 1.
All these relationships consider the oxidation rate depending on temperature according to an Arrhenius type formulation, and depending on a power of H_2O pressure, $(P_{H2O})^n$, with n= 1 or close to 1. As consequence the hydrogen production rate per unit area is a function of the same variable typically described by an Arrhenius-type equation [3] [4], [5], [6]. A kinetic study [4] covering 500–1300°C concluded that no single rate law can fully describe tungsten oxidation. Three growth-rate laws are considered:

— Logarithmic law: $\mathbf{M = k \cdot \log(\log(t - t_0)) + A}$

— Parabolic law: $\mathbf{M = (k_p \cdot t)^{0.5}}$

— Linear law: $\mathbf{M = k_l \cdot t}$

At temperatures below 650° which are not of the interest of the study the oxidation is boundary-controlled, forming a thin WO_2-like layer. Oxidation follows a logarithmic law until ~0.1 μm thickness. At higher temperatures parabolic and linear law are sequential. Parabolic law applies to compact lower oxide layers that act as diffusion barriers. The linear law describes open-structured oxides where oxygen access is unrestricted. A mixed parabolic-linear behavior is observed when the inner oxide layer hasn't reached its maximum thickness; cracking due to stress (from the high-volume ratio of WO_3/W, ~3.35) transitions the growth to a linear regime. This confirms that the oxide scale is non-protective, and volatilization continuously exposes the metal surface.
According to Belton [5]. Gaseous species such as $WO_3 \cdot H_2O$ may form via:

2) $\boldsymbol{WO3\,(s) + H_2O\,(g) \rightarrow WO_3\,H_2O\,(g)}$

Other reactions include:

3) $\boldsymbol{W(s) + H_2O \rightarrow WO\,(s) + H_2\,(g)}$
4) $\boldsymbol{WO(s) + 3H_2O\,(g) \rightarrow WO_3\,H_2O\,(g) + 2H_2\,(g)}$
5) $\boldsymbol{W(s) + 3H_2O\,(g) \rightarrow WO_3\,(s) + 3H_2\,(g)}$
6) $\boldsymbol{WO_3\,(s) + H_2O \rightarrow WO_3\,H_2O\,(g)}$
7) $\boldsymbol{W(s) + 4H_2O\,(g) \rightarrow WO_3\,H_2O\,(g) + 3H_2\,(g)}$

Rate-determining steps involve oxidation of WO_2(s) to volatile $WO_2(OH)_2$ and WO_3 polymers:

8) $\boldsymbol{W(s) + 2H_2O\,(g) \rightarrow WO_2\,(s) + 2H_2\,(g)}$
9) $\boldsymbol{WO_2\,(s) + 2H_2O\,(g) \rightarrow WO_2(OH)_2\,(g) + H_2\,(g)}$

A complete phase diagram for the tungsten-oxygen system remains only partially available due to its complexity, with a dozen oxides reported in the literature. The most well-defined tungsten oxides include grey $WO_{1.7}$, brown WO_2, blue W_2O_5, and yellow WO_3, as shown in TABLE 2 [6]. Transformations in WO_3, indicated by discontinuous property changes during heating, are particularly significant due to their association with non-protective behavior [3].

TABLE 2 CHARACTERIZATION OF THE TUNGSTEN OXIDES ACCORDING TO BERGER ET. AL. [6]

Phase	Characteristic oxide color	Rage of phase stability of the oxides
α	Yellow	WO_3 to $WO_{2.9}$
β	Blue	$WO_{2.9}$ to $WO_{2.8}$
γ	Violet	$WO_{2.8}$ to $WO_{2.2}$
δ	Brown	$WO_{2.4}$ to $WO_{1.}$
ε	Gray	$WO_{1.7}$ to W
γ- δ	Two-phase region	$WO_{2.4}$ to $WO_{2.2}$
δ- ε	Two-phase region	$WO_{1.7}$ to $WO_{1.0}$

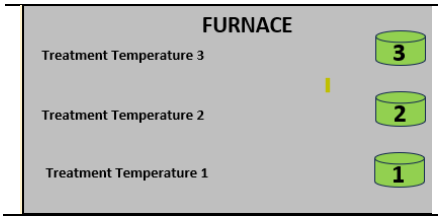
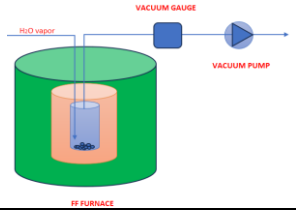
Volatilization increases with higher H_2O content and elevated H_2 partial pressure. However, volatilization in steam, especially under sub-atmospheric conditions, remains insufficiently studied. Based on actual knowledge the was necessary to investigate further.

2. EXPERIMENTAL

The study aims to understand mechanisms that lead to the oxidation of tungsten, especially in the early stages, the phases produced and the influence of the specific surface (Dust and Massive samples). Preliminary attention will be given to quantifying the rate of hydrogen production in an oxidizing atmosphere composed of argon and up to 70% water vapor at fixed flow rate (40 Nl/h). These tests recreate the partial vapor pressure conditions that may occur during an accidental scenario, to simulate environments where partial pressures and vapor content play a

significant role in reaction dynamics. Influence of the physical characteristics of tungsten samples specifically comparing powdered and bulk tungsten forms will be examined. Lastly, the study will explore the volatilization behaviour of tungsten oxides and hydroxides. Understanding how these compounds form and evolve, and how their presence influences the hydrogen production rate, is essential for interpreting the overall reaction mechanism and for anticipating potential material losses or contamination. Morphology and chemical composition of the oxide formed in the oxidation of Tungsten at different temperatures will be investigated through different technique analysis (SEM, LECO, optical microscope, XRD). Hydrogen production rate will be measured online with micro gas chromatograph (micro-GC) apparatus and evaluate different information from the analysis of the sample after tests (i.e. scale thickness, Oxygen content, mass gain, etc.) to verify the possibility to derive the Hydrogen production rate as indirect measurements after test. Firstly TGA (Thermogravimetric analysis) under atmospheric pressure and long exposure time was conducted for characterization of the tungsten oxidation. The aim of the tests conducted at constant temperatures was to evaluate the oxidation rate over time, to identify the onset of volatilization phenomena that lead to oxide loss and determine the temperature at which this occurs, and to assess the characteristics of the oxides formed after the oxidation process. Secondarily two experimental set-ups in different furnaces have been developed to conduct oxidation tests at high temperatures both under atmospheric and sub-atmospheric conditions for shorter oxidation time to characterize the oxidation phenomenon in the first stages of the exposure to oxidant agents in second phase TABLE 3. First set-up was useful to evaluate the influence of temperature on samples of equal size and under the same oxidizing conditions; in this specific case, furnace FV (Vertical Furnace) control the temperature in three different zones (800˚-1200˚C temperature range). Second set-up consist of single heating zone (800˚-1600˚C temperature range) furnace FF (Melting Furnace) equipped with a special reactor design to conduct test under sub atmospheric pressure. Experiments werebconducted by continuously aspirating a quantity of humidified gas to keep the reactor below atmospheric pressure using a vacuum pump.

TABLE 3 EXPERIMENTAL SETUP USED FOR STUDYING THE OXIDATION OF TUNGSTEN IN THE PRESENCE OF WATER VAPOR.

Atmospheric installation set up condition	Setup for sub-atmospheric dynamic conditions
	

The construction of the reactor required an in-depth study relating to the choice of construction materials and welding, the internal lining, its temperature and the partial pressure at the bottom of the reactor where the sample is placed. To prevent the inlet of air during steam oxidation tests.

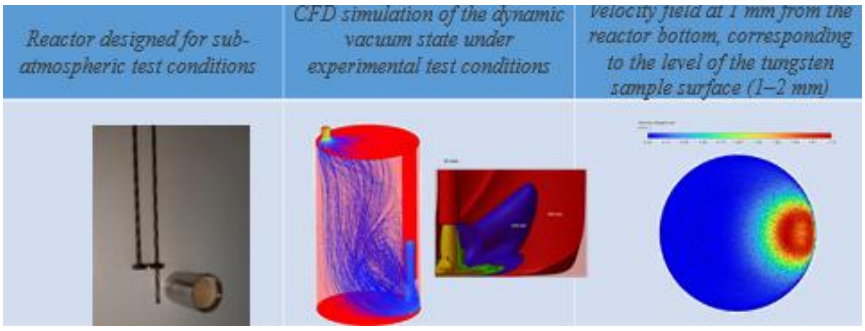


FIG. 1 Reactor and its CFD model for sub-atmospheric test

The construction of the reactor required an in-depth study relating to the choice of construction materials and welding, the internal lining, its temperature and the partial pressure at the bottom of the reactor where the sample is placed. To prevent the inlet of air during steam oxidation tests.
The reactor is built in AISI 316, which ensures good resistance at temperatures up to 1200 °C in protective atmospheres and is coated in alumina. The coating ensures that the online measurement of hydrogen made with a microGC is not distorted by the oxidation of the reactor material. CFD simulations were performed to assist the reactor design phase and to validate the oxidation tests by analysing the fluid dynamics condition within the reactor

(FIG. 1). These simulations provided insights into key parameters such as flow velocity, temperature distribution, and partial pressure, helping to characterize the reactor's fluid dynamic regime. This analysis was crucial for assessing the influence of flow conditions on the oxidation process. As shown in FIG. 1 the Flow under sub-atmospheric conditions correspond to convective regime and influence the tungsten oxidation rate.

2.1. Experimental activities on tungsten oxidation

The oxidation tests were performed on various tungsten samples in different forms, including a massive cylinder produced from powders through isostatic pressing and sintering with a diameter of 3.5 mm, massive samples with diameters ranging from 1 to 2 mm, and powder with an average particle size of 12 µm. TABLE 4 report the samples before the oxidation test and its specific surface area of the three different samples measured geometrically for the massive cylinder and using BET analysis for 1-2 mm massive samples and 12µm. powder.

TABLE 4 TUNGSTEN SAMPLES USED FOR OXIDATION TEST

Massive cylindric samples	Massive samples **Ø** 1-2 mm	12 µm powder
		
SSA 7.12 10^{-5} m^2/g	SSA 2.90 10^{-3} m^2/g	SSA 6.00 10^{-2} m^2/g

Massive samples commonly used oxidation tests and TGA. Dust samples were used in oxidation tests.

2.1.1. Preliminary TGA

Thermogravimetric tests were performed at constant temperature on tungsten samples. Initially, the sample is heated in an inert atmosphere up to a set temperature, then exposed to a controlled mixture of H_2O and Ar to initiate oxidation, which is detected by a weight increase. A small apparent weight gain in the inert phase is due to buoyancy changes as gas density decreases with temperature. Once oxidation begins, the mass gain is linear over time, indicating a constant oxidation rate under fixed temperature and H_2O pressure.

TABLE 5 EXPERIMENTAL MATRIX OF THE OXIDATION TESTS PERFORMED ON DIFFERENT TUNGSTEN SIZE SAMPLES IN TG; FLOW 40 NL/H

Nr.	Type of samples	Temperature [°C]	% Water vapor	Reaction time [s]
1	Massive Cylinder	800	30-70	6000
2	Massive Cylinder	1000		
3	Massive Ø 1-2 mm	800		
4	12 µm powder	800		
5	12 µm powder	1000		
6	12 µm powder	1200		

FIG. 2 shows two examples of weight gains of massive sample of the same mass and size, recorded at 800 °C and 1000 °C (mixture H_2O 30% and Ar 70 % at atmospheric pressure). In both cases the mass gain is linear with time for more than 100 minutes. Therefore, the oxidation rate is constant in time, at constant temperature. All the thermogravimetric curves in the graph shows a noise of the signal, which increases during the oxidation period. The oscillation main depends on the little variations of the weight due to oxidation, in comparison with the total weight (sample plus crucible plus holding system).

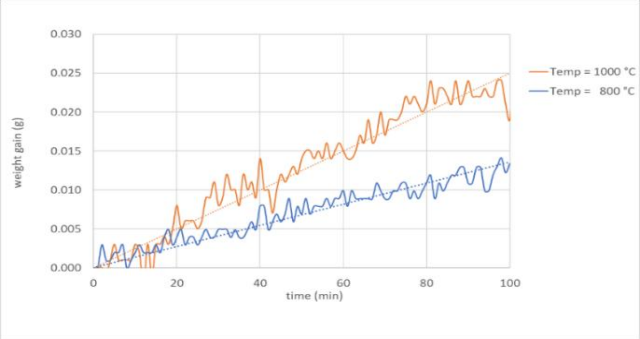
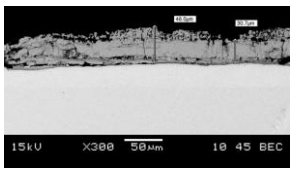
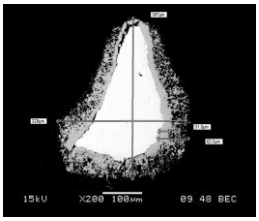
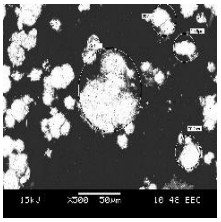


FIG. 2 Mass gain of massive tungsten samples recorded in thermogravimetric tests with mixture H_2O 30%/Ar70%, at 800 °c and 1000 °C; total pressure =1 atm.

TABLE 6 shows examples of oxides for three different tested types of tungsten materials analyzed at the SEM (Scanning Electronic Microscope) after tests in oxidizing atmosphere containing H2O 30%/Ar70%, for 100 minutes. In all cases, the former oxide results porous, not compact, not adherent to the metallic substrate, with holes and cracks. This type of oxide does not provide effective protection against further oxidation. The structure of the oxide is coherent with the linearity of the oxidation.

TABLE 6 CROSS SECTIONS SEM ANALYSIS OF ON DIFFERENT TUNGSTEN SIZE SAMPLES AND XRD ANALISIS OF THE MASSIVE CYLINDRIC SAMPLES AT 800 °C, IN OXIDIZING ATMOSPHERE CONTAINING H_2O 30%/AR70%, FOR 100 MINUTES

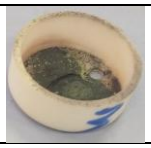
Massive cylindric samples	Massive samples **Ø** 1-2 mm	12µm powder	X-rays diffraction analysis
			
SSA 7.12 10^{-5} m^2/g	SSA 2.90 10^{-3} m^2/g	SSA 6.00 10^{-2} m^2/g	Oxide formed on a massive sample of tungsten at 800°C, for 100 minutes

To evaluate the composition of the formed oxide, X-rays diffraction analysis has been carried out, this analysis demonstrate that the formed oxide is tungsten trioxide, WO_3, which is the oxide with the maximum ratio O/W. The formation of the oxide at maximum oxidation degree confirms that the formed oxide does not form a dense oxide layer reducing the diffusion of oxygen and favouring the formation of defective oxides. On the contrary the oxygen can reach rapidly the metallic Tungsten, which is completely oxidised to WO_3.

2.1.2. H_2O oxidation influence of specific surface

After verifying that the results of the preliminary TGA tests align with findings in the literature specifically that prolonged exposure (over 1 hour) of tungsten samples to oxidizing atmospheres leads to significant oxidation this second experimental campaign was designed to investigate the oxidation mechanisms of tungsten under short exposure times, comparable to the initial stages of LOCA scenarios. Two types of tungsten grain size particle underwent an oxidation phenomenon at different temperatures and different vapour content in the oxidizing atmosphere. Experiments were conducted at two different test durations (10 and 20 minutes). The samples were subjected to varying vapor concentrations (16%, 31%, 38%, 47%, and 70%). Oxidation tests were conducted on tungsten powder (12µm–1-2 mm) at three temperatures: 800°C, 1000°C, and 1200°C. The matrix in TABLE 7 explores the influence of temperature and water vapor concentration on oxidation kinetics, maintaining consistent flow conditions. Based on Berger et al.'s [6] tungsten oxide characterization, a comparison was made with oxidation test results for 12µm samples, yielding the following findings.

TABLE 7 COMPARISON OF THE OXIDES FUNDED DURING THE OXIDATION OF THE 12MM DUST AND ITS CLASSIFICATION ACCORDING TO THE BERGER ET AL [6] AND CONDITION WHICH LEAD TO ITS FORMATION DURING THE EXPERIMENTAL CAMPAIGN.

	ε WO to $WO_{1.7}$	δ WO to $WO_{2.4}$	γ $WO_{2.2}$ to $WO_{2.8}$	α to $WO_{2.9}$
t				
600[s]	800 °C - 16%	800 °C - 30 % 1000°C – 16 % 1200°C -16%	800 °C - 47 % 1000 – 47 % 1200°C -30%	
1200[s]				1200 °C 70 %

There was observed that increasing the water vapor content in the oxidizing atmosphere significantly influences the formation of higher tungsten oxides. At 800 °C and with 16% water vapor, there is a notable tendency for the formation of sub-stoichiometric tungsten oxides such as WO, $WO_{1.76.}$ When the water vapor concentration increases to 47%, the oxides reach even higher states of oxidation $WO_{2..2}$ to $WO_{2.8}$ (e.g., 10 minutes). This phenomenon becomes even more pronounced with longer exposure durations, eventually leading to the formation of fully oxidized tungsten trioxide (WO_3), representing the highest oxidation state of tungsten. For what concern 1-2 mm the highest oxidation state is not reached for shorter oxidation duration time, further investigations are ongoing to compare the influence of specific surface area with oxidation behavior.

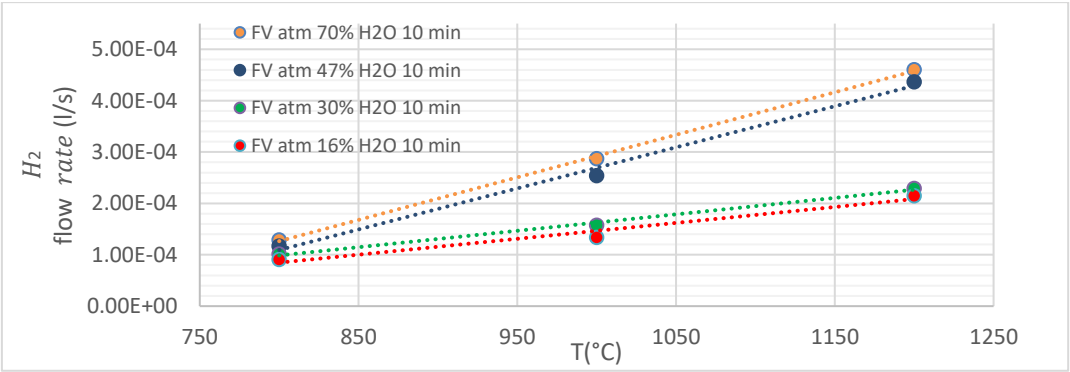


FIG. 3 Hydrogen production at varying temperature and water vapor content (70%, 47%, 30% and 16%) with 12mm sample

As discussed previously, the hydrogen production derived from tungsten oxidation process is strictly influenced by the temperature, vapor partial pressure and time exposed to the oxidative atmosphere. In FIG. 3, it is observed that an increase in water vapor content leads to a corresponding rise in hydrogen production. Specifically, at 800°C, the hydrogen flow rate remains almost constant across the studied conditions of 16%, 30%, 47%, and 70% water vapor, indicating that at lower temperatures, the oxidation process is minimally affected by the water content for short time exposure. However, at higher temperatures of 1000°C and 1200°C, there is a significant increase in hydrogen production.

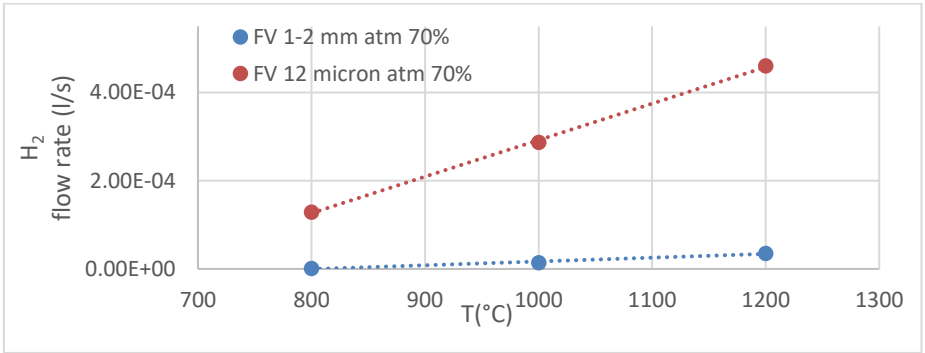


FIG. 4 Comparison of the Hydrogen production at varying temperature, at 10 minutes while maintaining constant the water vapor content (70%,) of 12μm and 1-2 mm sample

FIG. 4 shows the produced hydrogen flow rate as a function of temperature, with two types of samples (1-2 mm, and 12µm granulometry), tested at 3 different temperatures. Therefore, it is evident that there is a greater surface area exposed to oxidation, resulting in a higher amount of hydrogen produced, which is enhanced at high temperatures.

2.1.3. Sub-atmospheric oxidation under turbulent flow regime

In quiescent conditions, the transport of matter is purely diffusive, while convective transport of matter shall be considered if the fluid is in movement. In the latter case, fluid at higher concentration is continuously in contact with the surface. The presence of a flow determines always an increase in the exchange of matter with respect to quiescent conditions. Flow of mass is written according to the following relation:

$$n = K_c(C_\infty - C_s)$$

Where K_C is the convective mass exchange coefficient. Two parameters of interest with respect to their effects upon the rate of vaporization of tungsten-metal steam are the Reynolds number of the steam flow and the hydraulic diameter of the flow path. Mass transfer coefficient in turbulent flow is described with Sherwood (Sh) number that dependent -upon Reynolds number and Schmidt (Sc) number cconsidering Buckingham's theorem.

$$Sh = \frac{K_c d_h}{D} = f(Re; Sc) = CRe^x Sc^y$$

Sh represents a factor of exaltation of the transport of matter due to convection. In convective transport Sh>1 and always determines an increase in the mass flow compared to the diffusive case. The Schmidt number contains only physical parameters of the fluid, in particular viscosity, density, and diffusivity. In Laminar flow Sh number is equal to a constant independent to Reynolds (Re) number Sh = K. Mass transfer coefficient in laminar flow is a constant, dependent only upon diffusion coefficient D of the fluid and the hydraulic diameter δ. As reported by Unal et al [1] experimental data from Smolik et al. (1989) [7] and Smolik and Coates [8], Kilpatrick and Lott (1966) [9], and Elrick et al. (1995) [10] were produced at very low Reynolds numbers this leads to have a Sherwood number Sh = 1 indicating that the process is diffusion control and that there are no convective effects on the mass transfer.

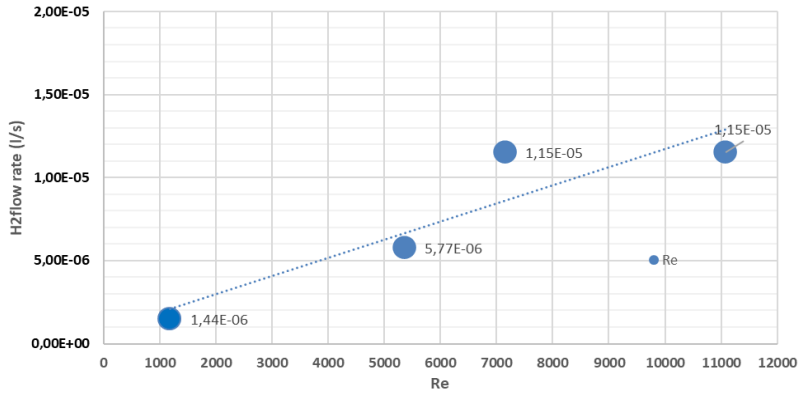


FIG. 5 Comparison of hydrogen production from tungsten oxidation vs Reynold number of different dynamic vacuum conditions

From the first results of the tests, conducted at 1000 °C at 70 % of water vapor content, reported in FIG. 5 it is evident how the flow regime affects the oxidation of tungsten and obviously the production of hydrogen. Further tests will be planned to validate the first results and expand the set of experimental tests.

3. CONCLUSION

Preliminary tungsten oxidation tests with air and with vapor H_2O in dedicated apparatus were performed at atmospheric and sub-atmospheric pressure. These tests gave first qualitative and quantitative indications about the oxidation phenomena and H_2 production rate and suggestions for further tests. Several factors must be considered in developing a unified understanding of the overall tungsten oxidation process: The oxide layer observed in each studied cases results porous, cracked and non-protective. In this case the oxidation rate is constant, the oxidation is linear with time, because the oxide layer is not a barrier. The controlling step is the surface reaction (including the steps of H_2O dissociation, adsorption, absorption and direct reaction of O and W). The oxidation is strongly dependent on treatment temperature and vapor partial pressure in the oxidizing atmosphere. The highest oxidation state is reached for dust after 20 minutes in higher water vapor partial pressure atmosphere and for massive samples it was needed longer exposure time higher than 1 hour. Future work will be done to enlarge the range of conditions (total pressure, H_2O partial pressure and temperature) and to explore the influence of surface characteristics on the oxidation rate. The sub- atmospheric tests will be carried out also in

batch conditions to study diffusive mass transfer phases. Moreover, other tests will be performed to study oxidation at different flow conditions (laminar flow vs turbulent flow) and to study air oxidation.

4. FURTHER INFORMATION

4.2. Author affiliation

RINA Consulting - Centro Sviluppo Materiali S.p.A; ITALY; damiano.capobianco@rina.org
ENEA Fusion Technology for Nuclear Safety and Security Department; danilo.dongiovanni@enea.it

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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The views and opinions expressed herein do not necessarily reflect those of the European Commission

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