Fusion Engineering and Design 127 (2018) 120–126

Contents lists available at ScienceDirect

Fusion Engineering and Design

journal homepage: www.elsevier.com/locate/fusengdes

Studies on synthesis of plasma fusion relevant tungsten dust particles and measurement of their hydrogen absorption properties

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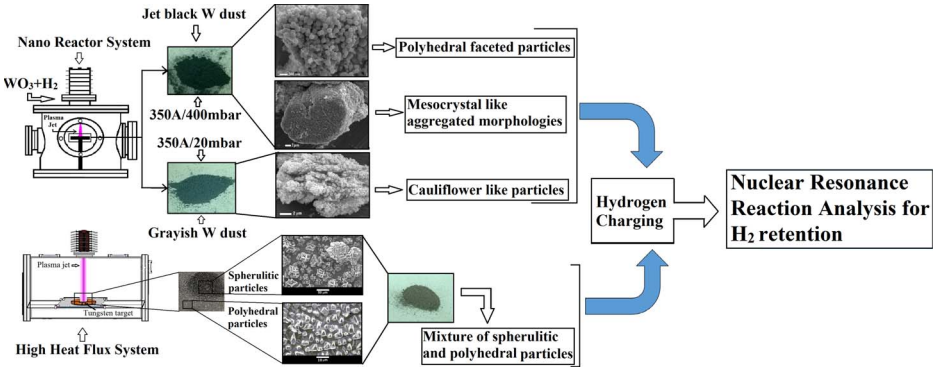
GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:
Tokamak
Plasma material interaction
Tungsten
Dust particles
Hydrogen retention
Nuclear reaction analysis

ABSTRACT

In this communication, we identify some thermal-plasma assisted synthesis techniques for the alternative production of fusion relevant tungsten dust particles in bulk amounts, to allow for the statistically meaningful study of their hydrogen absorption properties. Fast, single step synthesis of fine particles in the α-tungsten phase was demonstrated, using a high heat flux device for sizes in the micrometer region and an experimental nanoparticle reactor for dust with nanometer sizes. Production of both equilibrium polyhedral and nonequilibrium spherulitic crystal shapes was demonstrated, dust morphologies similar to which were identified before in existing tokamaks and divertor simulator systems. Their hydrogen retention characteristics were measured by the Nuclear Resonance Reaction Analysis technique, which was found to be orders of magnitude higher compared to the same metal in the bulk form. The mesoporous crystals generated during high power, high pressure synthesis conditions were assumed to be responsible for the unusually high gas retention of the nanoparticle sample. It was concluded that non-equilibrium tungsten dust morphologies would be dominant in the high power fusion machines in the future, which may aggravate the hydrogen isotope retention issues further.

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https://doi.org/10.1016/j.fusengdes.2017.12.005
Received 25 August 2017; Received in revised form 1 November 2017; Accepted 7 December 2017

1. Introduction

In magnetic confinement fusion devices such as the ITER tokamak, interactions between the intense plasma and the wall, made of tungsten in the high heat flux region of the divertor, might lead to the creation of fine dust particles, with sizes from few nano to several tens of micrometers [1]. Extreme deposition of heat, for example, during an Edge Localized Mode (ELM), a disruption, or arcing may lead to melting or even evaporation of tungsten that may later condense as fine tungsten dust particles. Tungsten in atomic form may also be released through the process of physical sputtering. These particles may lead to contamination of the hot plasma by high-Z impurities, and also absorb tritium gas from the fusion environment. In ITER, tritium (T) retention shall be limited to 1 kg in-vessel, and it is, therefore, important to quantify the relative importance of the different possible T retention mechanisms [2]. In addition, in future fusion reactors, tritium retention will negatively impact the efficiency of the fuel cycle. It is therefore important to understand the hydrogen retention characteristics of the dust particles that are typically produced inside fusion machines [3]. Bernard et al. have pointed out that the total amount of tungsten dust produced in existing tokamaks is actually insufficient to carry out such elaborate studies, hence justifying the use of alternative production routes for fusion dust like tungsten nanoparticles they utilized a pulsed laser ablation technique, whose tritium retention/release characteristics were also studied [4]. In the same endeavor, to produce tungsten dust like nanoparticles with fusion relevant sizes and with the widest variety of morphologies possible, Sarah Dine et al. explored several synthesis methods including ball-milling, reactive ball-milling, self-propagating high-temperature synthesis (SHS) and mechanically activated SHS (MASHS) techniques [3]. Grisolia et al. had used planetary ball-milling or magnetron sputtering systems for the synthesis purpose [5–7]. For an advanced understanding of their retention behavior, it is required to first identify robust synthesis techniques for the production of ITER relevant dust particles in sufficient amounts to allow for the statistically meaningful study of their hydrogen absorption properties. Towards that end, in this communication, we report utilizing some thermal plasma mediated synthesis techniques, which as the processing medium has the distinction of reproducing tokamak Divertor like parameters in the first place.

For the synthesis of fusion relevant tungsten dust like superfine particles in the nanometer size range, we have used an expanding plasma assisted synthesis technique, using pressure in the sample collection chamber and plasma current (power) as the experimental variables, through which we had before demonstrated fine control over the powder properties [8]. For generation of tungsten dust particles in the micrometer size range, we have utilized samples generated during a PSI experiment in the CPP-IPR High Heat Flux (HHF) device, experimental details of which we already had reported before [9]. As for the measurement of their hydrogen isotope retention characteristics, we have here used simple hydrogen gases only. Tanabe in his review on the absorption of hydrogen isotopes by tungsten materials has suggested that the relevant properties may not vary much among different isotopes [10]. The measured retention values are utilized primarily for comparison only among different particle morphologies and sizes generated through this series of experiments.

2. Materials and methods

2.1. Synthesis by segmented plasma torch assisted nanoparticle reactor

The experimental nanoparticle reactor along with the essential peripherals was described in details in a recent communication [11]. A segmented arc was used, which was utilized before to reproduce ITER Divertor like plasmas, characterized by very high ion density ($\sim 10^{20}\,m^{-3}$), extreme ion ($\sim 10^{24}\,m^{-2}s^{-1}$) and heat ($\sim 10\,MWm^{-2}$) flux parameters, and plasma temperature of few eVs [12]. Argon was injected at the rate of 15 liters per minute (lpm) near the cathode using an Alborg digital mass flow controller. The reactant (tungsten-oxide, Alfa Aesar, 99.8% purity) in micro-particle form was injected into the plasma with a particle feeder at a rate of 0.4 g/min. Hydrogen was injected at the same location at 5 lpm using mass flow controllers through the powder feeder. A high throughput roots vacuum pump (EH 4200 backed with an E2M 275 rotary vacuum pump) was utilized, which allowed reducing the ambient gas pressure in the sample collection vacuum chamber down to the sub-mbar level. The synthesis experiments were repeated for three different plasma currents: 150 Amp (7.5 kW), 250 Amp (15 kW) and 350 Amp (23 kW), and different pressure in the sample collection chamber as follows: 400, 20 and 0.8 mbar. The tungsten samples were collected from a substrate kept at a distance of 150 mm from the reactor nozzle. The plasma emission collected from the reactant injection region was analyzed with a McPherson 1.33 m spectrometer (1800 grooves/mm) and CCD camera (Andor DU940P, 2048 × 512 pixels). The plasma temperature and density were estimated by line ratio intensity and Stark broadening (H_β) techniques as detailed elsewhere [11].

2.2. Synthesis experiments in the CPP-IPR high heat flux device

A polycrystalline tungsten plate (99.7% pure, 2.5 cm square sides, 0.3 cm thick) from Plansee was exposed to a collimated argon plasma jet (300 A, 25 lpm argon, 9.3 MW/m^2 incident flux, and 20 mbar chamber pressure) for 30 min, during which the bulk target temperature was estimated as 4600 K. Melting of tungsten and its later condensation in this process was seen to lead to the growth of hierarchical, vertical microstructures at the central exposed region, whereas polyhedral microparticles were seen depositing on the peripheral areas, which were reported before in an earlier publication [9]. The corresponding plasma parameters in the HHF system as measured by OES techniques were as follows: plasma density $2.5 \times 10^{20}\,m^{-3}$ and temperature 0.3 eV. In this current communication, we report on the Nuclear Resonance Reaction Analysis (NRRA) measurement for one of the samples from these tungsten microstructures, which were collected by rubbing off the powders from the exposed area.

2.3. Microstructural characterization of the samples

The crystallographic phase analysis was done using an X-ray diffractometer (XRD, Rigaku, TTRAX-III, 5 kW, Cu Kα radiation). The crystallites size was estimated from the diffraction peak broadening using Scherrer's equation. The morphology of the particles at a relatively bigger scale was investigated by a field-emission scanning electron microscope (FESEM, JEOL JSM-7100F). The Energy Dispersive X-ray Spectroscope (EDX) attached with this system was utilized for the bulk elemental composition of the material samples. For detailed morphological analysis of the samples at nanometer resolution, we had utilized an FEI, Tecnai G^2 F30, S-Twin HRTEM equipped with a Gatan Orius CCD camera, operating at 300 KV. This had a high angle annular dark field (HAADF) detector attachment from Fischione (Model 3000), to perform high angle annular dark-field studies. This also had an EDX attached, which made possible quantitative elemental analysis of the particles with energy resolution ~130 eV, using Tecnai Imaging and Analysis (TIA) software from FEI. For all HRTEM measurements, the samples were first sonicated in ethanol and then deposited on carbon-coated copper grids for imaging. Thermal properties have been investigated using thermogravimetric analyser (TGA); model STA 7200, Hitachi, Japan. For this measurement, the sample was heated in an oxygen atmosphere (100 ml/min) up to 800 °C at a rate of 10 °C/min. Surface chemistry of few as synthesized samples was checked by X-ray photoelectron spectroscopy (XPS, VSW, UK machine using Al Kα radiation at 1486.6 eV). The surface area of these samples was measured by BET technique in a static volumetric adsorption system surface area analyzer using N_2 adsorption/desorption isotherms (Micromeritics