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ScienceDirect Acta Materialia 84 (2015) 305–316



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# Evolution of the emissivity of tungsten at high temperature with and without proton bombardment

E. Brodu,<sup>a,b</sup> M. Balat-Pichelin,<sup>a,\*</sup> J.-L. Sans<sup>a</sup> and J.C. Kasper<sup>b</sup>

<sup>a</sup>Laboratoire Procédés, Matériaux et Energie Solaire, PROMES-CNRS, 7 rue du Four Solaire, 66120 Font-Romeu Odeillo, France <sup>b</sup>Harvard-Smithsonian Center for Astrophysics, 60 Garden Street, Cambridge, MA 02138, USA

> Received 29 September 2014; revised 15 October 2014; accepted 21 October 2014 Available online 26 November 2014

**Abstract**—The Solar Probe Plus mission (NASA) will be the first mission to enter the solar corona. The spacecraft will orbit the Sun at 8.5 solar radii from the Sun's surface at closest approach. Some metallic parts of the two on-board instruments, SWEAP (a Faraday cup) and FIELDS (antennas), will directly face the Sun, while the rest of the payload will be protected by a heat shield. For application to these instruments, a candidate refractory material, tungsten, was studied, confronting conditions similar to the ones expected close to the Sun: high radiative flux leading to high temperatures (1100–2500 K) and proton bombardment (1 and 4 keV;  $10^{16}$ ,  $10^{17}$  and  $10^{18}$  ions  $m^{-2} s^{-1}$ ) to simulate the solar wind in high vacuum ( $10^{-4}$  Pa). Total directional and hemispherical emissivities in the two wavelength ranges 0.6-2.8 and  $0.6-40 \mu$ m were recorded in situ during treatments. Material characterization was performed before and after each high temperature and bombardment experiment to correlate a possible emissivity evolution to other material properties: mainly the microstructure and the surface topography. This paper reports some results on the evolution of the emissivity at high temperature for two different tungsten materials elaborated by two manufacturers – having thus different initial surface states, impurity contents and microstructures – and also with the addition of proton bombardment to the high temperature. However, the proton bombardment showed no effect on the surface topography or the emissivity, despite the fact that the ion fluxes used in our experiments were up to three orders of magnitude higher than the one expected from the solar winds.

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Keywords: Emissivity; Tungsten; High temperature; Proton bombardment; Surface topography

### 1. Introduction

Solar Probe Plus (NASA, launch planned for 2018) will approach the solar corona at  $\sim 6.6 \times 10^6$  km (8.5 solar radii,  $R_s$ ) from the Sun's surface into a region that no other spacecraft has ever encountered, in order to help solve two scientific mysteries: the heating of the solar corona and the acceleration of the solar wind. A thermal protection shield will protect the payload of the probe from the thermal radiation and solar wind. Nevertheless, two metallic parts of on-board instruments will directly face the Sun to perform in situ measurements: SWEAP (Solar Wind Electrons Alphas and Protons investigation, led by the Smithsonian Astrophysical Observatory (SAO), Univ. Harvard, USA) and FIELDS (Electromagnetic Fields investigation, led by the Space Science Laboratory (SSL), Univ. Berkeley, USA). The behavior of the metallic surfaces in these very specific and harsh conditions was studied at the PRO-MES-CNRS laboratory, where conditions similar to the ones expected near the Sun can be reproduced: high temperatures by using concentrated solar radiation and proton bombardment by using an ion gun.

The emissivity of the metallic surfaces directly exposed to the Sun's radiation is one of the decisive material properties for these two instruments. Indeed the total emissivity, along with the solar absorptivity, will govern the thermal equilibrium of these parts of the instrumentation.

Since the 1980s, many studies have been carried out on high-Z metals, at medium temperature and under ion bombardment, as candidate metals for divertor components of fusion devices, and reported in the review of Yoshida [1], but the emissivity was not a key property for this application. Also, if the environments close to the Sun and in fusion applications have a lot in common, temperatures, ion species, ion energies and fluxes are very different. For the safety of the mission, it is then crucial to run dedicated experiments on new industrial candidate metals, in the specific conditions found close to the Sun.

For tungsten, the temperature dependence of the total hemispherical emissivity [2,3] and the spectral behavior [4,5] are rather well-known. However, the effect of coupled treatment (high temperature and ion bombardment) on the emissivity remains an open subject. Indeed, the influence of the surface topography on the emissivity has been studied [6-8], and so was the effect of ion bombardment on the surface topography, by physical sputtering [9,10], blister formation followed by exfoliation [11] and porosity formation

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<sup>\*</sup> Corresponding author. Tel.: +33 4 68 30 77 68; fax: +33 4 68 30 77 99; e-mail: marianne.balat@promes.cnrs.fr

[12], but to our knowledge only one study tried to directly link the ion bombardment to the emissivity and the relationship remained unclear [13].

## 2. Experimental set-up

The MEDIASE facility (Fig. 1) has been designed and instrumented to reproduce the conditions expected near the Sun [14]. This test facility is placed at the focus of the 1 MW solar furnace in Odeillo, France. The tungsten samples (40 mm diameter, 2 mm thickness) are heated up to 2500 K using the concentrated solar radiation, through a hemispherical silica-glass window (35 cm in diameter) placed in front of the chamber. The use of concentrated sunlight presents three main advantages in comparison to more traditional heating systems: the temperature variation of the sample follows usually within a few seconds the opening of the doors as there is no thermal inertia to this system beside the thermal inertia of the sample itself. Also this heating system produces no pollution on the sample as controlling the environment using glass vessels is easy. Lastly, such a system makes it easy to keep the temperature-sensitive in situ instruments inside MEDIASE close to room temperature, although they are at very short distance from the sample. The MEDIASE set-up is composed of a chamber  $(0.06 \text{ m}^3)$  equipped with a turbo-molecular pumping system to work in high vacuum up to  $10^{-4}$  Pa. The front face of the chamber and the sample holder are water-cooled, while the heat flux exchange between the heated sample and the sample holder is minimized using three ceramic/metallic needles at 120° that maintain the sample. The facility can be instrumented to perform the following measurements, all on the back face of the sample: temperature, directional total or spectral, or in narrow ranges emissivity using a Heiman KT4 radiometer, mass spectrometry and mass loss rate via a quartz crystal microbalance. The temperature is measured using a bi-color optical fiber pyroreflectometer developed at PROMES-CNRS [15]. Finally, tubes at 30° and 45° to the normal of the sample surface have been placed around the chamber to accommodate the treatment instruments: the Thermo-Fisher EX05 ion gun (to simulate the solar wind) and the Omicron HIS13 vacuum ultraviolet (VUV) source (not used in this study, but whose purpose is to add VUV to the protocols for the study of ceramic materials, in order to reproduce the Sun's spectrum, particularly the simulation of the H Lyman  $\alpha$  line at 121.6 nm). The flux and the size of the ion beam hitting the sample were determined in situ by using a Faraday plate collector.



Fig. 1. Scheme of the MEDIASE facility in the emissivity measurement configuration.

Before performing emissivity measurements with MEDIASE, the radiometer is first calibrated on a blackbody. This calibration is done in a way that the entire optical path of the MEDIASE chamber is reproduced on the calibration bench. The bi-color optical fiber pyroreflectometer is also calibrated, but because of its complex functioning [15], it requires two separate calibrations: one on a blackbody and one on a reflectivity calibration bench using reference reflectivity samples (Labsphere references). The bi-color optical fiber pyroreflectometer, in opposition to classical pyrometers, provides the real temperature of the surface without having to measure or assume first an emissivity or use the gray-body hypothesis: by measuring simultaneously the reflectivity and the radiance of the sample at two wavelengths (1.3 and  $1.55 \,\mu$ m), it provides the real surface temperature without knowledge of the emissivity.

### 3. Test samples and protocols

The directional (measured) and hemispherical (obtained by integration of the directional data) emissivities of tungsten samples in the two wavelength ranges 0.6-2.8 and 0.6-40 µm were obtained at high temperature. A detailed description of the method for emissivity measurement can be found in previous papers [14,16]. Also, the ratio  $\alpha/\epsilon$  was approximated by dividing the hemispherical  $0.6-2.8 \,\mu\text{m}$  emissivity by the hemispherical  $0.6-40 \,\mu\text{m}$ emissivity (the validity of this hypothesis is discussed later in Section 4). The  $\alpha/\epsilon$  ratio, which is the ratio of the solar absorptivity to the total emissivity, fully determines the temperature of a free-standing surface facing the Sun in vacuum. The total emissivity in the range 8-14 µm was also recorded. The low emissivity in the IR range is specific to metals, in correlation to their high electrical conductivity; if oxidation occurs it should result in an increase of the emissivity particularly visible in the long wavelength range [17].

Two batches of tungsten samples were used in this study: a batch from Alfa Aesar (purity 99.95%) and a batch from Plansee (purity 99.98%), both purities being provided by the respective suppliers. The surface states of all the samples within a batch can be considered identical in their asreceived states. With identical surface patterns, the only difference lies in small roughness variations from a sample to another within a batch. In the Alfa Aesar batch, the RMS roughness varies in the range 1.8-2.2 µm. As for Plansee, only one sample was tested at high temperature and its RMS roughness is 1.1 µm. The emissivities of the tungsten samples from Alfa Aesar were measured at high temperature, from 1100 K up to a specific maximum temperature for each sample: 1700, 1900, 2100, 2300 or 2500 K depending on the sample. By increasing the maximum temperature from a sample to another, the goal is here to identify potential effects on the emissivity of the heat treatment itself. For this purpose, up to three consecutive heating and emissivity measurement cycles were performed on each sample up to its specific maximum temperature to identify irreversible changes of the emissivity induced by the heat treatment. The first, second and third cycles are respectively called (a), (b) and (c) on every emissivity figure. The emissivity is measured every 200 K and the temperature is typically maintained a few minutes (4-6 min) at each next temperature level, just the time to make sure that the temperature is stable and to perform the measurements. One of the

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