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A method for measuring the high temperature emittance of refractory metal surfaces

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ABSTRACT

For high temperature applications, such as lighting components or heating elements, the thermal emittance of refractory metals such as tungsten can be improved by altering the surface of the material. Laser structuring or porous sintered coatings have been shown to increase the emittance, which leads to improvements in process stability and component lifetime.

Measuring the emittance is generally performed at room temperature by reflectometry as high temperature measurement is extremely difficult. This has to be taken into account when comparing surface modifications, as emittance at application temperature might differ. In this paper, a method is described with which samples are heated by a direct current flow and the emittance is measured with an optical pyrometer by comparing temperature values with the actual surface temperature.

The method allows a comparative measurement of different surface treatments at the application temperature. Degradation of the surface emittance at the application temperature is also possible to observe. Measured values for tungsten with different surface modifications are shown and discussed.

1. Introduction

For high temperature engineering applications, the emittance is an important property. An increased emittance is often highly favourable to ensure sufficient cooling, to increase the energy efficiency or to provide stable and homogeneous process conditions. This provides benefits in very different fields of research, including the obvious thermal processes in power plants and furnaces [1–3]. In other areas, such as the aeronautical and spacecraft industry [4–8] or the lighting industry [9,10], heat is often generated as an undesired byproduct. In these applications, surfaces with increased emittance are used to remove the heat from the system to ensure longer component life time.

In this paper, two terms are used to describe the thermal radiation emitting from bodies: emittance and emissivity. While 'emittance' refers to a real body property which can be measured, the term 'emissivity' is used for the inherent material property which cannot be directly measured. In other words, the measured emittance is a product of both, the surface material and the superficial structure.

As the emittance is strongly dependent on the surface conditions, surface structure and material composition are both important factors for optical parameters. The relationship between structure and emittance has been studied in experimental investigations [1,11–14] as well as modelling approaches [2,3,15,16].

When radiation interacts with a sample, it may partially be reflected (reflection ρ), absorbed (absorption α) or transmitted (transmission τ). These characteristics are tied together by the principle of energy conservation as shown in Eq. (1).

$$1 = \alpha + \rho + \tau \tag{1}$$

According to Kirchhoff's law of thermal radiation, the emissivity ε of an opaque body (τ = 0) is equal to its absorption α at a given wavelength λ and temperature T. This can approximately be applied to obtain the emissivity via Eq. (2) where ρ is the reflectivity and β, φ specify the observing angle [17].

$$\varepsilon(\lambda, T, \beta, \varphi) = \alpha(\lambda, T, \beta, \varphi) = 1 - \rho(\lambda, T, \beta, \varphi) \tag{2}$$

The emissivity ε is defined as the ratio of energy radiated by the sample to an ideal thermal emitter, a blackbody. Therefore, real objects in use show an emissivity of $\varepsilon < 1$. While this method has its limitations because of non-ideal diffusely reflecting surfaces, it is often accurate enough to compare similar opaque bodies.

Because of the temperature dependency of ε, it would be of interest for high temperature applications (> 1600 °C), to measure different surface modifications in-situ. Since this is almost impossible to achieve, a method will be discussed in this paper to compare various surface conditions close to application temperature. For this, tungsten samples

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with different surface modifications were heated by a direct current flow up to 1800 °C. The emittance was then determined by comparing the optically measured surface temperature on the modified area of the sample with the actual surface temperature. The emissivity value stored in the pyrometer used for the optical measurement can then be altered until the two readings are equal. The ε value for which this condition is met, is the emittance of the surface. Several methods to obtain the actual surface temperature were used and are described in detail below.

2. Material and methods

2.1. Experimental setup

When measuring the high temperature emittance, special care has to be taken of how the heat is being generated and transferred to the sample surface. Simply heating the sample by thermal radiation from heating elements is not viable, because reflections of the heat emitted from the external heating elements interfere with the measurement on the sample surface. Furthermore, the sample surface needs to be accessible by optical measurement using a pyrometer. Because of the formation of oxides in air, the measurement has to be performed under inert atmosphere. Moreover in the case of tungsten, volatile oxides are formed under oxidizing conditions. By design, this excludes most available furnaces in which the sample of interest can be heated up to 1800 °C.

Argon atmosphere, direct current flow and optical accessibility were all found in a sintering press available at Plansee SE in Reutte. A FAST/SPS sintering press (DSP 515 from Dr. Fritsch) was adapted for emittance measurements as shown in Figs. 1 and 2. The sintering press was operated without powder, the ring mould and punches, but instead by using a W rod as the sample to be heated. A graphite foil was used on top and on the bottom of the sample to minimize gaps between the sample and the electrodes providing better electrical contact. As the sintering press has to be operated at a minimal load of 28 kN, the tungsten sample was designed to be 40 mm in diameter to withstand plastic deformation at 1800 °C.

As unwanted oxidation is one of the main difficulties to overcome for measuring high temperature emittance, metallic foils were added to the setup to ensure minimal oxygen uptake by the sample by reducing oxygen access (Mo foil) or by acting as an O_2-getter (Ta-foil). A graphite mat and a ring-shaped graphite casing to suppress reflections and further reduce oxygen access completed the experimental setup. Optical access to the sample was made possible by a window in the graphite casing. A picture of the sample inside the graphite casing is shown in Fig. 3. To exclude carburization of the tungsten surface, a test run with a pure W sample was performed. X-ray diffraction phase analysis did not show any sign of carburization after a complete run up to 1800 °C.

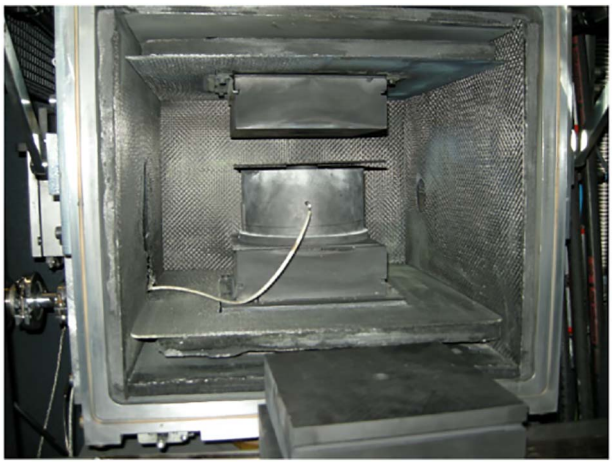

Fig. 1. Reaction chamber of a FAST/SPS sintering press adapted for the measurements.

Another challenge for investigating high temperature emittance is measuring the actual surface temperature of the sample. Two separate but both accurate temperature measurements are necessary for comparison. In principle, there are two ways to determine the sample temperature, by using a thermocouple or optically by pyrometric measurement. Both alternatives have been tested and shall be discussed in detail.

2.2. Temperature measurement

A very common method for temperature measurement is using a thermocouple. This allows for simultaneous measurement of the surface temperature and comparison to the optical signal from the pyrometer on the area of interest. The most significant advantage over optical measurements is that there are no assumptions on the material properties of the sample - such as the emissivity - needed to interpret the signal. On the other hand, measuring surface temperatures of up to 1800 °C by thermocouple is no straightforward task.

W-Re thermocouples which are suited to measure such high temperatures are very stiff and therefore difficult to bring into contact with the sample surface. Since it neither would be feasible to move a welded thermocouple together with the sample to the sintering press, nor can the connection be made in-situ, physical contact with the sample surface had to be established by contacting the thermocouple to the sample mechanically. The thermocouple was held in place with an alumina rod which was fastened to the sample with molydenum wire. This had to be done in such a way as to ensure that the connection of thermocouple wires was as close to the sample surface as possible. On top of that, the thermocouple had to be placed at the same vertical position as the pyrometer, due to a vertical temperature gradient observed on the sample. Fig. 4 illustrates the temperature measurement by thermocouple as performed for the tests presented in this paper.

In addition to bad contact of the thermocouple with the sample surface and the temperature gradient of the sample, the inherent mass of the W-Re wires and therefore their cooling effect were the main sources of error observed throughout the measurements.

Another method of temperature determination by thermocouple would be to measure the core temperature and estimate the surface temperature from that. While this would seem to eliminate the aforementioned sources of error, the surface temperature depends on the emittance of the surface itself. The influence of the surface emittance on the surface temperature is illustrated in Fig. 5. For these calculations, the core temperature of a tungsten rod with a diameter of 40 mm was fixed at 1800 °C with surface emissivities ranging from $\varepsilon = 0.4$ to $\varepsilon = 0.9$. Calculated surface temperatures range from 1760 °C to 1718 °C. In conclusion, measuring the core temperature for estimating the surface temperature of the sample requires good knowledge of the surface emittance in advance. From Fig. 5, it is also obvious how important correctly determining the surface temperature for emittance measurements is.

Instead of measuring the surface temperature by thermocouple, it can also be determined optically by pyrometric measurement. Pyrometers in use are calibrated against a cavity simulating an ideal blackbody. In order to serve as a blackbody, the cavity needs to fulfill certain conditions [18]. First and foremost, it needs to be wider than the spot size of the pyrometer. Secondly, the ratio between depth and width should be in the range of 10–12, depending on the emittance of the material inside the cavity. As a cavity like this cannot be easily drilled into tungsten, mechanical preparation on every sample would have been very expensive and was therefore avoided. Another difficulty when using this method is to compensate for thermal expansion of the sample. Because of that, the position of the cavity cannot easily be set at room temperature. On the other hand, at 1800 °C, the sample including the cavity are so bright, visual adjustments by laser optics are not viable. To compare the results to our high temperature thermocouple