Prague, Czech Republic June 21-25, 2021

Contribution ID: **245** Type: **Oral presentation**

#07-245 Development of a miniaturized furnace for Scanning Electron Microscopy: Thermal modelling, manufacturing and tests

Thursday, June 24, 2021 3:20 PM (20 minutes)

In the field of samples observations by scanning electron microscopy (SEM), imaging material samples at elevated temperatures has got an increase of interest during the last decades. Performing in-situ observations at high temperature facilitates the understanding of materials behaviour when they are used under severe environments [1]. It is for instance the case of superalloys used for turbine blade in jet engine which must withstand high mechanical loads and extremely high temperatures. Making high temperature SEM imaging possible requires specific instruments to be developed in order to overcome several constrains (thermoionic emission, detectors thermal sensitivity…). Heating samples at elevated temperature (1000℃ and more) can be achieved by the means of miniaturized furnaces directly implemented in the SEM chamber. Most of them are built with a thin electrical resistance that uses Joule effect to warm up the sample. The accurate control and monitoring of the sample temperature needs a precise understanding of the micro-furnace thermal behaviour that is conditioned by the material components properties. In order to predict the micro-furnace capabilities, thermal modelling can be done by using finite element analysis (FEA). This tool is very helpful to design and to make an efficient micro-furnace but it requires a validation by testing a real furnace prototype. It is in this aim that a new micro-furnace prototype called FurnaSEM is manufactured and tested in controlled environments by the means of a specific bench test. The comparison of experimental data and numerical solutions is used to validate the prototype operability at high temperature.

Numerical model of FurnaSEM

Thermal modelling of the micro-furnace is ensured by a FEA industrial software: SolidWorks Flow Simulation which allows to take into account and to calculate all the heat transfer mechanisms operating around a solid component (conduction, convection and radiation) in a large variety of situations. Relevant numerical solution requires to consider the material properties such as thermal conductivity, specific heat capacity and radiative emissivity in a large temperature domain. The use of thermophysical properties database is crucial to adjust the parameters for the thermal modelling of solids. These properties are not available in litterature for the temperature range of interest. Consequently we have performed sensibility studies as a function of several parameters (emissivity and thermal contact resistance) to better estimate the micro-furnace thermal behaviour. Results obtained by numerical modelling are compared with experimental measurement achieved within a housemade bench test. This bench test is designed to monitor the furnace under vacuum while measuring the temperature at different points of interest using both thermocouples and thermal cameras. The experimental data are used to identify the best fitted parameter values. Finally the more accurate thermal model (i.e. set of parameters adjusted between the numerical model and experimental datasets) is defined to predict the 3D temperature fields. The full thermal map of the micro-furnace gives tips about its weaknesses. This will help us to further optimise the design and efficiency of the micro-furnace and to develop a new generation of micro-furnaces that works at higher temperature (1300-1450℃).

Manufacturing

The manufacturing of the miniaturized furnace was essentially 3D computer-aided. The prototype design is cylindrical and contains few elements that simplify its use. The sample is directly put on a sample holder that is a metallic cylinder piece. This cylinder is directly sustained by the heating system. The heating system contains a heating element that is embedded in a cylindrical hot body. Both pieces are assembled together mechanically. They are associated to the body of the furnace by a thin metallic wire which limits the heat conduction leaks. Thermal shield pieces enclose hot components and are cooled by conduction with a cold casing. The casing temperature is maintained below 50℃ by a cooled fluid circulation. The chose of the material compositions were motivated by all these constrains. The heating element is made of a nickel superalloy that withstand high temperature constrains and presents a good resistance to high temperature oxidation.

The shields and the sample carrier are made with stainless steel and the casing is made with copper. The micro-furnace temperature is monitored using two K-type thermocouples which are located near the heating element and the sample holder. The diameter of the furnace as well as its height are lower than 30mm. It can be easily placed in the SEM chamber. The compacity of the whole system allows to perform in-situ observations at high temperature using a relatively low working distance between the sample surface and the objective lens of the microscope (10 to 15 mm).

Tests and SEM applications

The bench test was specially designed to assess thermal capacities before operating inside the SEM. It contains a vacuum chamber instrumented with a thermal infrared camera which provides surface thermography measurements. The system can operate under a minimum pressure of 3.10-3 Pa and was mainly used for ex-situ thermal characterisation of the micro-furnace. The main advantage of this system is the temperature monitoring by independent measurement devices (i.e. thermocouple sensors, infrared camera and pyrometer) which help to enhance robustness of experiment data. Moreover, continuous thermal cycling of the furnace in repeatable conditions is a good way to monitor the system durability and to identify the degradation mechanisms.

When it is implemented in the SEM chamber, the miniaturized furnace enables new imaging possibilities, mainly due to the flatness of the sample holder.

• The furnace can be tilted in the -5° / +5° range while recording images in the SEM chamber. Then, the tilted image series are used to calculate 3D images of the sample surface and thus observe directly the surface rugosity variations as a function of temperature [2].

• A high temperature backscattered electron (BSE) detector (provided Crytur company) can be used in combination with FurnaSEM. It allows to record BSE images at high temperature, i.e. to characterize continuously the sample chemical modifications during a heat treatment [3].

• Working with a shorter working distance allows to record images at high temperature with a lower high voltage and with a high resolution.

This was used in the field of nuclear materials: in-situ observations of uranium oxide microparticles sintering were achieved in order to understand better the physical and chemical mechanisms that operates during the fabrication of the nuclear fuel [4].

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Session Classification: 07 Nuclear Fuel Cycle, Safeguards and Homeland Security

Track Classification: 07 Nuclear Fuel Cycle, Safeguards and Homeland Security