

Development of a miniature 1300°C furnace for Environmental or Low Vacuum Scanning Electron Microscopy

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High-temperature environmental scanning electron microscopy has been a flagship activity of L2ME for 12 years now. It has led to the observation of many physico-chemical phenomena taking place in the temperature range 25-1400 ° C such as the incorporation of RuO₂ in nuclear glasses, the determination of the conditions of formation of molybdenum lakes during the elaboration of certain glasses or the description of the first stage of sintering of UO₂.

Recent developments carried out in collaboration with the company NewTEC Scientific (based in Nîmes), concerning the development of a new family of miniature ovens open up new fields of investigation. A new all-metal furnace, called FurnaSEM1300, achieves a maximum temperature of 1300°C (Figure 1), while guaranteeing excellent quality of the atmosphere surrounding the sample (high vacuum, oxidizing or reducing conditions) and the possibility of working at an optimized working distance. This furnace provides access to new functionalities and opens up new fields of exploration, in particular for the study of the behaviour of metallic materials.

Images of excellent quality were recorded on a model ceramic (Figure 2) up to 1338°C under 100 Pa of air, without loss of image quality (change in signal/noise ratio, resolution). The nature of the materials chosen to build this furnace makes it possible to limit the emission of thermal electrons and allows a better stability of the images during the rise in temperature, and in isothermal level. At the same time, the quality of the vacuum accessible with this furnace even when heated to high temperatures, and its intrinsic properties make it possible to achieve very fast temperature rises or quenching up to 10 ° C/s or 600°C/min. The intrinsic performance of this experimental device gives access to microstructural transformations taking place in metal alloys during rapid quenching (formation of austenite or bainite in steels). These transformations could be observed for the first time in a SEM (Figure 3).

These new materials and associated methodological developments are accessible to the ICSM by contacting Renaud Podor or Joseph Lautru.

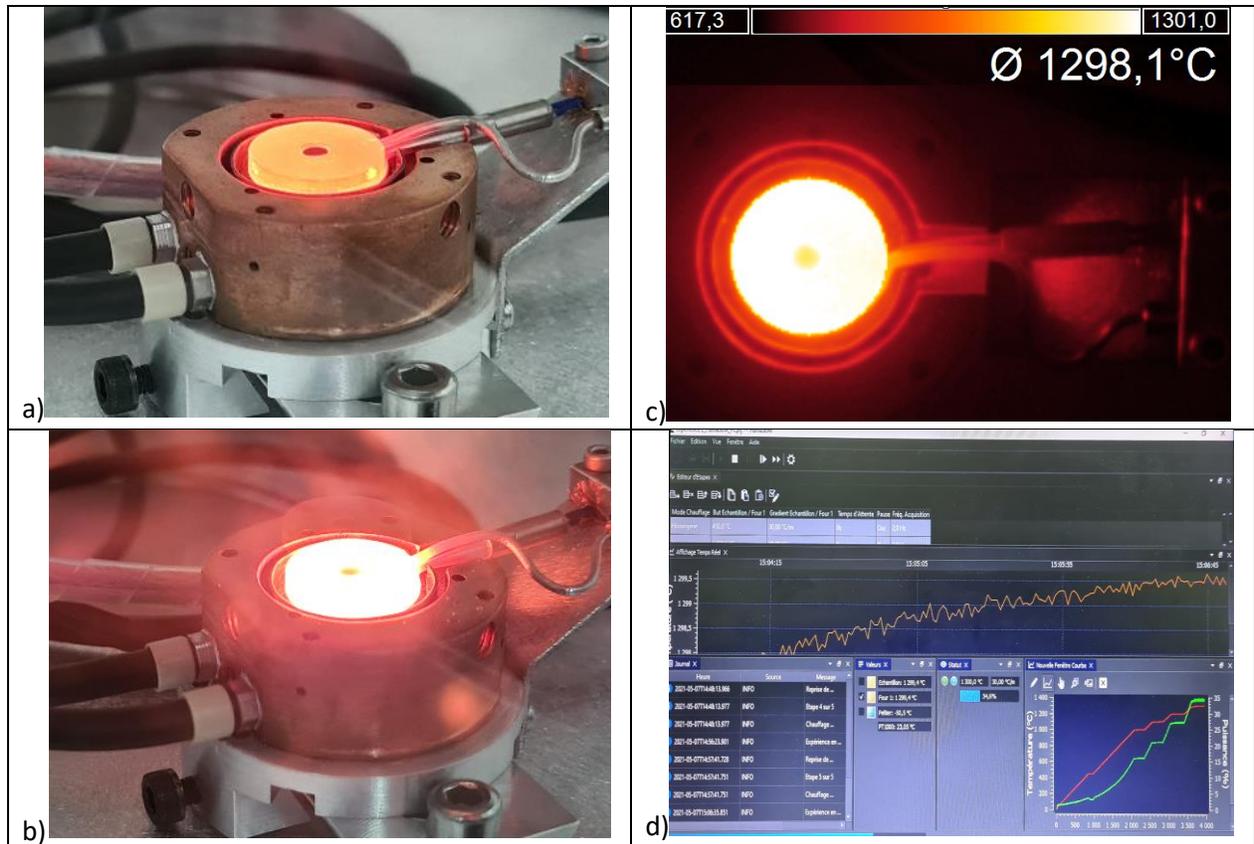


Figure 1. Photographs of the FurnaSEM 1300 furnace mounted on the test bench in operation at 1000°C (a) and 1300°C (b). Temperature map of the FurnaSEM 1300 furnace heated to 1300°C recorded with the infrared camera (c). Associated software conditions (d).

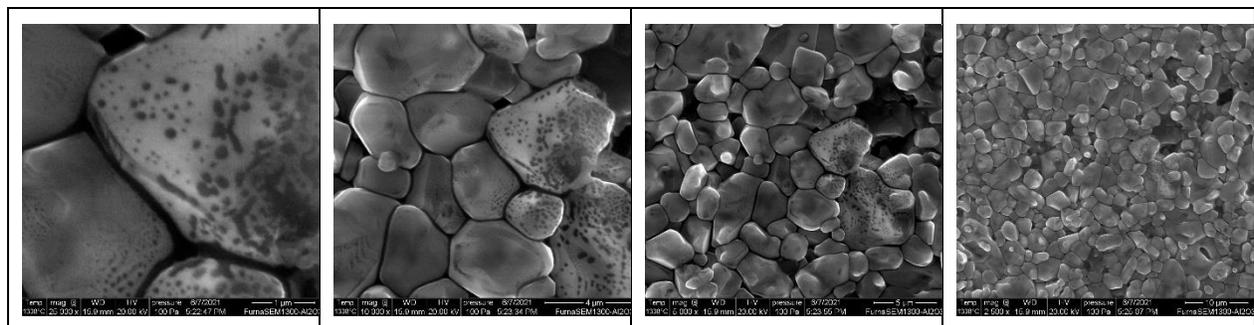


Figure 2. Images recorded at 1338°C on an alumina plate (Al_2O_3) at different magnifications.

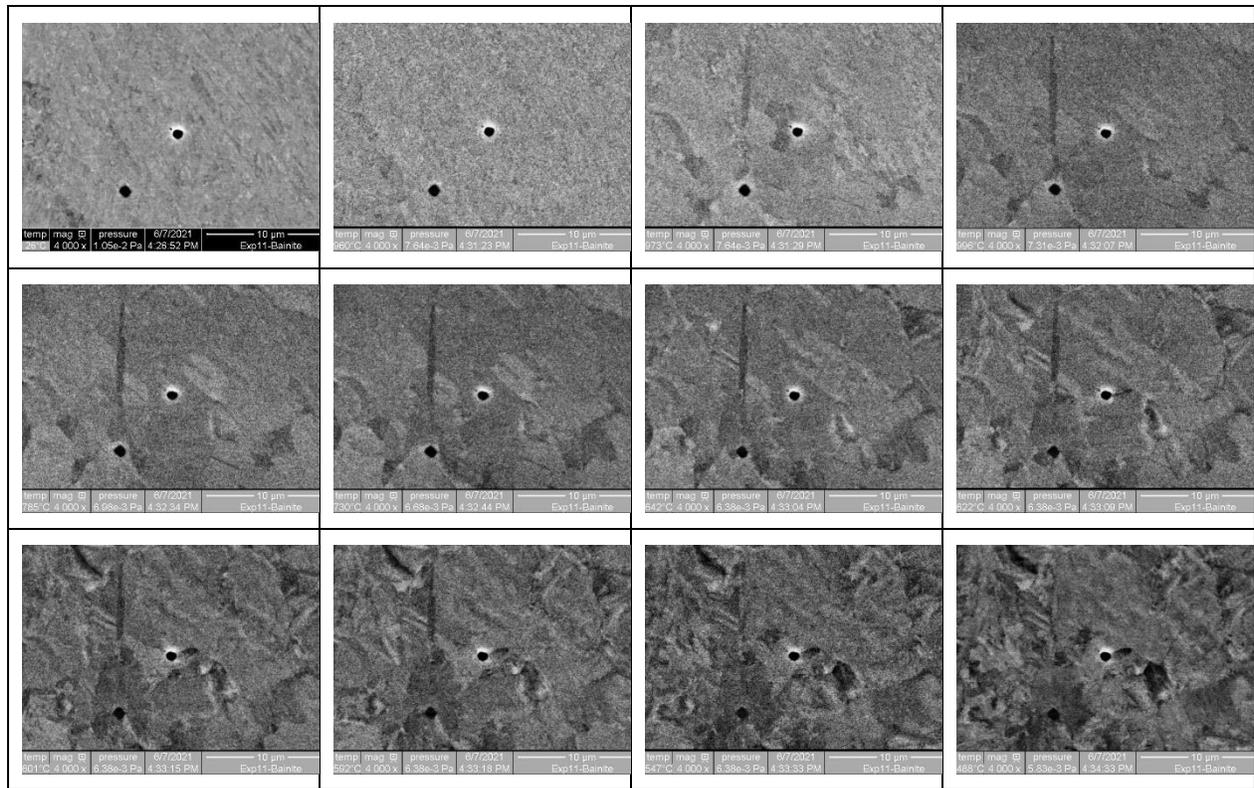


Figure 3. Series of SEM images recorded with the FurnaSEM1300 furnace under high vacuum during heat treatment applied to steel. The different transformations are directly observable on the recorded images.