SAMPLE ENVIRONMENT ELEVATED TEMPERATURE RECENT DEVELOPMENT AT ESRF

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Abstract

In the last few years a wide range of furnaces covering the range from room temperature up to 1350 °C has been designed and constructed to study in situ solid-state reactions, melting and crystallization processes with x-ray diffraction and imaging in transmission and reflection geometries. On one side of the evolution a miniaturization effort has led to a family of furnaces for tomography. On the other side specialized furnaces have been developed to reach high temperature stability, to heat under an oxygen atmosphere and a chamber for pulsed laser deposition (PLD). The latter includes novel aspects such as optical access for the laser and a thin walled aluminum vessel.

1. A High Stability Furnace

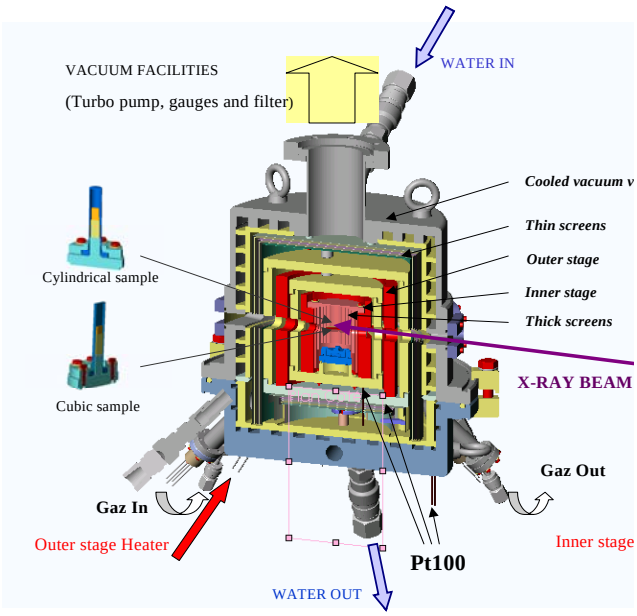

***Figure1**: Design section of the High Stability Furnace*

This furnace is well suited for phase transition studies [1]. A design section of the High Stability Furnace is presented below (figure1). The samples may be studied either in a confined atmosphere by means of a Beryllium container or in a vacuum environment. The level of the vacuum can be as low as 10^{-6} mBar.

The temperature stability of the sample is better than +/-2mK between 300K and 800K over several days. The furnace uses a Model-based Optimal Control algorithm [2] designed to achieve high stability together with fast response and no overshoots (figure2).

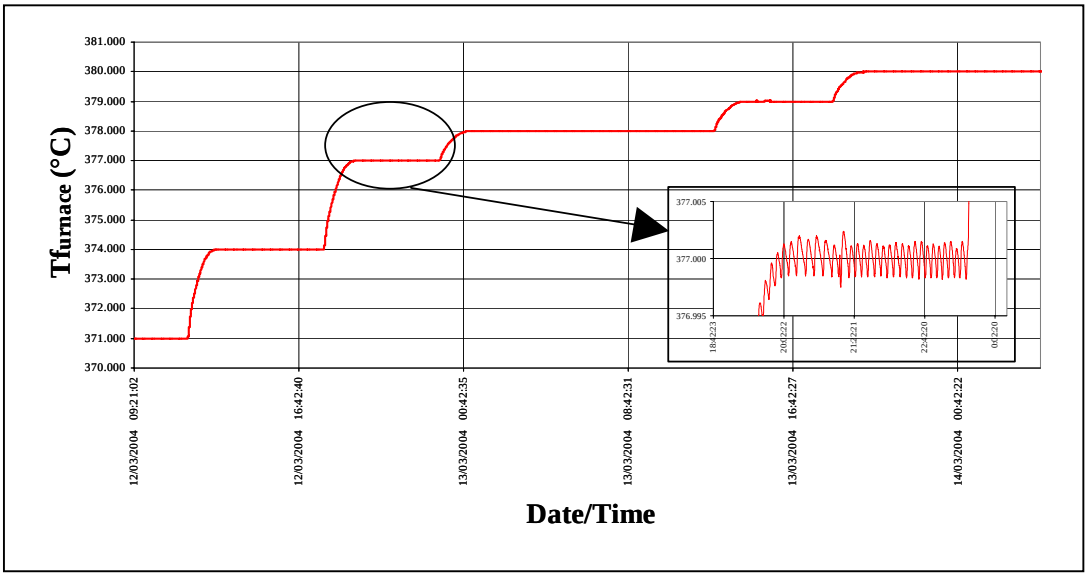

***Figure 2**: Raw data records showing the temperature profile of the furnace with fast response, no overshoot and high stability.*

2. A 1300K furnace for in situ X-ray diffraction [3]

A furnace covering the temperature range from 25 to 1000°C (figure 3) has been designed and constructed to study *in situ* solid-state reactions and melting and crystallization processes, with X-ray diffraction in transmission geometry using a two-dimensional-detector system. The oven can work in low vacuum and under a controlled atmosphere.

Keywords: ***in situ*** **X-ray diffraction; furnace; solid-state reactions; film-substrate.**

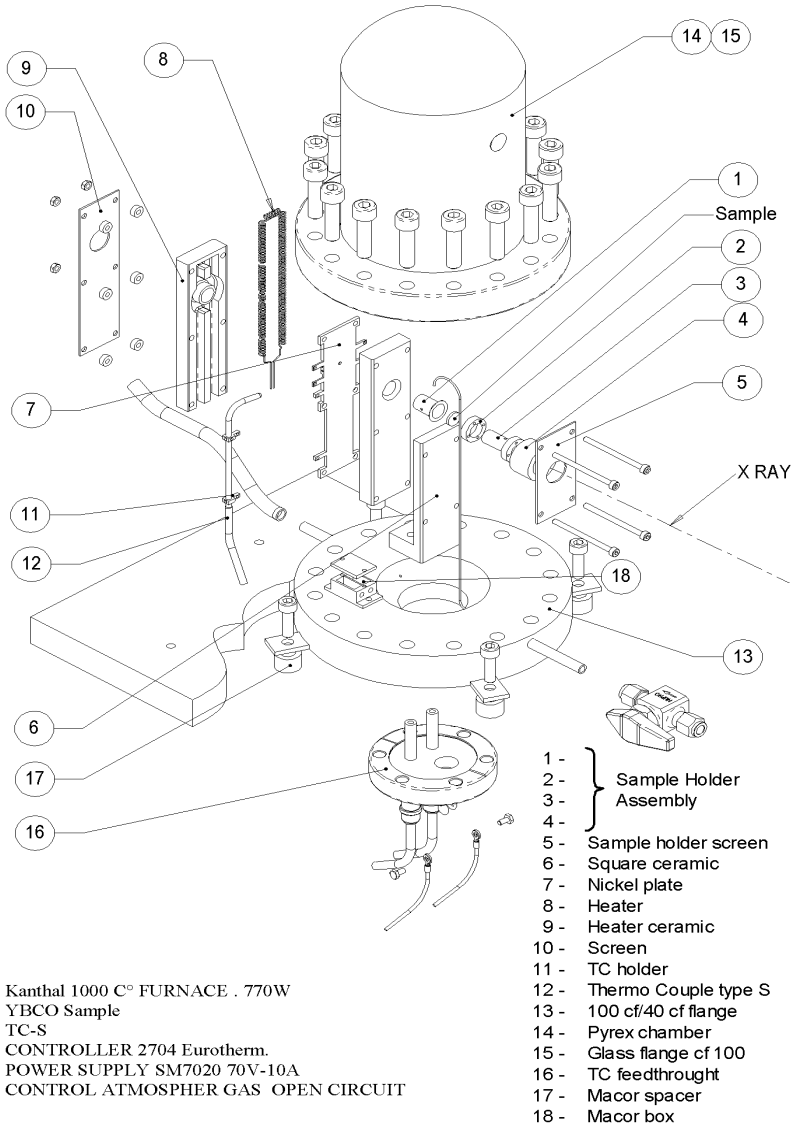

***Figure 3**: Schematic view of the Kanthal furnace*

3. A 1300 K furnace for *in situ* X-ray microtomography [4]

A furnace covering the temperature range from 298 to 1300K (figure 4) has been designed and constructed to study *in situ* the structural evolution of materials by using synchrotron X-ray microtomography. The furnace has cylindrical symmetry, compatible with the required 180° rotation, small dimensions and low absorption. It can also be used for other scattering methods, such as diffraction.

Keywords: tomography

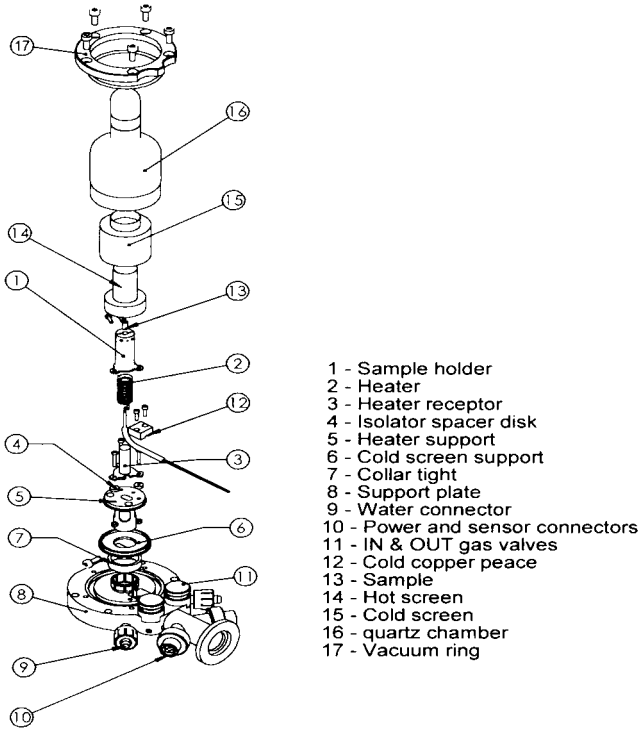

Figure 4: *Schematic view of the Microtomography furnace.*

4. Perspectives

The use of high energy synchrotron radiation is becoming more and more important to investigate phase transformations and re crystallization phenomena of metals and alloys in-situ at high temperatures. When oxidation has to be avoided, the experiments have to be done in vacuum or in a protective atmosphere (He/Ar). Currently a new heating resistance (Super Kanthal 1800°C) is under development which will give a possibility to design a new compact high temperature chamber. The goal is to observe phase transformations in Ti and Zr at 900C-1200°C.

Another project to reach even higher temperatures (20-2000°C) investigates the possibility to use induction and/or laser heating. The goal is to study the oxidation state of Cr at temperature up to 1700°C under oxygen atmosphere using X-ray absorption spectroscopy.

A novel aspect will also be to combine high temperature with other thermo dynamical parameters such as pressure and magnetic field.

5. References

[1] ID10 Beamline at ESRF (A. Madsen, A. Fluerasu, A. Moussaid, F. Zontone, G.Grübel, technical contact for this furnace: M. Mattenet).
[2] Pierre F. Poncet – Epteau – Grenoble (paper under preparation).
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[4] *J. Appl. Cryst.* (2003). **36**, 366-367 D. Bellet, B. Gorges, A. Dallery, P. Bernard, E. Pereiro and J. Baruchel.

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