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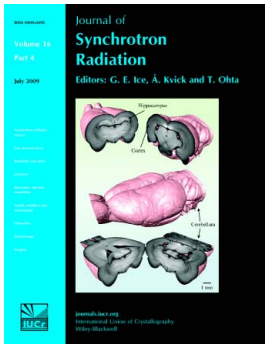

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A 1800 K furnace designed for *in situ* synchrotron microtomography

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A radiation furnace that covers the temperature range from room temperature up to 1800 K has been designed and constructed for *in situ* synchrotron microtomography. The furnace operates under a vacuum or under any inert gas atmosphere. The two 1000 W halogen heating lamps are water- and air-cooled. The samples are located at the focus of these lamp reflectors on a rotary feedthrough that is connected to a driving rotation stage below the furnace. The X-ray beam penetrates the furnace through two X-ray-transparent vacuum-sealed windows. Further windows can be used for temperature control, sample changing and gas inflow and outflow.

Keywords: microtomography; radiation furnace.

1. Introduction

Many scientific problems in materials, biological or medical sciences can be solved by microtomography; owing to the high flux of third-generation synchrotron sources it is possible to investigate movements or transformation processes in three dimensions on very short time scales. For this reason, several mechanical stress and strain rigs, heating and cooling devices, and climate chambers have been developed for use in tomography experiments in the last few years (*e.g.* Bellet *et al.*, 2003; Wang *et al.*, 2005). Interesting results have been obtained for very different material classes, such as metallic, ceramic and concrete foams (*e.g.* Haibel *et al.*, 2006; Maire *et al.*, 2003; Khor *et al.*, 2004; Weidemann *et al.*, 2007), sintered materials (*e.g.* Lame *et al.*, 2003; Nöthe *et al.*, 2007), creep damage or interdiffusion of alloys (*e.g.* Beckmann *et al.*, 2007; Pyzalla *et al.*, 2005; Haibel & Scheuerlein, 2007; Scheuerlein *et al.*, 2007), bones, wood and even for insects (*e.g.* Ritman *et al.*, 1997; Bleuet *et al.*, 2004).

Improvements in both the spatial and the temporal resolution down to the submicrometre and millisecond range make high demands on the mechanical precision of such *in situ* devices. The present article is devoted to the description of the special requirements and the technical specification of a radiation furnace that was designed to allow *in situ* microtomography experiments at temperatures up to 1800 K with very fast heating and cooling rates and with high mechanical precision. The furnace was developed at the Helmholtz Centre Berlin for Materials and Energy in cooperation with the Technical University Dresden, and operates at the beamline of the Federal Institute of Materials Research and Testing (BAM) at the BESSY II synchrotron in Berlin.

2. Requirements for an *in situ* furnace for tomography

An *in situ* furnace for synchrotron microtomography must have some specific features. In general, the furnace should work under a vacuum as well as under several controlled gas atmospheres in the sample chamber. The upper limit of the temperature range has to be high enough to cover the widespread demands of different scientific fields. The ability to achieve fast heating and cooling rates is another crucial design parameter. Moreover, the ability to maintain a desired temperature with minimal fluctuations is important. Furthermore, the area of homogeneous temperature must have a size of about 1 cm^3 to be sufficient for microtomography samples.

It is also necessary that the furnace has X-ray-transparent windows that should be large enough to permit flatfield measurements for the background correction of the radiographic images during the tomographic experiment. Flatfield images have to be taken at periodic intervals in the absence of the sample during the measurement. For this, the whole furnace can be moved several millimetres sidewards such that the specimen is moved out of the beam.

During rotation of the sample the error in the position of the rotation axis has to be significantly smaller than the spatial resolution of the set-up chosen. Furthermore, the temperature of the sample has to be controlled without any contact between the sample and the heater in order to allow for sample rotation. An interesting aspect is the opportunity to combine different sample manipulations such as heating under stress or strain. Therefore additional structural features have to be taken into account (*e.g.* additional mounts).

To protect the surrounding set-up, the temperature difference between the heated area inside the furnace and the

temperature outside the furnace has to be very high. This protects the scintillator screen as well as the rotation stage and guarantees measurements with minimal errors. Whereas the temperature of the focus is up to 1800 K, the temperatures outside the furnace should be below 350 K.

3. Technical characteristics

The furnace operates with a radiation technique using two 1000 W heating bulbs and allows for very fast heating and cooling rates. These heating and cooling rates depend only on the thermal absorption of the sample material, because the lamps can be tuned to full power directly by switching on. Because the energy of the bulbs is focused in a small heating spot the maximum sample temperature is up to 1800 K. Owing to the small heating spot the maximal dimension of the specimen is limited to 1 cm. The effective diameter of the samples is also limited by the X-ray beam width. (Special tomographic techniques such as dual circle tomography or zoom tomography are not applicable owing to the limited time scale for the measurements while heating.) However, for high spatial resolutions of about 1 µm, the sample size decreases below 2 mm diameter for a 2048 × 2048 pixel CCD detector, and a size of 1 cm^3 for the heating spot is therefore sufficient for most applications. The precision of the temperature control and stability of the furnace is approximately 1 K. A schematic view of the inner layout of the furnace is presented in Fig. 1.

The furnace is mounted on a translation stage that is located below the rotation stage and allows the furnace to be moved a few millimetres perpendicular to the beam, as required for flatfield measurements. The sample inside the furnace is rotated during the measurements by a vacuum rotation feedthrough that is connected to the rotation stage of the tomography set-up below the furnace.

Because the furnace itself is not rotating, it is not necessary to use rotary mounts for the gas and cooling water tubes or the electric cables. The vacuum rotation feedthrough is installed in a flange at the bottom of the furnace and allows for a controlled atmosphere or even a vacuum to be maintained

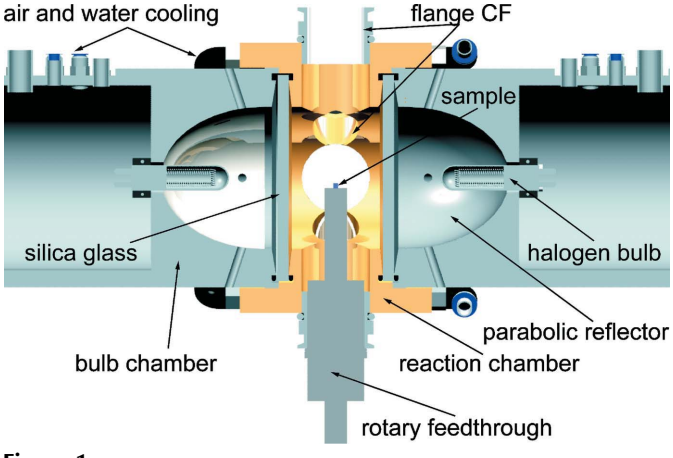


Figure 1
Cross section of the radiation furnace.

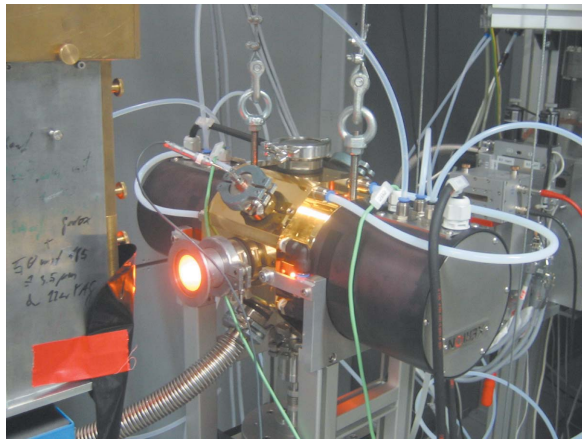


Figure 2
Radiation furnace in operation at the tomography set-up at the BAMline (BESSY II).

inside the sample chamber. An axis error of this feedthrough below 0.3 µm has been measured, *i.e.* sufficient for spatial resolutions of about 1 µm.

The upper part of the feedthrough, where the samples are mounted, is made of a refractory alumina rod. The top of this rod is located near the focal spot and provides an efficient temperature decoupling of the rotation stage from the furnace. Thus, only the sample itself and the refractory alumina rod that has a minimal thermal expansion coefficient of 8×10^{-6} K^{-1} are heated up during the measurements. Fig. 2 shows the furnace in operation.

The parabolic lamp chambers are made of polished aluminium and separated from the sample chamber by two silica glass windows. These windows transmit the radiation, but separate the lamp atmosphere from the sample atmosphere, making it possible to cool the water-cooled lamps using compressed air in the lamp chambers. In addition, owing to the separation of the sample chamber from the lamp chambers, the volume of the sample chamber is minimized. This reduces the required volume of the process gas or the evacuation time of the sample chamber.

The sample chamber is also water-cooled by a meander-shaped channel inside the housing. This allows for a large thermal gradient from the focus point of the lamps with up to 1800 K to the housing with an outer temperature of about 340 K even by working under gas atmosphere.

The sample chamber is equipped with eight flanges (see Fig. 3) for various purposes: one flange for mounting the rotation feedthrough from below, one flange for positioning and changing specimens from above, two flanges for passage of the X-rays through the X-ray-transparent vacuum-sealed windows in the front and back, two flanges for gas inlet and outlet, and two flanges for mounting thermocouples. The flanges can also be used to integrate other *in situ* devices, *e.g.* stress or strain rigs. The X-ray-transparent windows are exchangeable and made of thin metal (*e.g.* aluminium) or Kapton foils, depending on the required temperature inside the furnace, on the X-ray energy and on the gas atmosphere or vacuum. For taking flatfield images, the windows are designed

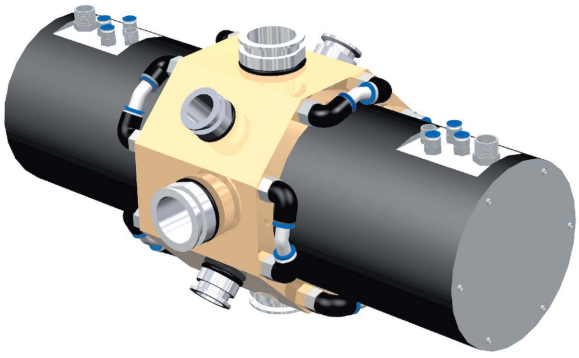
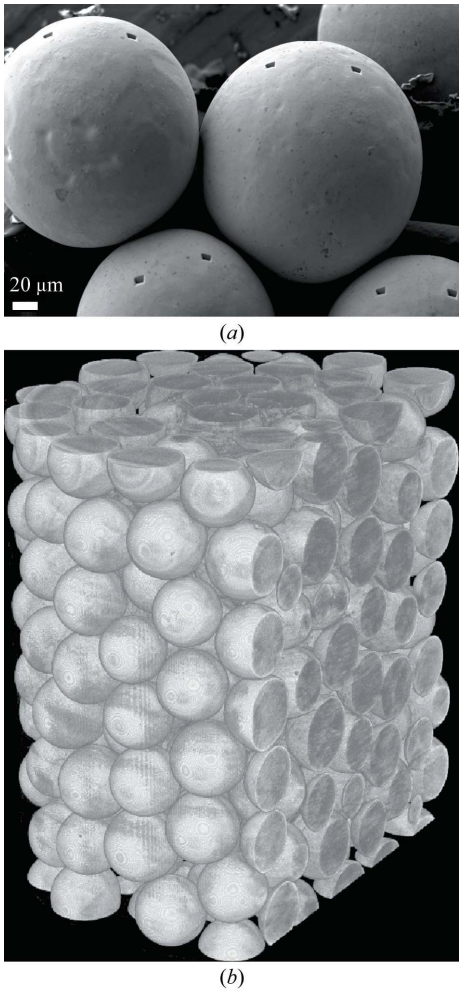


Figure 3
The furnace. The connections for water and compressed air cooling, and electric cables are shown on the left and right (on top of the lamp chambers). In the middle the sample chamber flanges and joints for the meander-shaped water-cooling can be seen.



(*a*)

(*b*)

Figure 4
Top: scanning electron microscope image of spherical monocrystalline copper particles marked with small holes by focused ion beam. Bottom: tomogram of these particles in an early stage of sintering (Grupp, 2008).

to be large enough to allow the furnace to be moved *via* the translation stage several millimetres perpendicular to the beam.

To know the exact temperature of the samples during the experiments, a thermocouple near the sample position has to be calibrated. During calibration, one thermocouple is fixed in the hot spot 2 mm away from the specimen and a second one is positioned inside a reference material. During tomographic measurements, the second thermocouple is removed and the temperature is tracked by the calibrated thermocouple near the sample position. Depending on the furnace temperature, K (below 1400 K) or B (above 1400 K) thermocouples are used. Certainly, this calibration depends on the sample material and has to repeated for each new material class.

The length and the diameter of the furnace are 499 mm and 240 mm (from flange to flange), respectively. Including the flanges, the total weight is about 40 kg. Owing to the weight of the furnace it is necessary to integrate manipulation stages in the tomographic set-up (beside the rotation stage) that can carry this load while preserving the mechanical accuracy. Alternatively, the excess load on the manipulation stages must be compensated by a cord grip.

This furnace has been used successfully for tomographic investigations of sintering processes of copper powder. Fig. 4 shows a typical example (Grupp, 2008). More than 300 spherical copper particles were marked by focused ion beam with a small borehole. Combined with a custom image analysis it was possible to detect particle rotations during sintering. Furthermore, changes in the number of contact partners and the centre approach of the particles could be measured *in situ* too. It is planned to provide the furnace for various *in situ* user experiments as well. Application of this furnace at the BAMline at BESSY in synchrotron tomography experiments will allow for study of phenomena in materials science that previously were not accessible.

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References

Beckmann, F., Grupp, R., Haibel, A., Huppmann, M., Nöthe, M., Pyzalla, A., Reimers, W., Schreyer, A. & Zettler, R. (2007). *Adv. Eng. Mater.* **9**, 939–950.
Bellet, D., Gorges, B., Dallery, A., Bernard, P., Pereiro, E. & Baruchel, J. (2003). *J. Appl. Cryst.* **36**, 366–367.
Bleuet, P., Roux, J.-P., Dabin, Y. & Boivin, G. (2004). *Proc. SPIE*, **5535**, 129–136.
Grupp, R. (2008). PhD thesis, Technical University Dresden and the Helmholtz Centre Berlin for Materials and Energy, Germany.
Haibel, A., Rack, A. & Banhart, J. (2006). *Appl. Phys. Lett.* **89**, 154102.
Haibel, A. & Scheuerlein, C. (2007). *IEEE Trans. Appl. Superconduct.* **17**, 34–39.
Khor, K. H., Buffiere, J.-Y., Ludwig, W., Toda, H., Ubhi, H. S., Gregson, P. J. & Sinclair, I. (2004). *J. Phys. Condens. Matter*, **16**, 3511–3515.
Lame, O., Bellet, D., Bouvard, D. & Di Michel, M. (2003). *Defect Diffus. Forum*, **216–217**, 285–292.

Maire, E., Elmoutaouakkil, A., Fazenkas, A. & Salvo, L. (2003). *MRS Bull.* **28**, 284–289.
Nöthe, M., Schulze, M., Grupp, R., Kieback, B., Haibel, A. & Banhart, J. (2007). *Mater. Sci. Forum*, **534–536**, 493–496.
Pyzalla, A., Camin, B., Buslaps, T., Di Michiel, M., Kaminski, H., Kottar, A., Pernack, A. & Reimers, W. (2005). *Science*, **308**, 92–95.
Ritman, E. L., Jorgensen, S. M., Beighley, P. E., Thomas, P. J., Dunsmuir, J. H., Romero, J. C., Turner, R. T. & Bolander, M. E. (1997). *Proc. SPIE*, **3149**, 13–24.
Scheuerlein, C., Di Michiel, M. & Haibel, A. (2007). *Appl. Phys. Lett.* **90**, 132510.
Wang, Y., Uchida, T., Westferro, F., Rivers, M. L., Nishiyama, N., Gebhardt, J., Lesher, C. E. & Sutton, S. R. (2005). *Rev. Sci. Instrum.* **76**, 073709.
Weidemann, G., Goebbels, J., Haibel, A., Hillemeier, B. & Stadie, R. (2007). *Proceedings DGZfP-Jahrestagung 2007*, DGZfP-Berichtsband BB 104-CD, ISBN 978-3-931381-98-1.