International workshop Sample Environment at Neutron Scattering Facilities 2008
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ESRF GRENOBLE

MICRO-FURNACE FOR TRANSMISSION X-RAY DIFFRACTION IMAGING

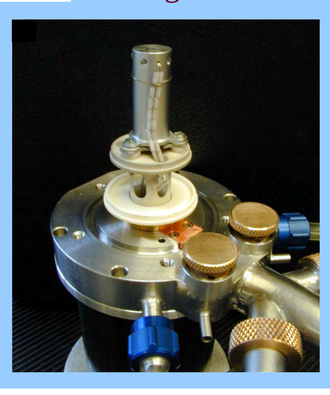
A heating radiative cell has been design to study the thermal evolution of the phase contrast of domains on Bragg diffracted images of poled ferroelectric crystals on BM05 and ID 19

B GORGES, Sample Environment group, ESRF, GRENOBLE

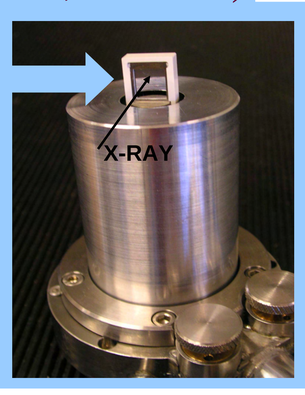
COLLABORATION PROJECT: XR Imaging: J Baruchel, R Klünder MX: P Pernot. Warwick University: users. Optics group: J P Vassali
SES group: B Gorges, H Vitoux

A new transmission furnace has been developed to work under gas flow up to 800 ºC. Most X-ray furnaces offering wide angle access work in reflection mode; X-ray topography measurements have to be done in transmission mode but still necessitate a wide angle. The reflection setup also gives a larger distance between the sample and the detector. This transmission furnace had to comply with a number of additional constraints: thermalisation of a low thermal conducting crystal sample, stress less sample mounting and small temperature gradients over a 7x7mm vertical sample surface. The construction also had to take the possibility of chemical reactions at high temperature between the sample, the construction materials and the gas into account.
The validation of the new setup has been done on ESRF beamline BM05 through the characterization of the ferro-electric phase transition of BaTiO3.

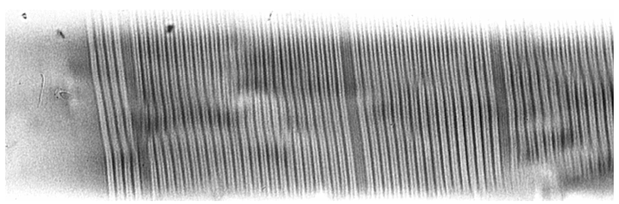
Heating cell working in air (no chamber, no window)



800℃ compatible reactive gas ΔT 10℃ on 7x7 silicon black body radiator plates



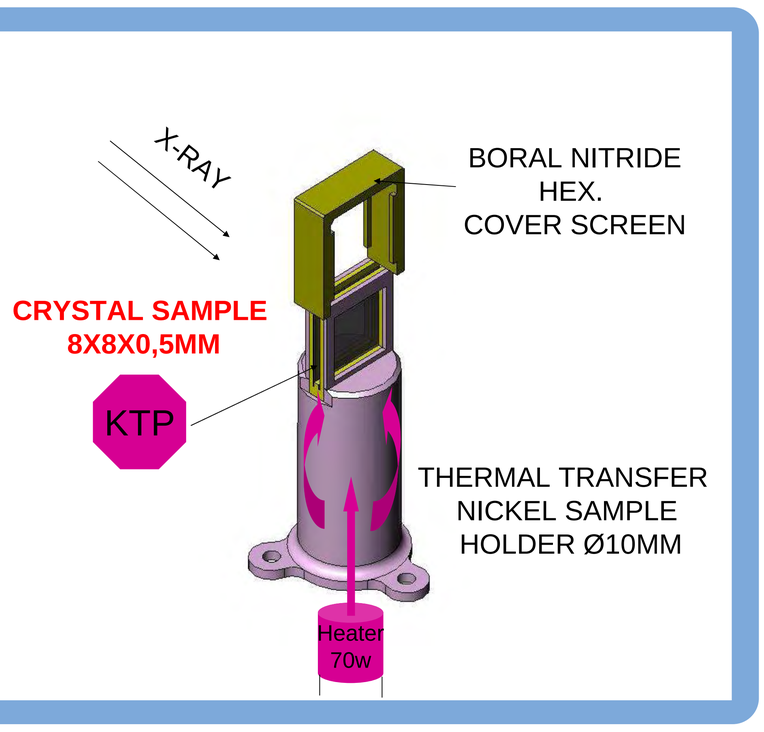
PERIODICALLY POLED KTP



ESRF: coherent beams

Phase contrast
Bragg diffraction imaging

SILICON RADIATOR 0,15MM, SINGLE CRYSTAL POLISHED (PHASE CONTRAST IMAGING)

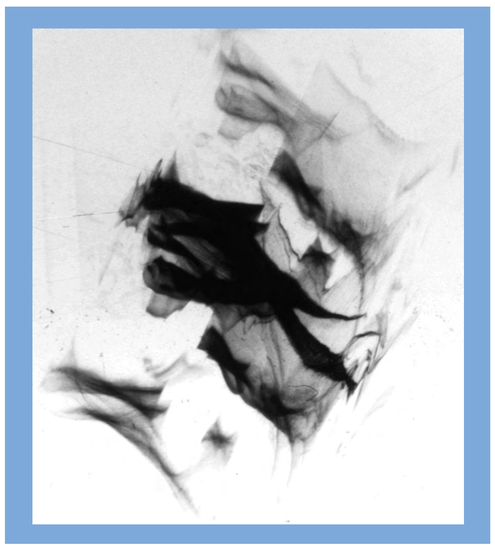


X- RAY TOPOGRAPHY: KTiOPO4 (KTP)

The effect of increasing temperature on the phase-contrast of ferroelectric domain walls in a domain-inverted KTP (Potassium Titanyl Phosphate) crystal has been investigated.

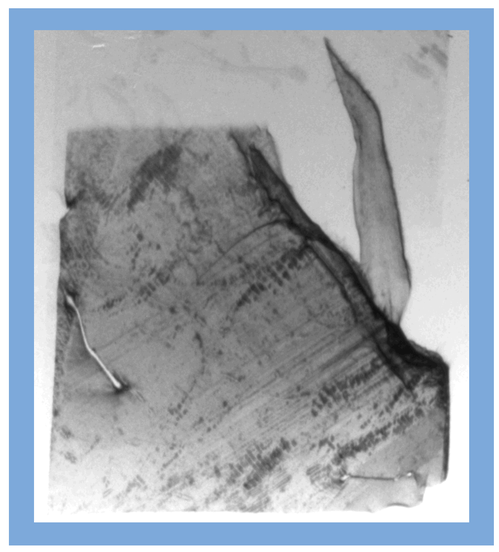
The goal was to learn the behavior of the domains when passing the Curie-temperature and to raise the quality of KTP as an optical crystal for non-linear effects.

KTP's are used as frequency doubling diodes for diverse IR-LASERS such as Nd:YAGor in green Laser pointers.).



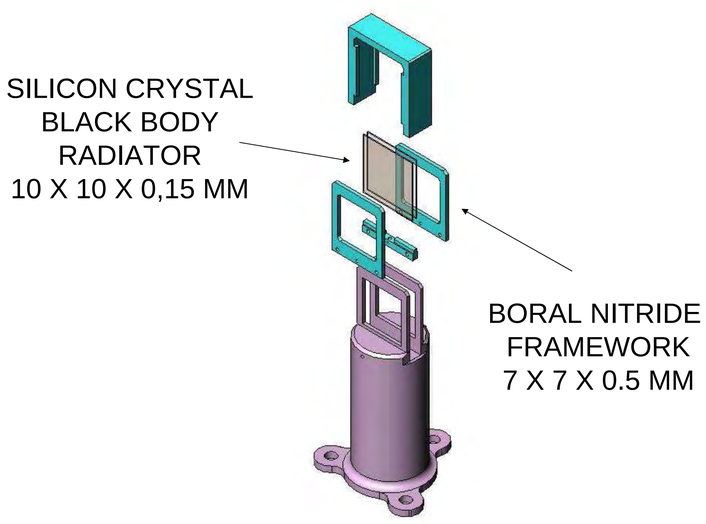
20°C DISTORDED CRYSTAL
FERROELECTRIC DOMAINS

BARIUM TITANYL



125°C NO DISTORDED CRYSTAL
PARAELECTRIC NO TENSION

Thermal gradient 3°C at 125°C over 6mm distance



ADVANTAGE OF THIS TRANSMISSION SET UP

THE SILICON BLACK BODY RADIATOR WORKED WELL, **NO STRESS ON THE SAMPLE**

GOOD STABILITY AND HOMOGENEITY OVER 7MM ΔT< 10°C, NO REACTIVITY WITH MATERIALS (NB between Ni and Si)

LARGE TRANSMISSION SURFACE 7 x 7mm, WORKING IN AIR or GAS
THE FURNACE IS VERY SMALL AND CONVIVIAL

Acknowledgements SES group, BM05 staff, ID19 staff