SR powder diffraction patterns of $CaCu_3Ti_4O_{12}$ (CCTO) has been measured using high quality polycrystalline sample. The observed diffraction patterns showed high crystallization of the prepared sample as well as high purity of CCTO with impurities level below 1%. The experiment covered temperature range between RT and 950°C.

High temperature

There were two main questions the proposal was to answer. The first one was to study changes of the lattice constant a (CCTO crystallise in cubic I-3 symmetry) for temperatures above RT. It was stated in the literature [1] that there is a change in the lattice constant behaviour at 440 °C with possible phase transition. It was also reported this change of the lattice constant is associated with the change of dielectric properties.

SR diffraction patterns collected below and above the suggested phase transition region near 440 °C did not show any change in the crystal symmetry. Data analysis performed by using the Rietveld method showed no change of thermal expansion coefficient, giving monotonic change of the lattice constant without any significant change in the slope of the lattice constant function. Fig. 1 shows the temperature dependence of the lattice constant a in CCTO above RT.

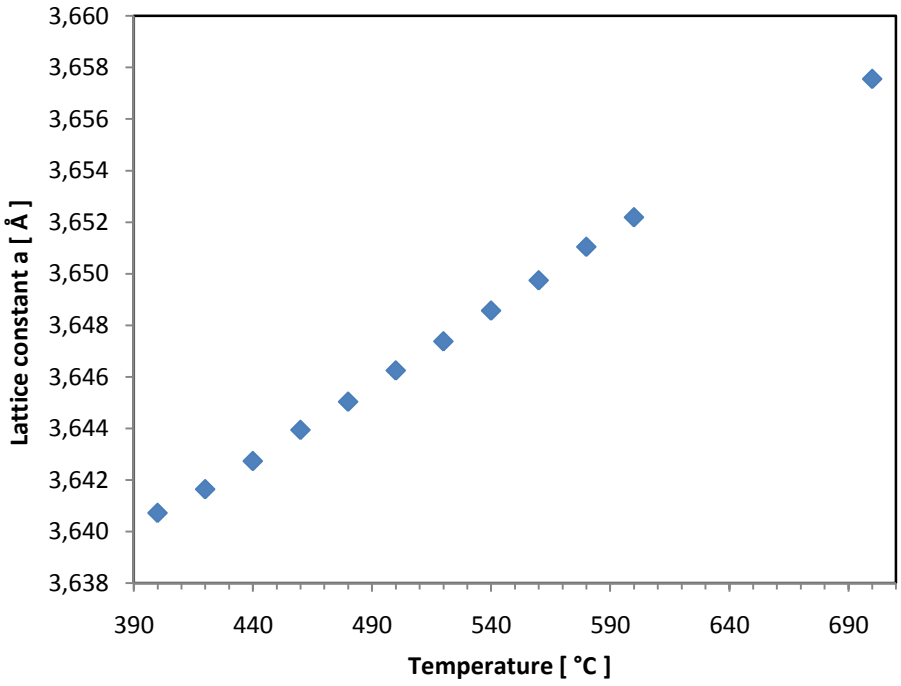

Fig. 1 Temperature dependence of the lattice constant *a* for $CaCu_3Ti_4O_{12}$.

Phase separation

To solve the problem of the phase separation and reduction of CCTO at temperatures above RT SR diffraction patterns have been measured for samples in different conditions: opened to air capillary, sealed capillary, capillary sealed under vacuum and capillary sealed under nitrogen atmosphere. Several temperature scans has been performed to receive better description of the reduction process.

Pure metallic Cu Bragg reflections has been observed only for CCTO measured in sealed capillary. The reduction of CCTO to pure metallic Cu phase was observed above 300 °C. The amount of the pure metallic Cu phase increases while increasing the temperature above 300 °C. The relative mass amount of pure metallic Cu phase was refined as 0.634(12) %.

For the temperatures above 700 °C pure metallic Cu phase oxidise completely.

This unusual phase separation identified for CCTO strongly depend on the environment of the sample in as well as the procedure of the temperature treatment.

[1] A. Onodera, M. Takesada, K. Kawatani, S. Hiramatsu *Japanese Journal of Applied Physics* **47** 9 (2008) 7753