	Experiment title: Solid state transformations of Au-Cu-Pt alloys studied by high-energy X-rays synchrotron radiation	**Experiment number**: HS3560
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Report:

The AuCu alloy is a classical example of order-disorder transformations in the solid state. Upon cooling from elevated temperatures, stochiometric AuCu transforms successively from the disordered fcc structure (α) to the ordered pseudo-orthorhombic AuCuII structure (long-range ordered periodic antiphased structure built of $L1_0$ cells) to the equilibrium ordered AuCuI structure ($L1_0$). The addition of a third element to the Au-Cu alloy induces changes in the domain of existence of the different phases, modifies the transition temperatures, and affects ordering kinetics.

The aim of this experiment was to identify *in situ*, during heating or cooling, the different phases, their volume fraction and measure the degree of order, for several Au-Cu-Pt alloys given in Table 1 and the subject of current research at EPFL. The Laue ring patterns were recorded in transmission at an energy of 86 KeV at regular time intervals during heating, dwell or cooling (acquisition time no longer than 60 s). The detector placed at a distance of 809.5mm of the sample was a Pixium 2D with a resolution of 2640x1920 pixels (pixel size: 154x154μm^2). The position and intensity of the different peaks were analyzed using the Fit2D software. Figure 1 shows a typical Debye-Scherrer ring of Alloy 3 at 250°C after 10^6s.

at. %	Au	Cu	Pt
Alloy 1	49.2	50.8	0.0
Alloy 2	50.9	47.4	1.7
Alloy 3	53.1	45.2	1.7
Alloy 4	54.8	39.7	5.5
Alloy 5	57.2	37.1	5.7
Alloy 6	61.9	25.6	12.5
Alloy 7	64.8	22.3	12.9

Table 1: Alloys studied during the experiment.

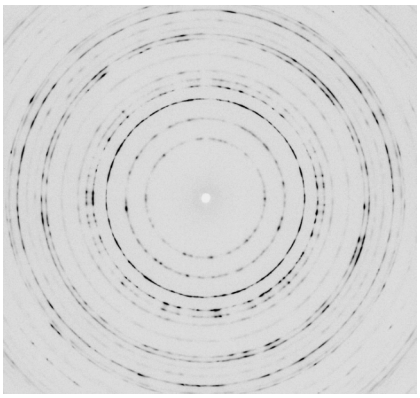

Fig. 1. Debye-Scherrer ring of Alloy 3 at 250°C after 10^6s.

Round samples 3mm in diameter and 100μm in thickness were used. Prior to the X-ray experiments, all samples were quenched in water from the disordered state. Then, *in-situ* heating was performed during data acquisition.

The evolution of the ratio c/a (a characteristic of the $L1_0$, ordered tetragonal phase) defined to measure the tetragonality level of Alloy 3 during heating/cooling cycles at 2K/min, is plotted in Fig. 2. The evolution of this ratio is smoother during heating than during cooling. The abrupt change during cooling is typical of a first-order transformation, involving nucleation and growth of ordered domains from the disordered state. These observations, as well as the measured order-disorder temperature transition, agree with previous Differential Scanning Calorimetry results.
Fig. 3 shows the evolution of the c/a ratio of Alloy 3 during a dwell at 250°C and at 400°C. The longer treatments were realized in a separate oven ending with a water quench. These samples were then reheated *in-situ* to the corresponding temperature and dwelled for 5000s before the X-ray patterns were recorded.
The decrease of the c/a ratio indicates a higher tetragonality level. The 250°C dwell seems to lead to a constant value after 10^6s, while the 400°C dwell shows dispersion in the data, which can be explained with prior transmission electron microscopy results that show a partial disordering after heat treatment at 400°C for 10^6s, leading to a two-phase $\alpha/L1_0$ structure.

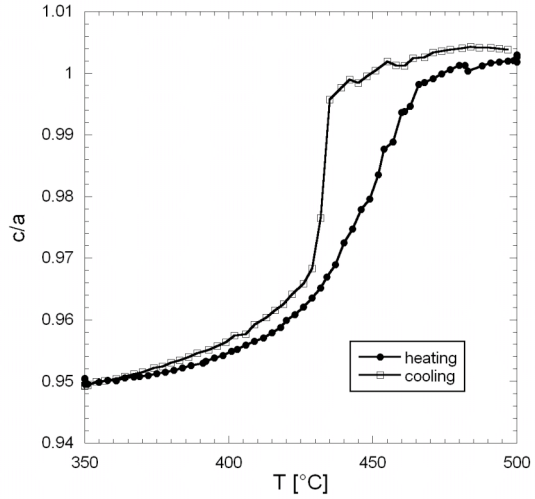

Fig. 2. Evolution of the c/a ratio during heating followed by cooling of Alloy 3.

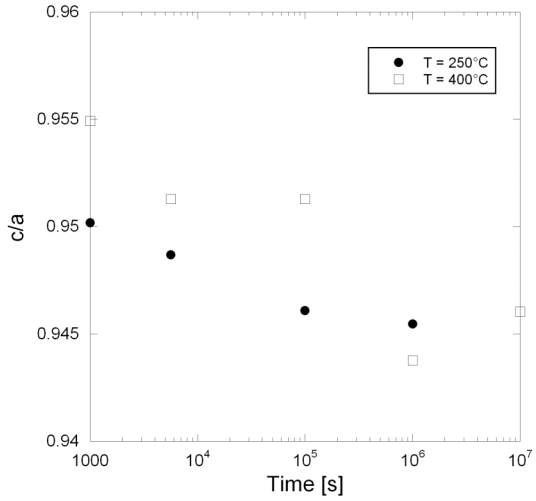

Fig. 3. Evolution of the c/a ratio in Alloy 3 during dwell at 250°C and 400°C.

Similar experiments have been carried out on the other alloys of Table 1. The transformation sequence AuCuI -> AuCuII -> α is observed in the Alloy 1. The comparison between the transition temperatures measured at the ESRF and the data from the literature (same heating-cooling rate) indicate that the temperatures have to be adjusted with an increase by 20K.
The results collected at the ESRF were both much richer and more precise for all alloys than those acquired at EPFL by Differential Scanning Calorimetry for the determination of the temperature of transition. For example, DSC measurement with a heating rate of 10K/min does not detect any phase transition for Alloy 6 and Alloy 7, while during in-situ heating under synchrotron radiation, the formation of $L1_2$ was evidenced.