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B-XRD1117 - Observation of a phase transition at high temperature under various atmospheres

Introduction

In order to develop new materials that have desired properties, it is essential to evaluate the materials under various atmospheric environments. The infrared heating high-temperature attachment Reactor X has a corrosion-resistant sample chamber separated from the heater section, so it can be used to perform high-temperature XRD measurements under various atmospheres, such as hydrogen, ammonia, high humidity and so forth. Using Reactor X with a 2D detector capable of high-speed XRD measurement, it is possible to investigate in detail rapid phase transitions under heating in various atmospheres.

Measurement and analysis

The redox reaction of a Cu powder sample was observed with Reactor X. The X-ray diffraction measurement (duration time: 1 min) was repeated while increasing the temperature at 50°C/min from RT to 800°C, at which point H₂ (4%) gas was introduced while keeping the temperature at 800°C. Fig. 1 shows the obtained profiles. During the temperature increase from RT to 800°C (from the lower to the middle part of Fig. 1 (left)), the diffraction peaks of Cu shifted to lower 2θ angles due to thermal expansion. Then the diffraction pattern changed to that of Cu₂O and CuO. After introducing H₂ gas (from the middle to the upper part of Figure 1 (left)), the diffraction pattern changed to Cu₂O and finally Cu. Figure 2 compares the transition of the mass fractions of Cu, Cu₂O and CuO. The chart indicates that the redox reaction of Cu occurred through states where Cu₂O coexisted.

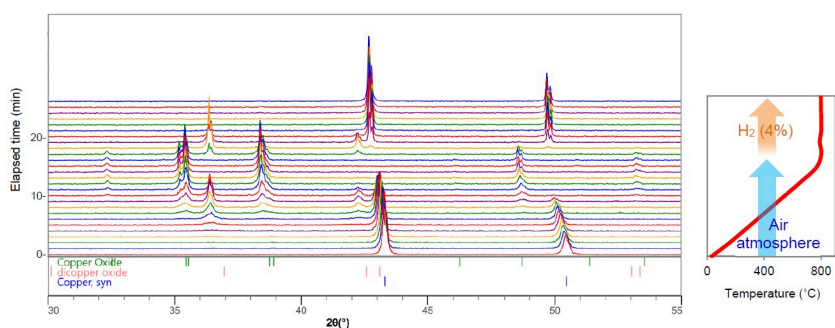


Figure 1: X-ray diffraction profiles (left) and the heating and atmosphere condition (right).

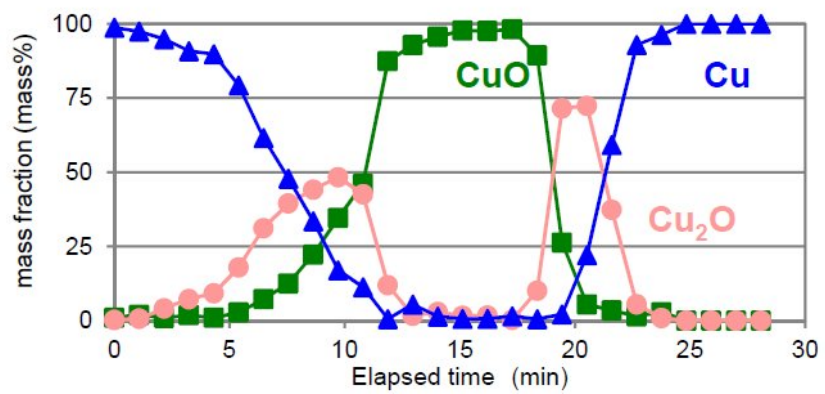


Figure 2: Result of quantitative analysis by Rietveld method.

Related products



HyPix-3000

Compact photon counting x-ray detector



SmartLab

Advanced state-of-the-art high-resolution XRD system powered by Guidance expert system software



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Highly versatile multipurpose X-ray diffractometer with built-in intelligent guidance



Reactor X

Allows measurements to be performed under high temperature.