

decomposition of CaCO_3 (99.9%) at 1000°C . SrCuO_2 and Sr_2CuO_3 were prepared in advance by solid-state reactions in air between SrCO_3 (99.9%) and CuO . Appropriate amounts of the starting materials were mixed in an agate mortar and sealed into a gold capsule. By adjusting the mixing ratios of the starting materials, we could change not only the metal contents but also the oxygen content independently. All procedures were performed in a glove box to avoid contamination of carbon dioxide and water in air. The mixtures were allowed to react in a belt-type high-pressure apparatus for 1–3 h at 6 GPa and 1300 – 1350°C , followed by quenching to room temperature before releasing pressure. The weight of the gold capsule was checked before and after the high-pressure run. The weight change was always less than 0.2 mg against the sample weight of ~ 300 mg. Therefore, the oxygen release did not occur during the high-pressure treatment and the net oxygen content in the starting mixture was kept unchanged.

X-ray powder diffraction (XRD) data were collected using a powder diffractometer (Phillips PW 1800) with $\text{CuK}\alpha$ radiation. Lattice constants were determined by the least-squares method. XRD Rietveld analysis was performed using a program RIETAN-2000 [8]. Transmission electron microscopy observations were carried out for a selected sample by a microscope (Hitachi H-1500) operating at 800 kV. Electron probe micro-analysis (EPMA) was done using an angular-dispersion analyzer (JEOL JXA-8600 MX) for a selected sample to know cation ratios of a high-pressure phase. In EPMA, a small ceramic specimen was well polished using diamond paste to obtain a flat surface of $2 \times 2 \text{ mm}^2$ and, several relatively large grains were selected and analyzed. Magnetic measurements were carried out using a SQUID magnetometer (Quantum Design, MPMS) for a lower temperature region and using a vibrating sample magnetometer (VSM, PAR155) for a higher temperature region.

3. Results and discussion

3.1. Synthesis, phase identification and structure analysis

About 50 and 20 starting mixtures were tested for the Sr–Re–Cu–O and Sr–Ca–Re–Cu–O systems, respectively. Some of these high-pressure samples showed ferromagnetism at room temperature. From synthesis experiments of various samples of the Sr–Re–Cu–O system, it was found that the system contains an unknown phase whose XRD pattern is fairly simple. In the lower part of Fig. 1, XRD pattern of a sample with a nominal composition of $\text{Sr}_{1.15}\text{Re}_{0.35}\text{Cu}_{0.5}\text{O}_{3.0}$ is shown. The peaks in this pattern could be indexed assuming a simple cubic lattice with $a = 8.07(1) \text{ \AA}$. This sample had the best phase purity on the phase in

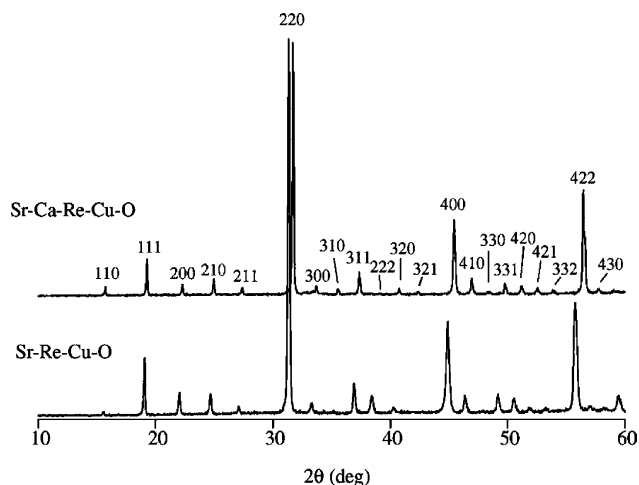


Fig. 1. X-ray powder diffraction patterns of the high-pressure samples with nominal compositions of $\text{Sr}_{1.0}\text{Ca}_{0.175}\text{Re}_{0.325}\text{Cu}_{0.5}\text{O}_{3.15}$ (upper part) and $\text{Sr}_{1.15}\text{Re}_{0.35}\text{Cu}_{0.5}\text{O}_{3.0}$ (lower part).

question among various starting compositions that we tested. An isostructural phase was found in the Sr–Ca–Re–Cu–O system. The best phase purity was attained in this case for a nominal composition of $\text{Sr}_{1.0}\text{Ca}_{0.175}\text{Re}_{0.325}\text{Cu}_{0.5}\text{O}_{3.15}$. The XRD pattern of the Ca-containing sample (the upper part of Fig. 1) could be indexed by a cubic lattice of $7.97(1) \text{ \AA}$ which is slightly smaller than that of the Ca-free phase. The samples shown in Fig. 1 were almost of single phase but trace amount of impurities were included in them though not visible in Fig. 1.

In order to confirm the cubic unit lattice, electron diffraction patterns were observed for the Ca-free phase because it was nonmagnetic (see below) and suitable for the electron microscope observation. Fig. 2 shows electron diffraction patterns projected along the [001] and [111] directions. The pattern projected along [111] has six-fold rotation symmetry (three-fold rotation symmetry in the real structure) confirming the cubic lattice. No systematic extinction is observed in the patterns indicating a space group of $Pm\bar{3}m$ (No. 221) if we select the highest symmetry group.

The nominal compositions of the Ca-free and Ca-containing phases mentioned above may be rewritten as $\text{Sr}(\text{Sr}_{0.15}\text{Re}_{0.35}\text{Cu}_{0.5})\text{O}_{3.0}$ and $\text{Sr}(\text{Ca}_{0.175}\text{Re}_{0.325}\text{Cu}_{0.5})\text{O}_{3.15}$, respectively. They suggest the perovskite-type composition of ABO_3 with the *A*-sites occupied by the Sr atoms and the *B*-sites by the three metals of Sr (Ca), Re and Cu. On the other hand, their lattice parameters ($\sim 8 \text{ \AA}$) imply double periodicity along all the three axes of the perovskite lattice, i.e., $a = 2a_p$, $b = 2b_p$, $c = 2c_p$, for the perovskite lattice of a_p , b_p , c_p .

According to the above consideration, Rietveld analysis of the XRD pattern was carried out for the Ca-containing sample of $\text{Sr}(\text{Ca}_{0.175}\text{Re}_{0.325}\text{Cu}_{0.5})\text{O}_{3.15}$. Fig. 3 shows our structure model for the Rietveld