not confirm the literature data [2], instead we obtained the new phase Sr$_2$Ni$_3$ (P $\bar{3}$lm (Nr. 164), $a = 412.617(1)$ pm, $c = 868.567(1)$ pm) which represents a new structure type. These results focussed our interest on the neighboring system Ba-Ni. For the preparation of Ba$_2$Ni$_3$ elemental barium and nickel are mixed in the molar ration 2:3, pelletized, sealed in niobium ampoules under argon and heated in a high frequency furnace at 1000 °C for 10 minutes. Subsequent annealing at 580 °C for 48 hours results in single phase samples with a metallic luster. DTA investigations yield 610 °C as high-melting metal. Therefore, the synthesis of these compounds with the high-melting RE metal is rather a difficult task. However, two new isomorphic compounds, Lu$_6$B$_3$.33S$_{14}$ (yellow) and Ho$_6$B$_3$.33S$_{14}$ (red), were obtained by a high-temperature/high-pressure route from the elements [1] at 3 GPa (5 h, 1400 °C). The crystal structures were solved and refined using the Rietveld method. Both compounds adopt the Ce$_6$Al$_3$.33S$_{14}$ structure type [2] (hexagonal, P6$_3$, No. 173, Z = 1), with the lattice parameters $a = 9.2104(2)$ Å, $c = 5.8880(2)$ Å for RE = Lu and $a = 9.3703(1)$ Å, $c = 5.8826(1)$ Å for RE = Ho. In the crystal structure of RE$_6$B$_3$.33S$_{14}$ the boron atoms occupy two different sites (2b and 2a) with tetrahedral and trigonal planar coordination by sulfur, respectively. Isolated [BS$_4$]$^{3-}$ tetrahedra, all pointing in the [001] direction, are stacked along the three-fold axis. The [BS$_3$]$^{3-}$ triangles, in which boron atoms are slightly shifted out of the trigonal plane, propagate along the 63 axis. The RE$^{3+}$ ions are surrounded by two [BS$_3$]$^{3-}$ and three [BS$_4$]$^{3-}$ units resulting in an overall 6+1 coordination by sulfur atoms which form a mono-capped trigonal prism. All sulfur atoms coordinate to three RE$^{3+}$ ions. Vacancies in the isotopic Ce$_6$Al$_3$.33S$_{14}$ structure occur at the octahedral Al site (2a site, on 63 axis). Reflecting this fact on the crystal structure, the vacancies are also expected to be at the 2a site. This is in agreement with the results of density-functional-theory based electronic structure calculations [3], carried out on 1x1x3 super cells to model vacancies either at the B1 (2b) or the B2 (2a) positions. The total energies of the models containing vacancies on the B2 site are found to be significantly lower (60 - 70 meV/atom) than those of the models with vacancies on the B1 site. Therefore, the chemical formula can be given as RE[3][B$_2$/3S$_3$][BS$_4$].

Keywords: intermetallic phases; crystal structures; nickel

Multilayer X-ray optics are used on diffractometers mostly for Cu, Co or Cr Ka radiation. For these wavelengths the Q-range in reciprocal space is limited to approximately 8Å⁻¹ which is not enough for some applications. This can be overcome by use of harder X-rays like Ag radiation. Advantages of this higher photon energy are the higher penetration depth and the increased number of detectable reflections in the reciprocal space up to 20Å⁻¹. It is also possible to use capillaries with larger inner diameters without having problems with sample absorption. We will show powder diffraction results measured with parallel beam geometry (TMA – Twin Mirror Arrangement) as well as with focusing geometry. By using high quality X-ray multilayer mirrors it is possible to resolve even very close reflections within a certain angular range as we will show for a LaB₆ capillary powder sample. The diffraction pattern also shows a vastly increased number of reflections. The large Q-range also allows to determine the pair distribution function even for amorphous and nanocrystalline samples.

Keywords: multilayer(s); X-ray diffraction instrumentation; powder diffraction


The usual scheme for crystal structure determination from powder diffraction data consists of (1) indexing the powder pattern, (2) space group determination, (3) structure solution, and (4) structure refinement. Despite the success of methods for powder pattern indexing there are often cases in which the very first step is non-trivial or simply impossible. This means that the subsequent steps are not accessible and structure determination as a whole fails.