not confirm the literature data [2], instead we obtained the new phase Sr_2Ni_3 ($P\bar{3}1m$ (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_2Ni_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 h results in single phase samples with a metallic luster. DTA investigations yield 610 °C for the eutectic temperature and 801 °C for the peritectic decomposition of Ba_2Ni_3. According to these investigations, the Ni-rich part of the system contains further binary phases, which are not yet completely characterized.

Ba_2Ni_3 ($P\bar{3}1m$ (Nr. 164), $a = 419.97(2)$ pm, $c = 913.32(3)$ pm) is isotypic to Sr_2Ni_3. The dominant structural motif consists of corrugated Ni layers which are separated along [001] by planar double layers of barium. The complete coordination sphere around Ni(1) is built up by 6 Ba and 6 Ni(2), Ni(2) is surrounded by 3 Ni(1), 3 Ni(2) and 4 Ba, whereas Ba features 3 Ni(1), 4 Ni(2) and 9 Ba as nearest neighbors. Distances Ni-Ni (245-254 pm) and Ba-Ba (420 pm within the layers, 464 pm between the layers) correspond well with values from the elements (Ni: 244 pm [5], Ba: 434 pm [6]); distances Ba-Ni range between 294 pm and 354 pm.
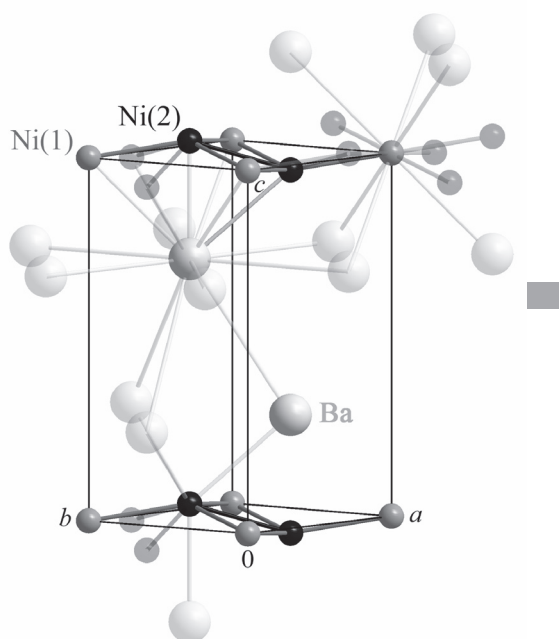
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Keywords: intermetallic phases; crystal structures; nickel

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Crystal Structures of the Rare Earth (*RE*) Thioborates $RE_6B_{3.33}S_{14}$ (*RE* = Lu, Ho). Marija Borna[a], Jens Hunger[a], Dirk Zahn[a], Alim Ormeci[a], Rüdiger Kniep[a]. *[a]Max-Planck-Institut für Chemische Physik fester Stoffe, Dresden, Germany.*
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Our recent experiments have shown that the formation of *RE* chalcogenoborates from elements and/or binaries under autogenous pressure takes place only above the melting point of the *RE* metal. Therefore, the synthesis of these compounds with the high-melting *RE* metals is rather a difficult task. However, two new isomorphic compounds, Lu6B3.33S14 (yellow) and Ho6B3.33S14 (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 Ce6Al3.33S14 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 $RE6B3.33S14$ the boron atoms occupy two different sites (2b and 2a) with tetrahedral and trigonal planar coordination by sulfur, respectively. Isolated $[BS4]^{5-}$ tetrahedra, all pointing in the [001] direction, are stacked along the three-fold axis. The $[BS3]^{3-}$ triangles, in which boron atoms are slightly shifted out of the trigonal plane, propagate along the 6_3 axis. The RE^{3+} ions are surrounded by two $[BS3]^{3-}$ and three $[BS4]^{5-}$ 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 isotypic Ce6Al3.33S14 structure occur at the octahedral Al site (2a site, on 6_3 axis). Reflecting this fact on $RE6B3.33S14$ 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 $RE3[B2/3S3][BS4]$.
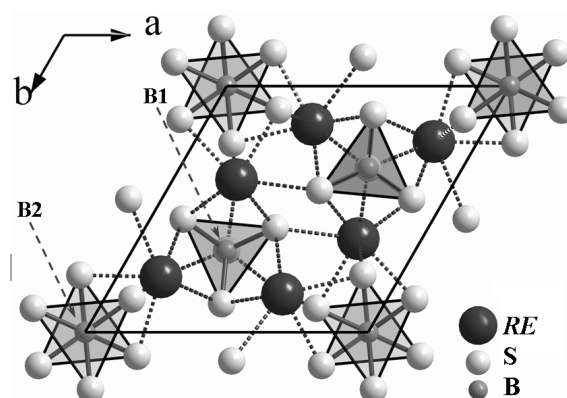

Figure 1. *Crystal structure of RE6B3.33S14 viewed along [001].*

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Keywords: rare earth thioborates; crystal structure analysis; electronic structure calculations

FA5-MS01-P34

Powder Diffraction and Pair Distribution Function Analysis by Using Multilayer X-ray Optics and Ag Radiation. R. Dietsch[a], Th. Holz[a], H. Borrmann[b]. *[a]AXO Dresden GmbH, Siegfried-Raedel-Street 31, 01809 Heidenau, Germany. [b]Max Planck Institute for Chemical Physics of Solids, Nöthnitzer Str. 40, 01187 Dresden, Germany.*
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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 $8 Å^{-1}$ 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 $20 Å^{-1}$. 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 LaB_6 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

FA5-MS01-P35

FIDDLE: Powder Pattern Indexing and Structure Solution Hand in Hand. Jan Smits[a], Carmen Guguţă[a], René de Gelder[a]. *[a]Solid State Chemistry, Institute for Molecules and Materials, Radboud University Nijmegen, The Netherlands.*
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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.

FIDDLE (to be pronounced as 'fit-all') is a direct-space method for the determination of crystal structures from powder diffraction data that uses a different approach for indexing problems. By optimizing *all* model parameters, including the unit cell parameters, the information in peak positions and peak intensities is used simultaneously for finding both the unit cell and the crystal structure. A genetic algorithm, together with a pattern matching technique based on auto- and cross-correlation functions[1], is applied to find the optimal match between observed and calculated diffraction patterns. FIDDLE has the possibility to search through the most common space groups, while varying Z', and does not rely on energy or packing considerations.
Although the method was developed for the complete structure determination process, experience shows that finding the unit cell is very efficient, even when structure solution is not fully complete[2].
Examples of successful applications of the method will be presented.

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Keywords: powder indexing; powder structure determination; optimization algorithms