ESRF	**Experiment title:** Structural and Morphological Investigation of $Ti_{1-x}Hf_xNi_ySn$ Half-Heusler Compounds	**Experiment number**: 31-01-17
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Shifts: 9	**Local contact(s)**: Wouter Van Beek	*Received at ESRF:*

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Report:

The investigation of half-Heusler (HH) compounds in the $Ti_{1-x}Hf_xNi_ySn$ system was performed to recognize the role of excess Ni and/or phase segregation on their structural and thermoelectric properties.

A series of compounds with a nominal composition $Ti_{1-x}Hf_xNi_ySn$, where x = 0.00, 0.10, 0.15, 0.20 and y = 1.0, 1.1, was measured with a high-resolution powder X-ray diffraction instrument at room temperature. During experiment powders were sealed in boron-glass capillaries (d = 0.3 mm).

Collected powder diffraction patterns showed formation of a single HH phase in a sample with the nominal composition $Ti_{0.1}Hf_{0.9}Ni_1Sn$ (Figure 1). In the powder with the same Ti:Hf ratio but containing overstoichiometric Ni, additionally full-Heulser (FH) phase was present (Figure 2).

Based on obtained results, we were able to separate the effect of isovalent substitution of Ti by Hf and presence of extra Ni on the phase formation mechanism in studied compositions. In all $Ti_{1-x}Hf_xNi_ySn$ samples formation of HH compund(s) was confirmed. Presence of overstoichiometric Ni enhanced formation of FH compunds, which were not observed in the 1:1:1 compositions. Rietveld analysis confirmed mixed occupancy of the *4a* site by Ti and Hf atoms in all studied $Ti_{1-x}Hf_xNi_ySn$ phases. Obtained results also showed that Ni atoms were orderly populated over *4c* site in investiagted HH crystal structures and the disordered extra Ni at *4d* site was present only in selected HH compositions. Apart from that, a higher nominal concentration of Hf resulted in an increased abundance of FHs in the sample for all $Ti_{1-x}Hf_xNi_{1.1}Sn$.

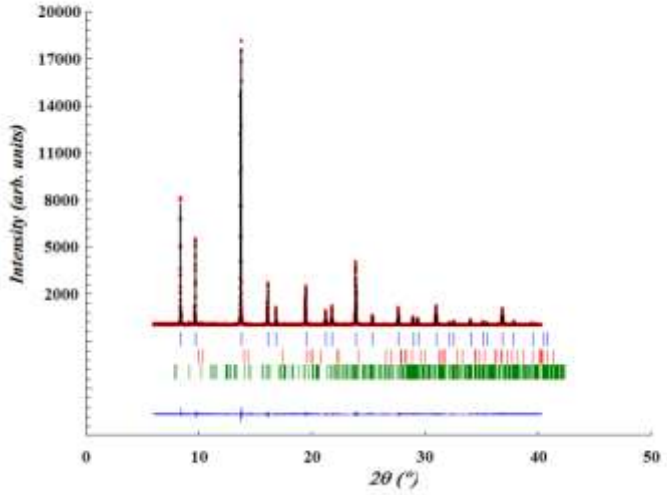

Figure 1. Observed (red line), calculated (black line) and difference (blue line) high resolution diffraction profiles (λ = 0.50218 Å) obtained for $Ti_{0.1}Hf_{0.9}NiSn$ (space group: *P-43m*). Vertical bars indicate Bragg peaks positions of the crystalline phases, from top to bottom: HH, Sn and HfO_2.

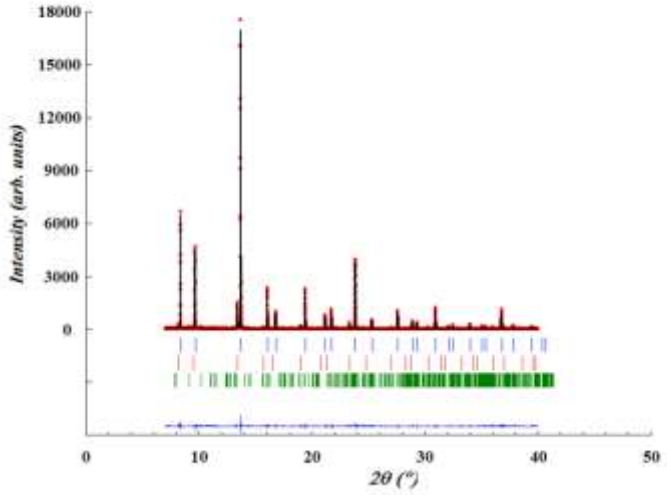

Figure 2. Observed (red line), calculated (black line) and difference (blue line) high resolution diffraction profiles (λ = 0.50218 Å) obtained for $Ti_{0.1}Hf_{0.9}Ni_{1.1}Sn$ (space group: *P-43m*). Vertical bars indicate Bragg peaks positions of the crystalline phases, from top to bottom: HH, FH and HfO_2.