Rapid microwave preparation of thermoelectric TiNiSn and TiCoSb half-Heusler compounds

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Experimental details

Electron microprobe analysis

Characteristic X-ray intensity maps were collected using a Cameca SX100 Electron Microprobe with a LaB_6 electron source and 5 wavelength-dispersive spectrometers. X-rays were generated using a 15 kV accelerating voltage, with 50 nA of beam-current in a focused electron beam. Pure element standards were used to tune wavelength dispersive spectrometers and pulse-height analyzer response for each elements of interest. The following characteristic X-ray lines and analyzing crystals were used: Sn L-α on an LPET crystal, Ti Kα on a PET crystal, Ni Kα on an LLIF, Sb Lα on an LPET, and Co Kα on an LLIF. Secondary electron and Backscatter electron signals were also collected concomitantly. To construct intensity maps, raw on-peak X-ray counts (no background subtraction) were acquired on a 256$\times$256 pixel grid of 1 μm steps with a dwell time of 100 ms per pixel, using continuous microscope-stage/specimen movement and a fixed electron probe.

Hot pressing conditions

Ball-milled powders were filled into the graphite die (POCO EDM-3) whose inner wall was covered with a thin BN layer to protect the material from the graphite. The top surface of the spacer and the bottom surface of the plunger were also treated that way thus that the sample is only surrounded by the BN coating.

The die was loaded into the hot press (OXYGON mini hot press), the load was applied and

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the chamber set under vacuum for several hours (usually over night). All samples were hot pressed under the following conditions: Under pressure, the system was first heated to 323 K with a heating rate of 5 K/min, and then to 573 K with a heating rate of 10 K/min where it was held for 60 min to remove any surface-bound organics. With a heating rate of 15 K/min the temperature was then raised to 1173 K where the powders were pressed for 60 min. After the pressing, the pressure was released and the material was allowed to cool down with a rate of 10 K/min.

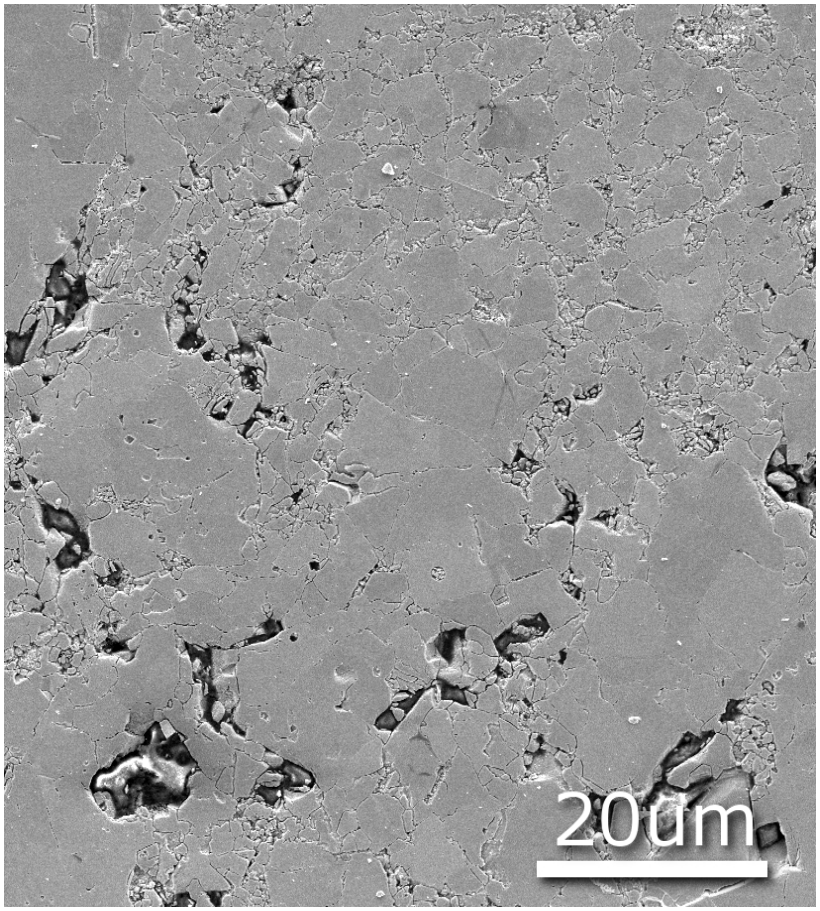

Figure 1: SEM image of the surface of hot-pressed TiNiSn (AM).

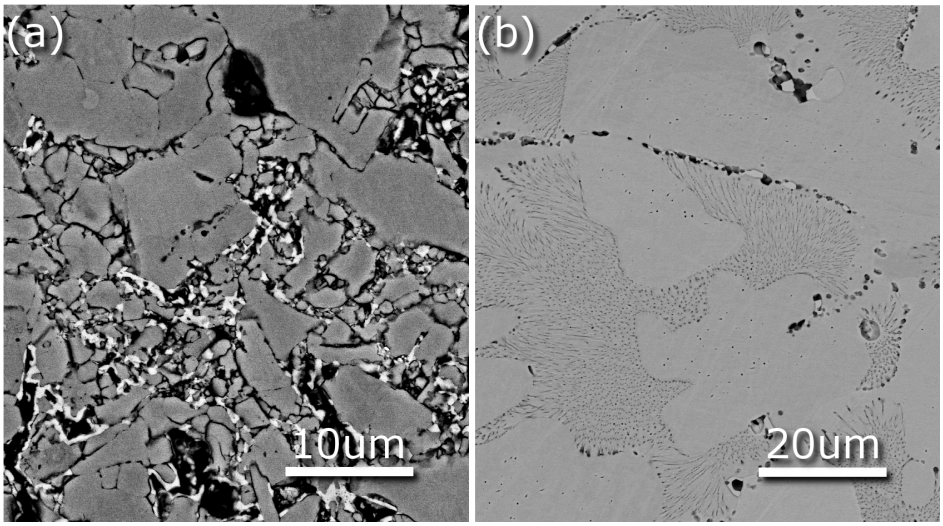

Figure 2: SEM image obtained in backscattering mode of hot-pressed (a) AM TiNiSn and (b) AM TiCoSb. Areas containing elements with high atomic weights show up as lighter regions.

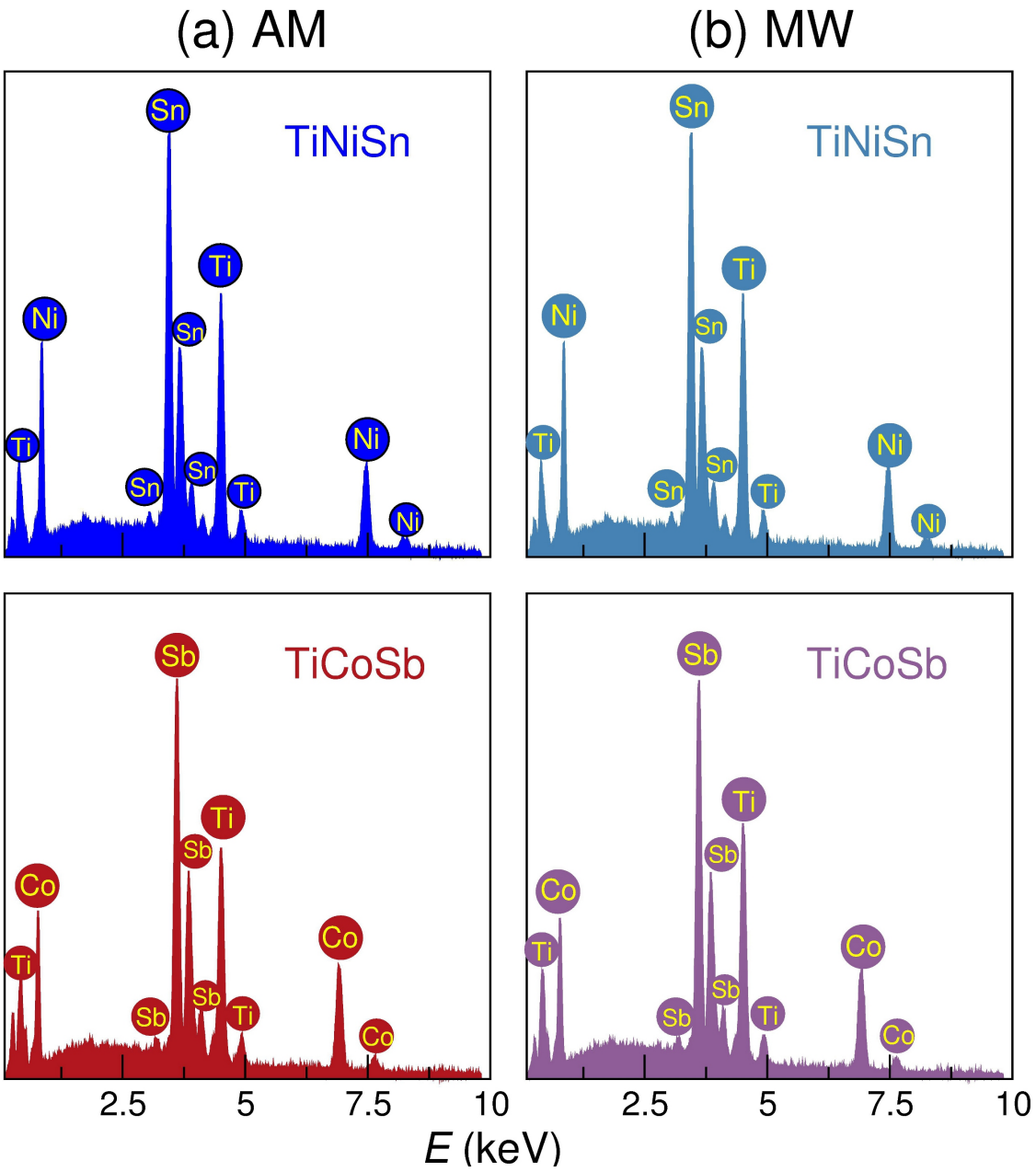

Figure 3: EDX analysis of the surface of the four different samples. The average elemental composition corresponds to a 1:1:1 ratio.

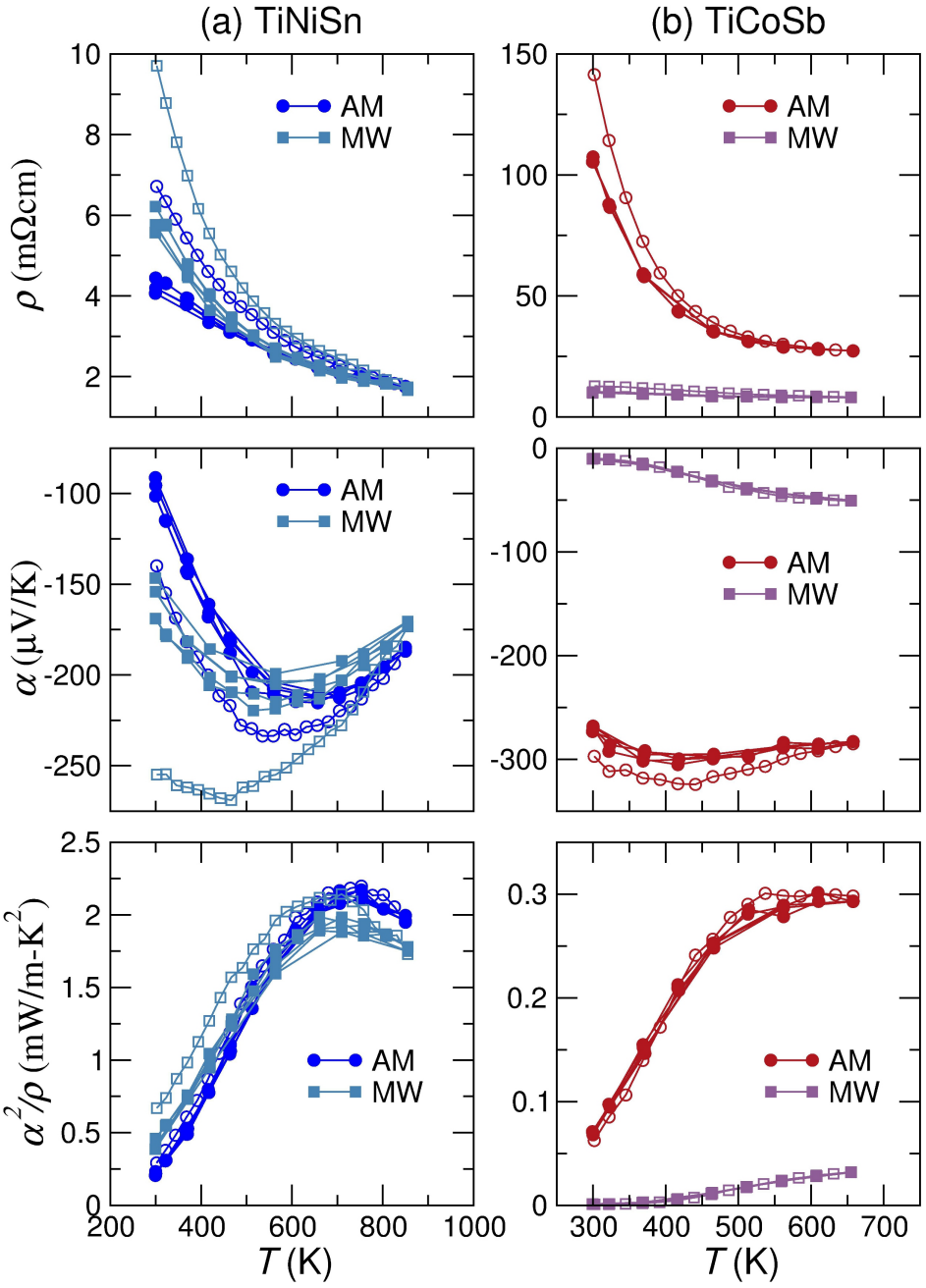

Figure 4: Electronic properties (resistivity, Seebeck coefficient and power factor) of the differently prepared TiNiSn (a) and TiCoSb (b) samples. Open symbols correspond to data obtained during heating while filled symbols correspond to data obtained during cooling and two full additional cycles.