Manufacturing & Prototyping

Mechanical Alloying for Making Thermoelectric Compounds
Constituents are ball-milled into a powder, which is then hot pressed.

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An economical room-temperature mechanical alloying process has been shown to be an effective means of making a homogeneous powder that can be hot pressed to synthesize a thermoelectric material having reproducible chemical composition. The thermoelectric materials to which the technique has thus far been applied with success include rare earth chalcogenides \([\text{La}_x\text{Te}_4 (0 < x < 0.33)\) and \(\text{La}_x\text{Yb}_7\text{Te}_4 (0 < x < 1, 0 < y < 1)\] and \(\text{Zintl}\) compounds (including \(\text{Yb}_3\text{MnSb}_3\) and \(\text{Yb}_3\text{IrSb}_3\)). The synthesis of a given material consists of the room-temperature thermomechanical-alloying process followed by a hot-pressing process. Relative to synthesis of nominally the same material by a traditional process that includes hot melting, this synthesis is simpler and yields a material having superior thermoelectric properties.

The room-temperature mechanical alloying process is, more specifically, a ball-milling process. It begins inside an argon-filled glove box, wherein elemental constituents in amounts corresponding to their desired proportions in the thermoelectric material to be synthesized are loaded into a vial that contains milling balls. The vial and milling balls are made of a material compatible with the material to be synthesized. (For synthesizing \(\text{Yb}_3\text{MnSb}_3\), one uses a vial and balls made of tungsten carbide; for synthesizing \(\text{La}_x\text{Te}_4\) or \(\text{La}_x\text{Yb}_7\text{Te}_4\), one uses a vial and balls made of stainless steel.) Next, the filled vial is removed from the glove box and clamped onto a commercially available mixer/mill machine, which is used to shake the vial for as long as 40 hours to effect ball milling. After ball milling, the vial is returned to the glove box, wherein the powder produced by the ball milling is loaded into a graphite die for hot pressing.

In the case of \(\text{Yb}_3\text{MnSb}_3\), it is necessary to sandwich the powder between two graphite foil layers at each end. In ascending order, the resulting assembly inside the die consists of one or more spacer(s), two graphite foil layers, the powder, two more graphite foil layers, and a plunger that presses down on the aforementioned components.

In the case of \(\text{La}_x\text{Te}_4\) or \(\text{La}_x\text{Yb}_7\text{Te}_4\), the plunger is made of graphite, the inside of the die is lined with graphite foil, and the powder touches the top and bottom spacers, which are coated with boron nitride to prevent adhesion.

The die and its contents are then placed in a hot press, wherein the powder is subjected to a temperature-vs.-time and a pressure-vs.-time profile, specified for the material to be synthesized (for example, see figure), to consolidate the powder into a solid mass of requisite density. After this hot pressing, the mass is removed from the die.

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