High-pressure crystal growth of LnFeAsO (Ln=rare earth)

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Further progress in understanding the nature of superconductivity in the Fe-based compounds depends crucially on the availability sufficiently large single crystals of high-quality. Till now, crystal growth of LnFeAsO oxypnictides (Ln-1111 with Ln=rare earth) has proven to be a difficult task. We adopted the high pressure method and carried out a systematic investigation of the parameters controlling the growth of crystals, including the thermodynamic variables (T, P), reagent composition and kinetic factors, such as reaction time and cooling rate. NaCl/KCl, NaAs, and KAs fluxes were used to grow Ln-1111 crystals at a pressure of 30 kbar. From NaCl/KCl flux, crystals with linear sizes up to 300 μ m were reproducibly obtained. The reaction time was one of the key parameter that influences the crystal size. Millimetersized superconducting Nd-1111 and Sm-1111 single crystals were successfully grown from NaAs and KAs fluxes. This crystal growth starts with a 2 h dwell at 1450 °C, followed by a slow cooling (4-5 °C/h) to $1150 \, {}^{o}C$, and a final to room temperature within 2 h. The size of Ln-1111 crystals suggests that liquid NaAs or KaAs are sufficiently effective solvents, and allow oxygen to diffuse at high temperatures. In addition to substituting F for O, superconductivity has also been induced by substituting Th for Sm, Co for Fe, and P for As. Studies of the crystal structure confirmed high structural quality, and show modifications due to substitutions, which are linked to superconducting properties. The magnetic and transport properties of Ln-1111 crystals are compared with other Fe-based pnictides.