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Crystal growth and detailed structural characterization of superconducting and non-superconducting phases in the  $\mathbf{K}_{1-x}\mathbf{Fe}_{2-y}\mathbf{Se}_2$  system DANIEL SHOEMAKER, DUCK YOUNG CHUNG, MELANIE FRANCISCO, HELMUT CLAUS, SEVDA AVCI, Argonne National Laboratory, ANNA LLOBET, Lujan Neutron Scattering Center, Los Alamos National Laboratory, HEFEI HU, JIAN-MIN ZUO, University of Illinois at Urbana-Champaign, MERCOURI KANATZIDIS, Argonne National Laboratory and Northwestern University — Amid the flurry of activity on  $K_{1-x}Fe_{2-y}Se_2$  superconductors, it remains established that the stoichiometric compound  $K_2Fe_4Se_5$  is an antiferromagnetic semiconductor. This raises the question of whether subtle  $\operatorname{Fe}^{1+/3+}$  doping causes  $\operatorname{K}_{1-x}\operatorname{Fe}_{2-y}\operatorname{Se}_2$  to become a bulk superconductor, and if so, is there a structural distinction between superconducting and non-superconducting phases? We have grown  $K_{1-x}Fe_{2-y}Se_2$ samples that show superconductivity with  $T_C = 31$  K, even when growth conditions are starkly different from those reported in the literature. Here we present high-resolution synchrotron X-ray diffraction measurements, alongside single-crystal x-ray and electron diffraction, to elucidate the phase space in this system. Combined with magnetometry, heat capacity, and transport measurements, our structure-property relations help prescribe how chemical composition and heat treatment induce superconductivity and vacancy ordering in the  $K_{1-x}Fe_{2-y}Se_2$  system.

> Daniel Shoemaker Argonne National Laboratory

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