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A study on iron-chalcogenides superconductors: from samples preparation to physical properties

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Abstract

In the scientific community there is a great interest to explore new superconducting materials suitable for high field applications in order to meet the needs of industrial claims. In this framework, newly discovered Fe-Based Superconductors (IBSC) are a promising choice, especially due to their critical temperature intermediate between low and high *Tc* materials, as well as an extremely high upper critical field.

The aim of this work has been the preparation and the study of physical properties of iron-chalcogenides superconducting samples, in particular polycrystalline FeSe and FeSeTe. The iron-chalcogenides family has been choosen mostly because of its interesting superconducting properties and also due to its simple crystalline structure and to the lack of poisonous elements in its composition.

Opening a completely new research field at the ENEA CR Frascati, several routes of samples production have been carried out. I achieved part of the necessary know-how working also in other laboratories that have great experience on iron-based superconductors preparation, in particular the National Institute for Materials Science (NIMS) laboratories of Tsukuba in Japan, where I worked at the Nano Frontier Materials Group, under the leadership of Prof. Dr. Takano. I also had the chance to spend a brief period at the laboratories of CNR SPIN Genova and the Physics and Chemistry Departments at University of Genova, where I could meet researchers skilled in the production of iron-based samples. Most of the know-how was achieved by direct experience. Even if some of the routes for samples preparation did not brought to the expected results, some of these techniques gave interesting results, other routes deserve further optimization.

Concerning the FeSe compound, two preparation processes have been implemented: the electrochemical deposition on iron substrate, and the solid state reactive synthesis. The former gave FeSe thin films containing the right tetragonal β-phase, but the optimization of the superconducting properties in these samples would be very challenging and time-consuming. The solid state reactive sintering lead to the preparation of superconducting samples with good *Tc* onset but containing several impurities, which compromised the steepness of transition and the current carrying capability. This route requires further optimization, which can be achieved keeping cleaner all the process steps.

Three routes were implemented for the preparation of FeSeTe samples, the solid state reactive synthesis, the mechano-chemical synthesis and the synthesis by fusion. The first two routes, as happened for FeSe samples, need further optimization.

The third route brought to the preparation of several very good polycrystalline samples by a melting process, with heat treatment (HT) at temperatures of about 970 °C followed by cooldown to about 400 °C. It was verified that, as a consequence of the fusion process, impurities and spurious phases between grains are mostly removed, a preferential orientation of the samples is promoted and the critical current is enhanced. Therefore this fabrication route is recommended in view of applications, even if further efforts are needed to develop the material ready to use for example as a target for films deposition or eventually for the preparation of actual strands.

In this work the main physical characterizations performed on all kinds of produced samples are shown. The reproducibility of the superconducting properties of samples

prepared with the same procedure has been verified and only the representative samples for each group have been shown for clarity and readability.

In particular the performing samples have been object of an extensive characterization, carried out in different superconducting labs at ENEA CR Frascati, at Master lab of CNR-SPIN Salerno and Physics Department of University of Salerno. Beside structural, magnetic, transport and calorimetric measurements, several analysis concerning the pinning mechanisms acting and competing inside the produced samples have been performed, within the framework of several literature models. As expected, pinning properties strongly depend on the preparation procedures which induces the defect structure into the samples. Magnetic relaxation measurements have supported this analysis, giving a corroborating possible interpretation of the measured peak effect, if present, and to the behaviour of the effective energy barrier as a function of the current density.

In conclusion, despite the undeniable polycrystalline nature of the FeSeTe samples, those obtained by melting process present superconducting properties closely resembling the single crystals ones, with onset temperatures of about 15 K and quite steep transitions. Best performing samples have large hysteresis cycles well opened up to 12 T (at about 9 K) and up to 18 T (at about 7 K) with a robust critical current density weakly dependent on the applied field in the high field range.