Dipartimento di Ingegneria Industriale, Elettronica e Meccanica
Dottorato in Elettronica Applicata - Ciclo XXXVIII

ROMA TRE UNIVERSITÀ DEGLI STUDI

ENEA Agenzia nazionale per le nuove tecnologie, l'energia e lo sviluppo economico sostenibile

TITLE:"DEVELOPMENT OF IRON-BASED SUPERCONDUCTING MATERIALS AND THEIR ELECTROMAGNETIC CHARACTERISATION".

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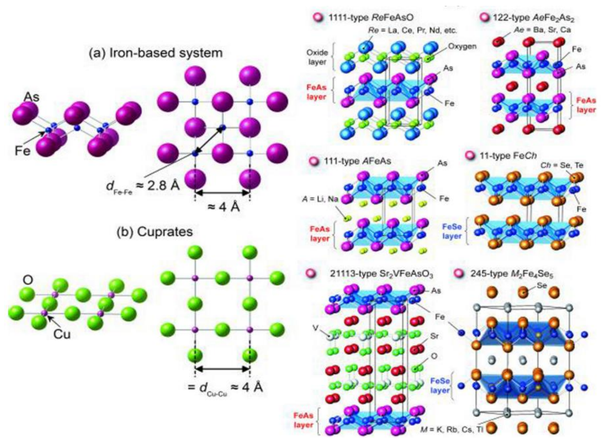
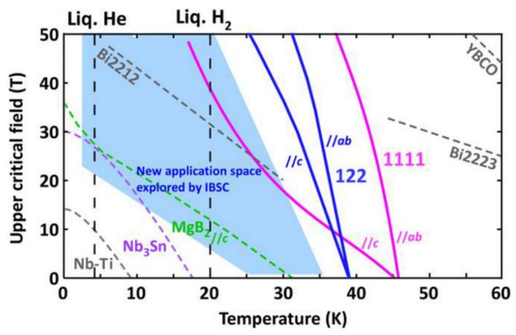
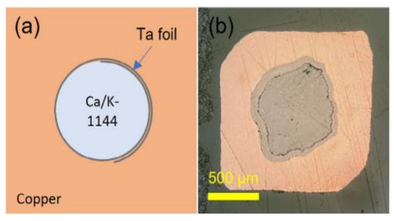


IRON BASED SUPERCONDUCTORS (IBSC), 1144 $AeAFe_4As_4$

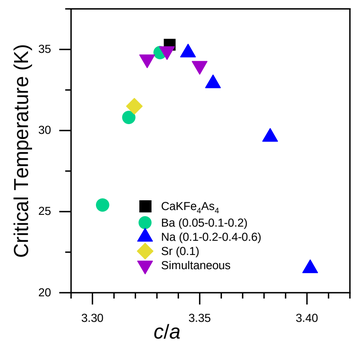
Iron Based SuperConductors (IBSCs) are formed from pnictides (P, As) or chalcogenides (Se, Te) interspersed with various elements and were discovered in 2008 [1]. The different combinations of the elements lead to the formation of compounds with different structural characteristics that are reflected in the superconducting properties and group them into different families fig 1. The critical temperatures Tc of some of these materials reach the threshold of 60 K, with critical ranges exceeding 100 T.

In the application perspective, IBSCs have clear advantages in terms of grain boundary nature, low anisotropy and high crystallographic symmetry of the superconducting phases, all of which make them advantageous in the application of the standard process for bonded superconductors, the PIT method, for wire and tape fabrication. The figure highlights the plausible application space of the Tc-magnetic field relationship. One of the main potential application areas for IBSC superconducting wires is high magnetic fields, where they show excellent robustness [4].





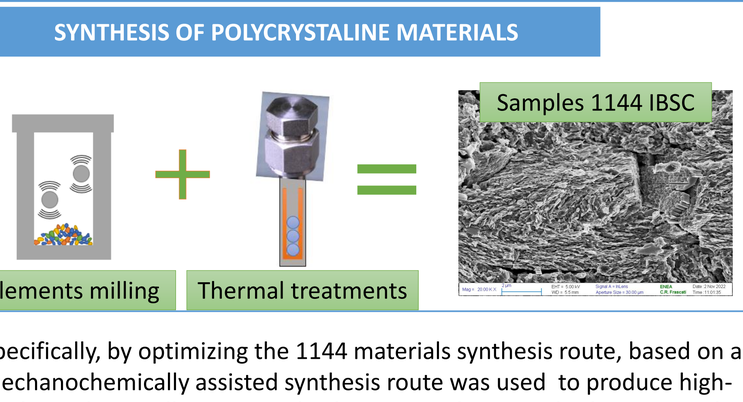
The potential of the 1144 family has not yet been fully reached, showing possible improvements in the production of superconducting wires using the Power in Tube (PIT) method [2] and [3] .
The quality of policristalline powders needs improvements (less contamination, enhanced sintering).



It has been discovered through targeted chemical substitutions how this plays a key role on critical temperature values [5]. Now it is crucial to understand how the chemical composition influences the intrinsic properties of the system, in particular for example the critical current density..

AIM I: INVESTIGATION AND OPTIMISATION OF THE MICROSTRUCTURAL CHARACTERISTICS OF POLYCRYSTALLINE MATERIAL.

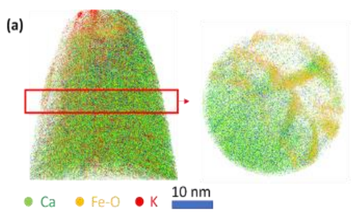
SYNTHESIS OF POLYCRYSTALINE MATERIALS



Specifically, by optimizing the 1144 materials synthesis route, based on a mechanochemically assisted synthesis route was used to produce high-quality polycrystalline materials, that were subsequently characterized morpho-structurally and with Critical Temperature measurements.

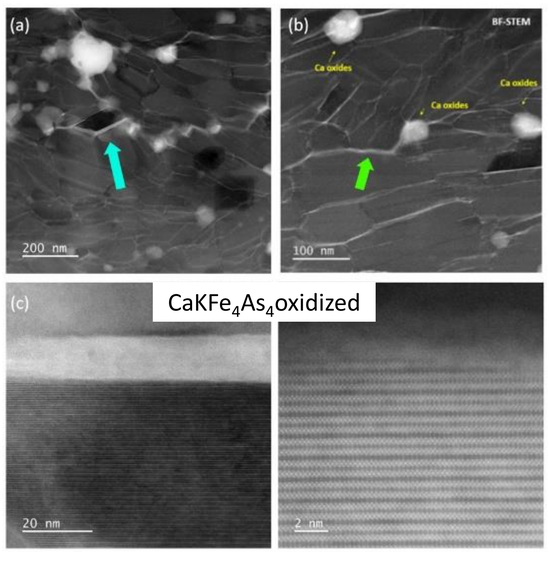
ANALYSIS OF THE CHEMICAL AND MORPHOLOGICAL NATURE OF GRAIN EDGE AND ITS WEIGHT ON T_c

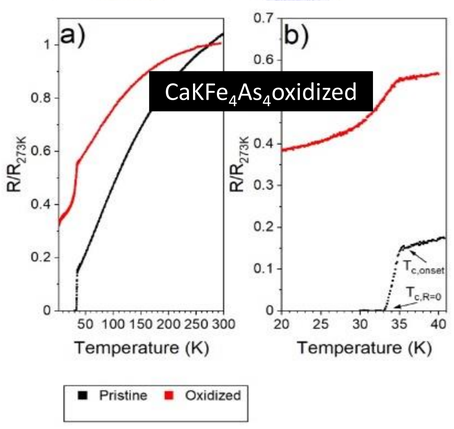
$CaKFe_4As_4$oxidized



The chemical compositions in the grain boundary of $CaKFe_4As_4$ show that oxygen is unevenly distributed and segregated at the grain boundaries. The distribution of Ca indicates that Ca is also segregated at the grain boundaries. Ca has the highest affinity for O among Ca, K, Fe, and As, and **the segregation of Ca in grain boundaries is rationalized by the formation of Ca oxides in the grain boundaries of $CaKFe_4As_4$** [6] .

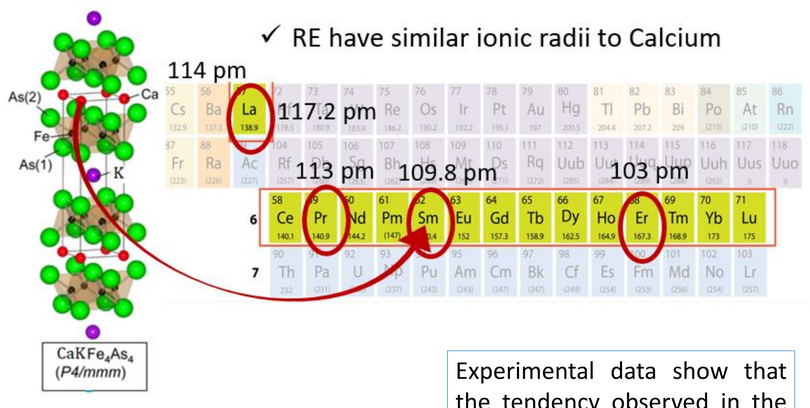
The image shows an elongated grain structure, with grain sizes ranging from 50 to 200 nm, and **bright contrasts clarifying oxidations at the boundaries** [6] .





The oxidized samples show a significant broadening of the superconducting transition. The onset does not vary significantly, as is evident in Fig. 5, but zero resistance is not observed until low temperature. Considering the nature of the resistance measurement, which probes the existence of percolative pathways through the sample, this suggests that **oxidation phenomena promote the formation of non-superconducting blocking layers through the material** [6] .

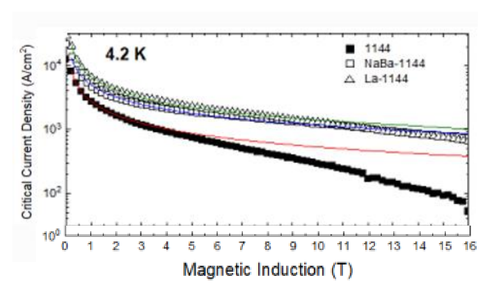
AIM II: THE STUDY OF THE EFFECT OF CHEMICAL COMPOSITION ON THE SUPERCONDUCTIVE AND STRUCTURAL PROPERTIES OF SYNTHESIZED MATERIALS.

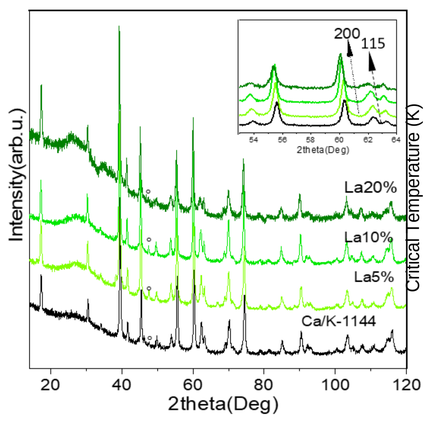


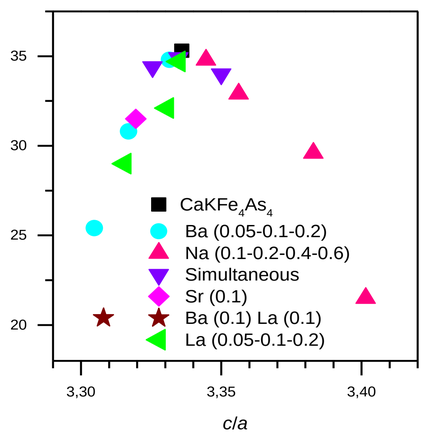
The possibility of obtaining partially substituted Ca/K-1144 compounds with the with rare-earths (RE, Rare - Earths) (La, Pr, Sm and Er) and with RE together with A or Ae (Na and La, Ba and La) and the effects of the substitutions with the material properties were investigated [7].

From XRD analyses, it was observed that La can be used as substituent in Ca/K-1144 compounds, giving rise to a clean phase and to a distortion of the lattice [7].

Experimental data show that the tendency observed in the trend of critical temperatures as a function of tetragonality ration (c/a) is confirmed with Rare Earths and not only limited to alkali and alkaline earth metals [7].







Aliovalent substitution results in improved Jc(B) dependencies, compared to the unsubstituted CaKFe4As4 sample [8]. The chemical substitution brings with it both structural changes in the sample and a change in the chemical nature of the grain boundary, since in polycrystalline samples we cannot separate the contribution of the grain boundary on the superconductive effects it is not possible to attribute this enhancement effect to either factor. This remains an aspect to be investigated.

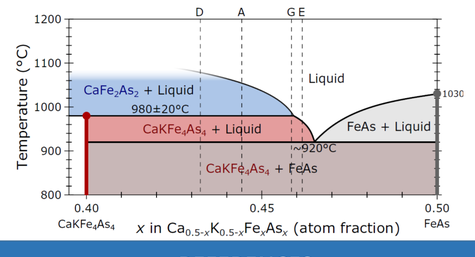
THE EXPERIMENTAL EVIDENCE ALLOWED US TO OBSERVE ISSUES RELATED TO EXTRINSIC PROPERTIES (THE DEGREE OF AGGREGATION, DENSITY, QUALITY, AND GRAIN EDGE ORIENTATION) OF 1144 POLYCRYSTALLINE SYSTEMS, SUGGESTING THE NEED FOR THE STUDY OF SINGLE CRYSTALS OF 1144 SYSTEMS THAT WOULD ALLOW BY COMPARISON, TO BETTER ISOLATE THE CONTRIBUTION OF EXTRINSIC PROPERTIES PROVIDE, AND TO ANALYZE THE INTRINSIC PROPERTIES (THE CHEMICAL COMPOSITION, CRYSTAL STRUCTURE, AND ELECTRONIC STATE WILL BE) AND THEIR CONTRIBUTION ON THE SUPERCONDUCTIVE PROPRETIES OF THE MATERIAL.

Future developments: Growth and caracterization of variosly substituted single crystals

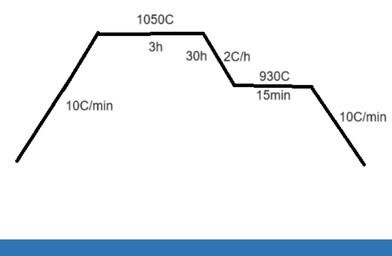
The synthesis of crystals of compounds 1144 is complex because unwanted secondary phases such as FeAs, KFe_2As_2 and $CaFe_2s_2$ can be formed, and to avoid this, the experimental conditions of FeAs -flux synthesis will have to be investigated and studied.

Reactor design

Phase diagram



Thermal profile



SCIENTIFIC PRODUCTION

[I] Z-H Sung, A Masi, J-Y Lee, **A Duchenko**, X Bing, D Isheim, D N Seidman, and G Celentano "Detailed microstructural investigation of oxidation phenomena in 1144 FBS" accepted by the review IEEE TAS."
[II] A.Masi, Achille Angrisani Armenio, Andrea Augieri, G. Celentano, **A. Duchenko**, N. Pompeo, A. Rufoloni, A. Vannozzi, F. Varsano" Development of Ca/K-1144 IBS wires with composite Cu/Ta sheaths", under review by IEEE TAS.
[III] **A. Duchenko**, A. Augieri, L. Barba, G. Celentano, A. Masi, N. Pompeo, F. Rizzo, A. Rufoloni, F. Varsano "Influence of Rare-Earth inclusion on structure and properties of Ca/K-1144 IBSC" under review by IEEE TAS.
[IV] **A. Duchenko**, A. Augieri, L. Barba, G. Celentano, A. Masi, N. Pompeo, F. Rizzo, A. Rufoloni, F. Varsano, "Relation between Structure and Superconductivity in Substituted CaKFe4As4" accepted by the review IEEE TAS
[V] A. Augieri**, A. Duchenko**, G. De Marzi, F. Varsano, G. Celentano, N. Pompeo, A. Masi"Pinning properties of 1144 polycrystalline samples with aliovalent doping" under review by IEEE TAS
[VI] Sung, Z., Duchenko, A., Celentano, G., Lee, J., Hu, X., Pompeo, N., Varsano, F., & Masi, A. (2023). Oxidation in Ca/K-1144 iron-based superconductors polycrystalline compounds. Superconductivity, 8. https://doi.org/10.1016/j.supcon.2023.100062

Oral presentation titled "Influence of Rare-Earth inclusion on structure and properties of Ca/K-1144 IBSC" at the international conference 16th European Conference on Applied Superconductivity (EUCAS).

REFERENCES

[1] Y. Kamihara, T. Watanabe, M. Hirano, H. Hosono, "Iron-based layered superconductor La[O1- xFx]FeAs (x= 0.05-0.12) with Tc = 26 K." *Journal of the American Chemical Society*, Vol. 130 (2008), 3296–3297.
[2] A. Masi, A. A. Armenio, A. Augieri, G. Celentano, **A. Duchenko** "Cu/Ta sheaths for iron- based superconductors: First experimental findings in Ca/K-1144 wires" ELSEVIER, Vol. 2,(2022).
[3] A.Masi, Achille Angrisani Armenio, Andrea Augieri, G. Celentano, **A. Duchenko**, N. Pompeo, A. Rufoloni, A. Vannozzi, F. Varsano" *Development of Ca/K-1144 IBS wires with composite Cu/Ta sheaths*", in fase di revisione da *IEEE TAS*.
[4] Hosono, H., Yamamoto, A., Hiramatsu, H., & Ma, Y. (2018). Recent advances in iron-based superconductors toward applications. In *Materials Today* (Vol. 21, Issue 3, pp. 278–302). Elsevier B.V. https://doi.org/10.1016/j.mattod.2017.09.006
[5] Masi, A., **Duchenko**, A., Celentano, G., & Varsano, F. (2022). Tailoring the critical temperature of Ca/K-1144 superconductors: The effect of aliovalent substitution on tetragonality. *Superconductor Science and Technology*, *35*(6). https://doi.org/10.1088/1361-6668/ac6630
[6] Z-H Sung, A Masi, JY Lee**, A Duchenko**, X Bing, A G Kim, and G Celentano"*Oxidation in Ca/K-1144 Iron based superconductors polycrystalline compounds" accepted by IEEE TAS*
[7] **A. Duchenko**, A. Augieri, L. Barba, G. Celentano, A. Masi, N. Pompeo, F. Rizzo, A. Rufoloni, F. Varsano "*Influence of Rare-Earth inclusion on structure and properties of Ca/K-1144 IBSC" under review by IEEE TAS.*
[8] A. Augieri**, A. Duchenko**, G. De Marzi, F. Varsano, G. Celentano, N. Pompeo, A. Masi"*Pinning properties of 1144 polycrystalline samples with aliovalent doping" under review by IEEE TAS*

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