Phase separation and microstructure in superconducting $FeSe_{1-x}Te_x$ materials

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Aim

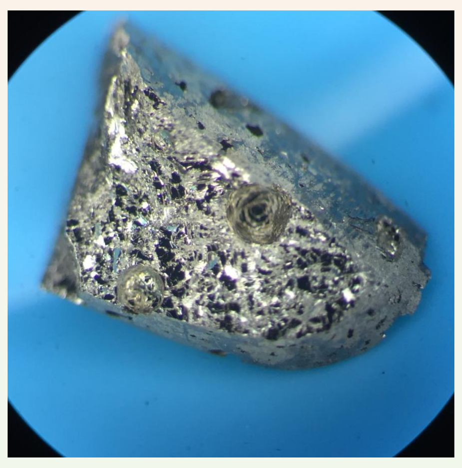
- ❖ Explore the relations between the crystallization processes occurring in the Fe(Se,Te) system and the materials properties
- ❖ Focus on $FeSe_{0.5}Te_{0.5}$ materials obtained from high temperature melts
- ❖ Evaluate how the cooling step influences nucleation and growth processes

Experimental

Fe, Se, Te elemental powders, mixed and compressed into small pellets and sealed in vacuum in quartz vials

Thermal treatment:
- ➢ R.T. → 1000 °C at 1 °C/min
- ➢ 4 h @1000°C
- ➢ Different cooling procedures:
  - ❖ Controlled cooling at 0.2 °C/min
  - ❖ Controlled cooling at 5 °C/min
  - ❖ Quench in liquid nitrogen

Shiny and brittle specimens are obtained, easily cleaved through specific directions

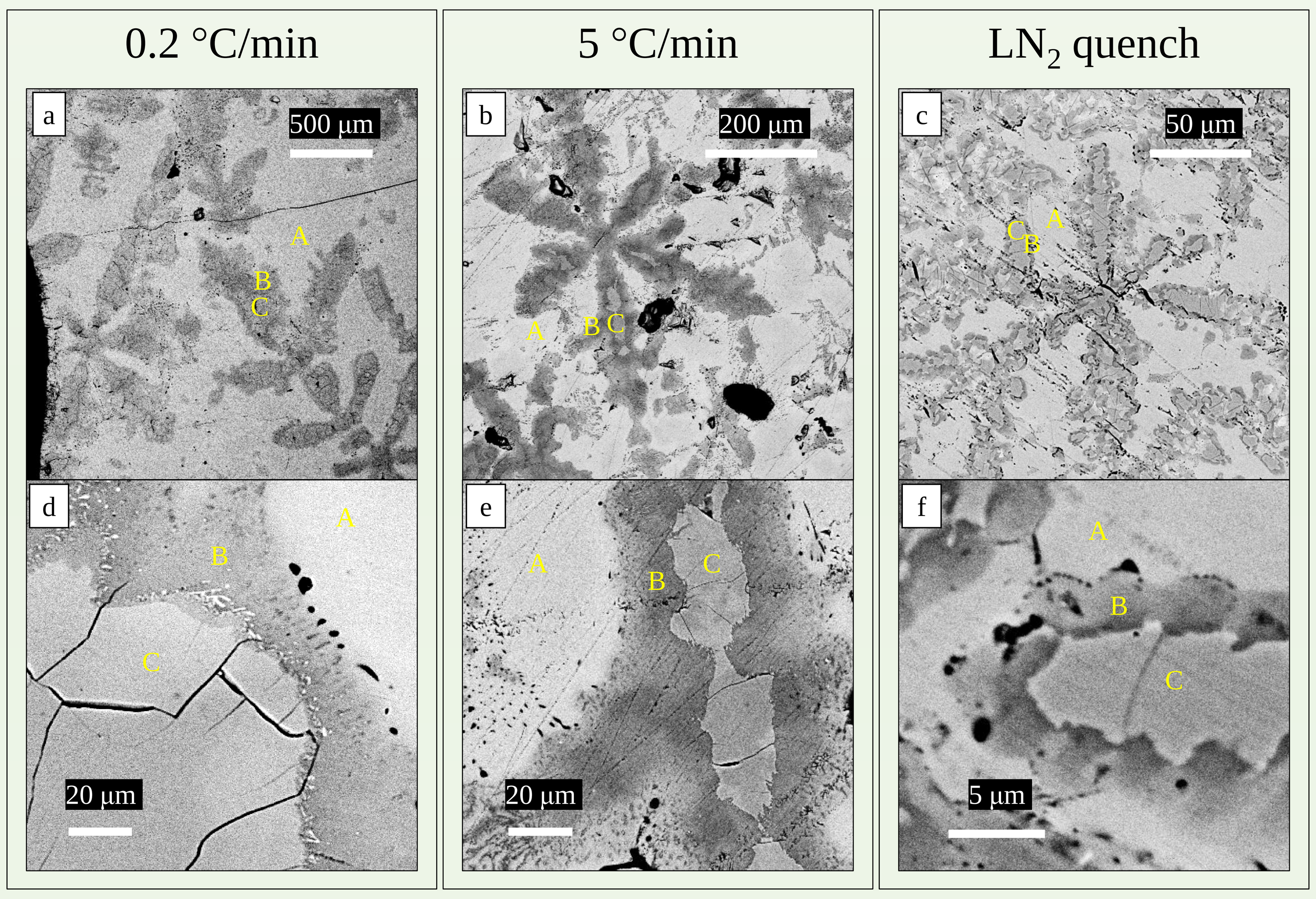


density = 6.00 ± 0.05 g/cm^3

Characterization techniques
SEM/EDX analysis: LEO 1525 FE-SEM, polished sections
XRD analysis: Rigaku SmartLab, 9 kW rotating anode, pulverized samples
Magnetic susceptibility: Oxford Instrument VSM, ZFC, irregular fragments
Electric resistance: cryo-free system, 18 T superconducting magnet, irregular fragments

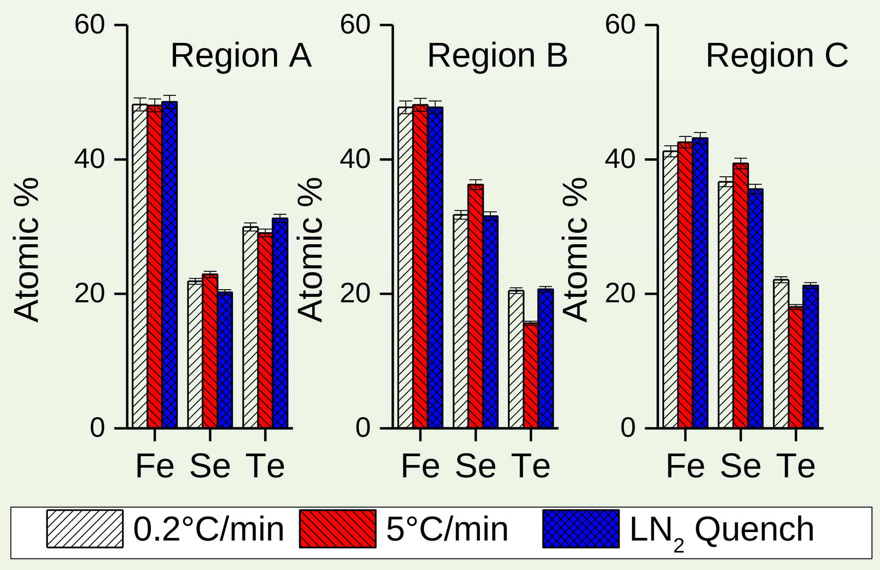
Results

SEM images

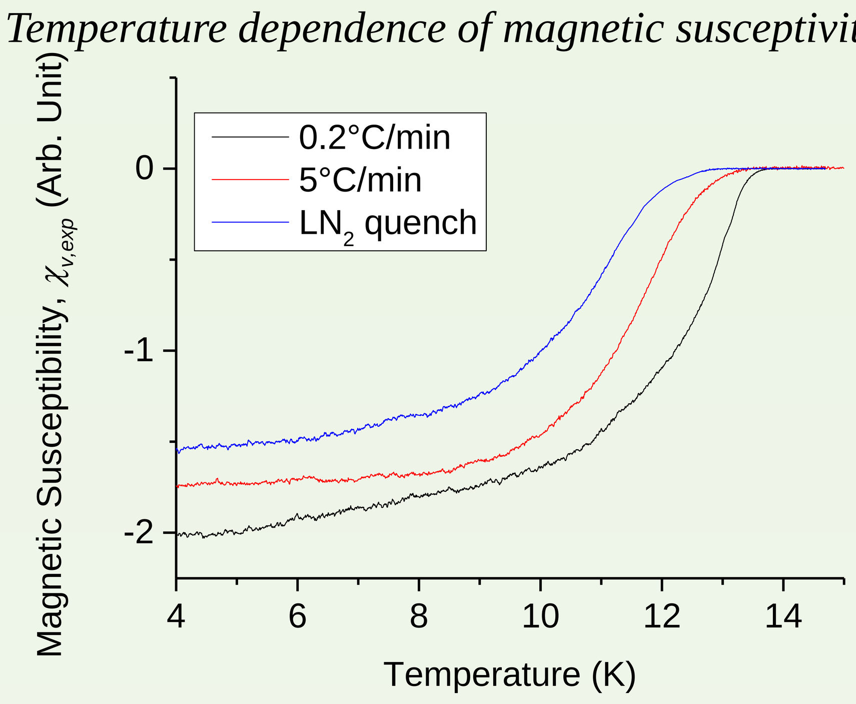


SEM/EDX
- Multi-phase structures observed in all samples.
- Size of dendritic precipitates inversely related with cooling rate
- All samples characterized by the presence of three regions: an interconnected matrix (A in figure), an interfacial zone (B), and the core of the dendritic precipitates (C)
- Among the three samples, the composition of each different regions is similar

Summary of EDX results

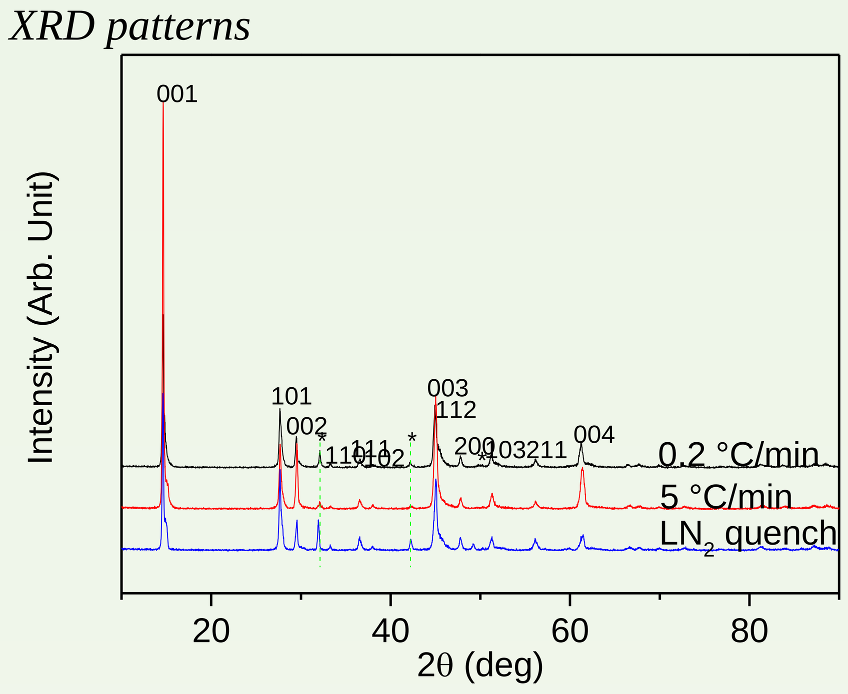


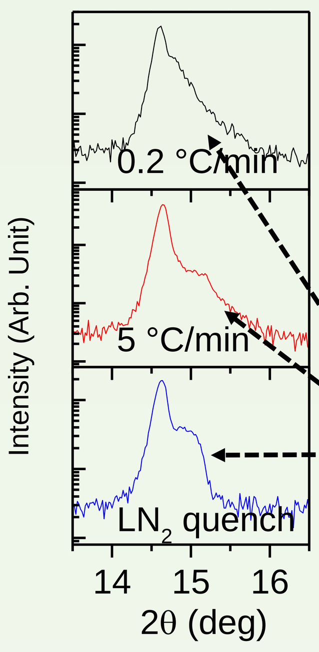
Temperature dependence of magnetic susceptivity



Magnetic behaviour
All samples exhibit a clear diamagnetic signal below T_c. The critical temperature decreases from 13.5 K to 12.9 K and 12.1 K when raising the sample cooling rate

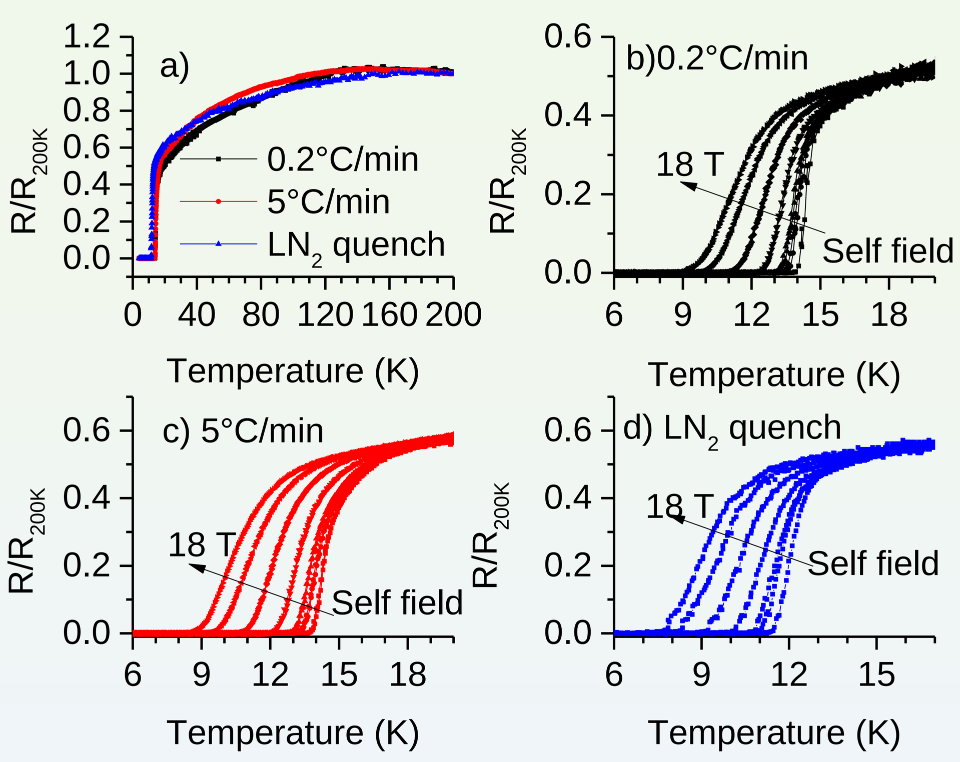
XRD patterns





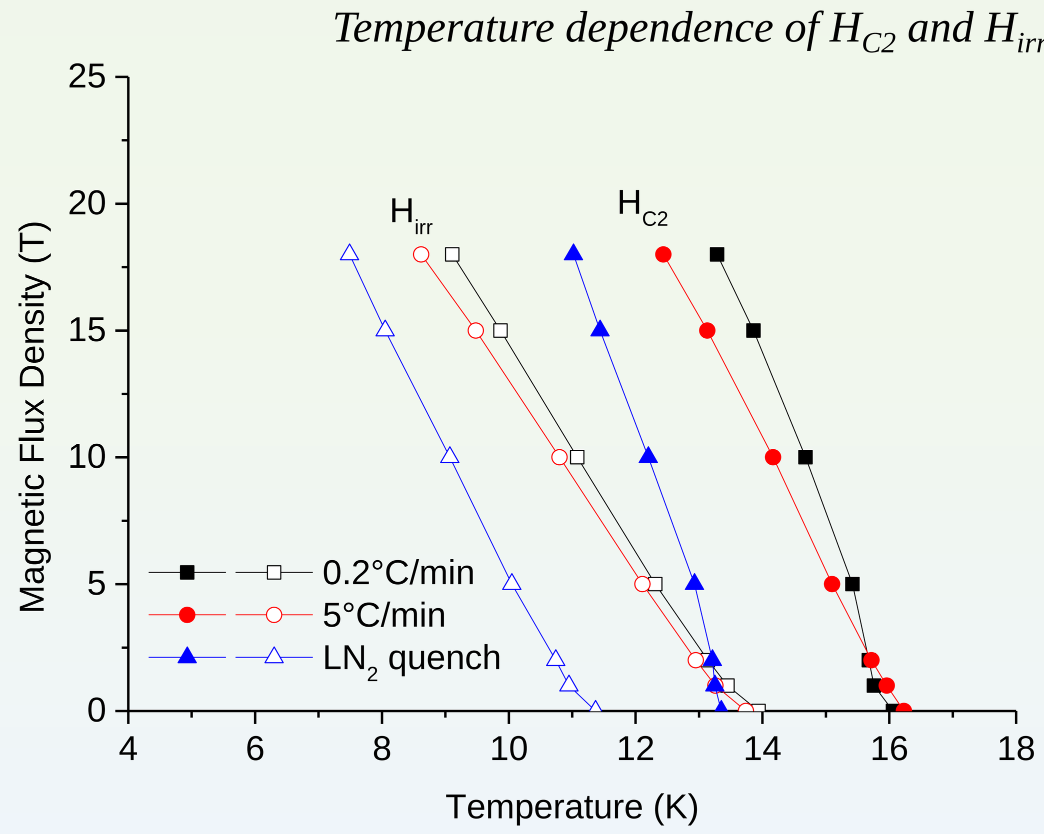
XRD
Samples are characterized by multi-phase patterns
- main peaks ascribable to a tetragonal β-Fe(Se,Te) phase
- Secondary peaks match with hexagonal δ-FeSe phase or Fe_7Se_8-like phase
- High angle asymmetrical broadening of (00ℓ) reflections is compatible with the presence of mixtures of tetragonal phases characterized by different Se content

Temperature dependence of electrical resistance



Electrical behaviour
- At low temperatures, all the samples exhibit a sharp drop of the resistance
- With increasing fields, the resistive transition shifts to lower temperatures
- Higher critical temperature for conventional cooling
- Higher critical fields calculated when lowering the cooling rate

Temperature dependence of H_{C2} and H_{irr}



Conclusions

- The formation of dendritic precipitates, characterized by the presence of a Se-rich core, in a β-Fe(Se,Te) matrix has been observed for all samples. A "transition" interface between the core and the matrix is always observable in the different samples.
- The morphology and composition of the crystalline phases seem independent on the cooling rate, while the size of the precipitates is greatly affected, suggesting that synthesis cooling rate influences the kinetic of the occurring nucleation and growth phenomena rather than their nature.
- All samples exhibit broad magnetic and electrical superconducting transition, most likely due to their inhomogeneous nature.
- The superconducting behaviour improves decreasing the cooling rate.