

CRYSTALLIZATION OF AMORPHOUS FeF₃ FLUORIDES

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Several amorphous fluorides can be obtained from high-temperature vapour-deposition on a cold substrate. In the case of FeF₃, the nature of the deposited sample is quite influenced by preparation conditions: the presence of a small amount of divalent iron (rutile structure) is observed and is related mainly to pressure and temperature in the furnace.

In other hand, another amorphous fluoride FeF₃.xHF (0.4 ≤ x ≤ 1) was synthesized by a soft chemistry reaction: no divalent iron was observed in this compound.

Annealing treatments on both amorphous forms of FeF₃ were realized under vacuum in a Mössbauer cryofurnace: crystalline phases were obtained, and the resulting composition was strongly dependent on the heating modes and vacuum quality. These phases were identified by Mössbauer spectroscopy and X-Ray diffraction.

The more significant results are:

- FeF₃.xHF crystallizes into rhombohedral FeF₃ without any formation of divalent iron, whatever the heating procedure [1].
- Isothermal treatments under high vacuum showed that deposited FeF₃ led to rhombohedral FeF₃ [2].
- Vapour-deposited FeF₃ crystallizes into hexagonal bronze tungsten FeF₃ when hydrolysis phenomena are induced during the annealings; an increase of divalent iron rate is observed [3].

All these studies, discussed in [1,2,3], show that the structure of the 'ideal' amorphous FeF₃ is closely related to the rhombohedral phase.

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