



Experiment title: High pressure synthesis of nitrogen-rich iron nitrides

Experiment number: CH5255

Beamline: ID27	Date of experiment: from: October 25 th 2018 to: October 28 th 2018	Date of report: 20/02/2018
Shifts: 9	Local contact(s): Mohamed Mezouar	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):

Agnès Dewaele,* CEA, DAM, DIF, F-91297 Arpajon, France

Dominique Laniel,*CEA, DAM, DIF, F-91297 Arpajon, France

Report:

The abstract of the article in press in *Inorg. Chem.* is copied below. This article is based on the measurements performed during CH-5255.

The high pressure chemistry of transition metals and nitrogen was recently discovered to be richer than previously thought, due to the synthesis of several transition metal pernitrides. Here we explore the pressure-temperature domain of iron with an excess of nitrogen up to 91 GPa and 2200 K. Above 72 GPa and 2200 K, the iron pernitride FeN₂ is produced in a laser-heated diamond anvil cell. This iron-nitrogen compound is the first with a N/Fe ratio greater than one. The FeN₂ samples were characterized from the maximum observed pressure down to ambient conditions by powder X-ray diffraction and Raman spectroscopy measurements. The crystal structure of FeN₂ is resolved to be a *Pnnm* marcasite structure, analogously to other transition metal pernitrides. Based on the lattice's axial ratios and the recorded N-N vibrational modes of FeN₂, a bond order of 1.5 for the nitrogen dimer is suggested. The bulk modulus of the iron pernitride is determined to be of $K_0 = 344(5)$ GPa, corresponding to an astounding increase of about 208% from pure iron. Upon decompression to ambient conditions, a partial structural phase transition to the theoretically predicted *R-3m* FeN₂ is detected.

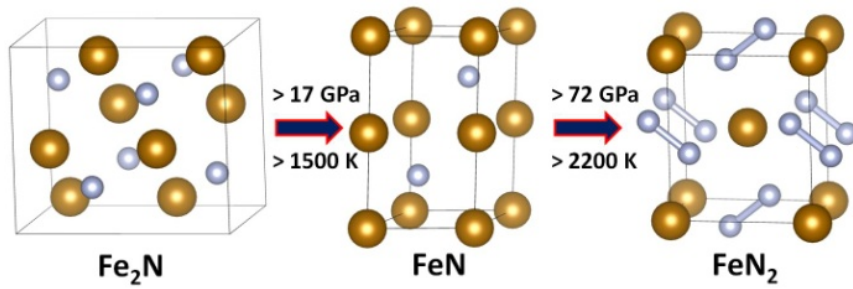


Figure 1: Pressure- and temperature-induced chemical reactions observed in iron embedded in an excess of nitrogen.