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ESRF	**Experiment title:** **Investigation of the high P and T structure of Fe alloys relevant to the Earth's core.**	**Experiment number:** **HS-737**
Beamline: **ID 30**	**Date of experiment:** from: **16/09/98** to: **20/09/98**	**Date of report:** **25/02/99**
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Report:

We performed angle dispersive X-Ray diffraction studies in a YAG laser-heated diamond anvils cell (DAC). There were two goals for this project. The first aim was to investigate the high pressure and high temperature phase diagram of iron-rich silicon alloys relevant to the Earth's core composition. Two samples were studied: $Fe_{0.91}Si_{0.09}$ and $Fe_{0.83}Si_{0.17}$. The second aim was to study some iron-rich alloys that could allow a better understanding of pure iron phase diagram. $FeNi_{0.1}Cr_{0.18}$ stainless steel was chosen for this study as it seemed able to quench high pressure polymorphs [1].

For these 2 experiments, we used a DAC with large optical aperture mounted with diamond anvils with Ø 300 µm culets. Rhenium gaskets were preindented to a thickness of 50 µm and drilled to a diameter of 100 µm. Powdered samples from Good Fellows were used as starting materials. Two different pressure transmitting media were used: NaCl (up to 25 GPa) and Ar (up to 65 GPa). Pressure was derived from the volume measurement of those media according to Birch's equation of state for NaCl [2] and Ross et al. equation of state for Ar [3]. For technical reasons we were not able to perform in-situ high temperature measurements. As a result, YAG laser heating was used only for annealing samples.

Angle dipersive measurements were performed at the ID30 high pressure beamline. The X-Ray beam of wavelenght 0.3738 Å was selected from an undulator using a channel cut Si(111) monochromator. Full reciprocal angle data were collected using the Fastscan detector. 2-D patterns were then integrated after geometrical corrections using the program Fit2d. Lebail and Rietveld refinements were performed using the

GSAS package.

Results obtained:

1) Compression of $Fe_{0.91}Si_{0.09}$ and $Fe_{0.83}Si_{0.17}$.

At ambiant conditions these two samples exhibit bcc structure with FeAl type surstructure. The 2 samples were compressed up to 51GPa for $Fe_{0.91}Si_{0.09}$ and 64 GPa for $Fe_{0.83}Si_{0.17}$. The most striking result is that both samples remain cubic at these pressures: no transition to an hcp phase was observed. Thanks to YAG laser annealing and the use of soft pressure transmitting media, the data collected are good enough to calculate accurate equations of state at ambiant temperature (see figure 1).

2) Compression of $FeNi_{0.1}Cr_{0.18}$.

At ambiant conditions, $FeNi_{0.1}Cr_{0.18}$ exhibits γ-fcc structure. We compressed the sample from 9 GPa up to 40 GPa with Argon as a pressure transmitting media. Up to 33 GPa, we observed a mixing between the γ-fcc phase and the ε-hcp phase. Above this pressure, only the ε phase remains. After the release of pressure the structure of the sample was found to be ε-hcp mixed with a small amount (about 15%) of γ-fcc (see figure 2).

This result is of extreme importance for further investigations: the stainless steels seem able to quench high pressure (and perharps high temperature?) pure iron-like polymorphs. This should allow us to perform more accurate studies of those polymorphs.

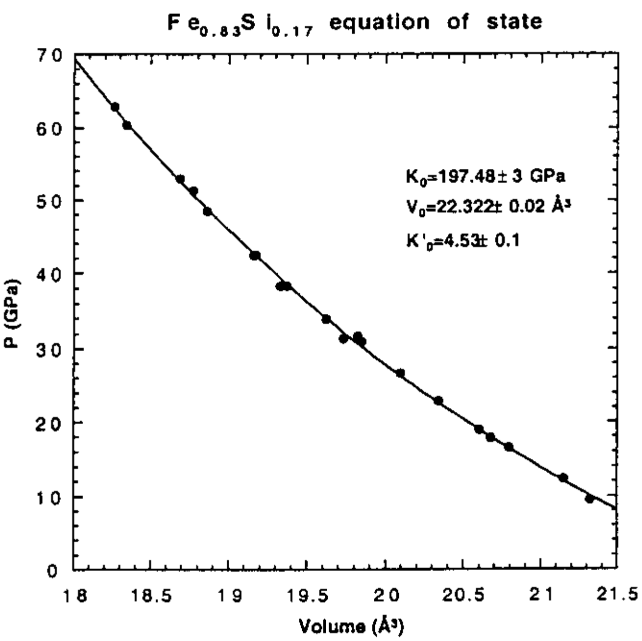

FIGURE 1: Equation of state of $Fe_{83}Si_{17}$. The plain curve is the third order Birch-Murnaghan equation of state derived from the data. Calculated parameters are listed above.

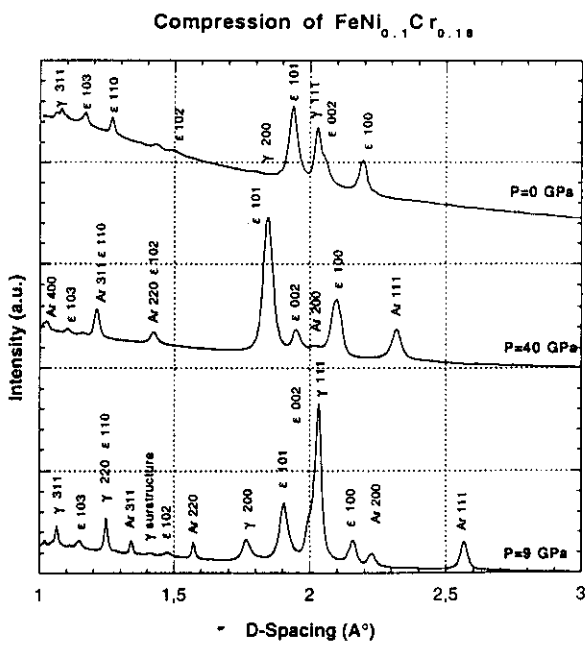

FIGURE 2: Compression of $FENi_{0.1}Cr_{0.18}$. After the release of pressure (upper curve) , the hcp phase remains stable. Only a small amount of the sample (about 15%) has back-transformed to fcc.

[1] Giles P.M., Marder A.R., Metallurgical Transactions, May 1971,Vol.2,pp.1371-1378

[2] Birch 1986, J. Geophys. Res. 91 (B5), p.4949

[3] Ross M., Mao H.K. et al., 1986, J. Chem. Phys. 85 (2), p.1028