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|   | <b>Experiment title:</b><br>Reactivity of xenon with metals under ultra-high pressure | <b>Experiment number:</b><br>CH-4280 |
| <b>Beamline:</b><br>ID27  | <b>Date of experiment:</b><br>from: 5/11/2014 to: 8/11/2014                           | <b>Date of report:</b><br>20/07/2015 |
| <b>Shifts:</b><br>9   | <b>Local contact(s):</b> M. Mezouar   | <i>Received at ESRF:</i>             |
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## Report:

The aim of the experiment was to observe experimentally possible reactions between xenon and transition metals in the 100-200 GPa range. Such reactions have been predicted by density-functional theory based structure search methods [1], for Xe-Fe and Xe-Ni systems. We have thus focused on these two systems. Six runs have been performed (see Table 1). For each run, a metallic foil of either Fe or Ni has been loaded with Xenon as pressure transmitting medium. The foil was insulated from the diamond anvil with a thin KCl layer.

| Name  | culet diameter (μm) | Heating P (GPa) | Reaction P (GPa) | Recyst. Xe | Recryst. Ni/Fe |
|-------|---------------------|-----------------|------------------|------------|----------------|
| XeNi1 | 100                 | 121-156         | none             | Y          | Y              |
| XeNi2 | 70                  | 153-185         | 153              | Y          | Y              |
| XeNi3 | 100                 | 150-160         | 150              | Y          | Y              |
| XeFe1 | 70                  | 180             | none             | N          | N              |
| XeFe2 | 70                  | 158-200         | none             | N          | ~N             |
| XeFe3 | 70                  | 180             | ?                | ?          | ?              |

**Table 1:** Conditions of the six experimental runs.

For each run, the sample has been compressed to a pressure slightly lower than the predicted reaction pressure (around 150 GPa for Xe and Ni, and 190 GPa for Xe and Fe [1]). It has been laser heated and if no reaction was detected, the pressure was increased by ~5-10 GPa steps and laser heated. The pressure was measured using the equations of states of the reactants [2,3,4].

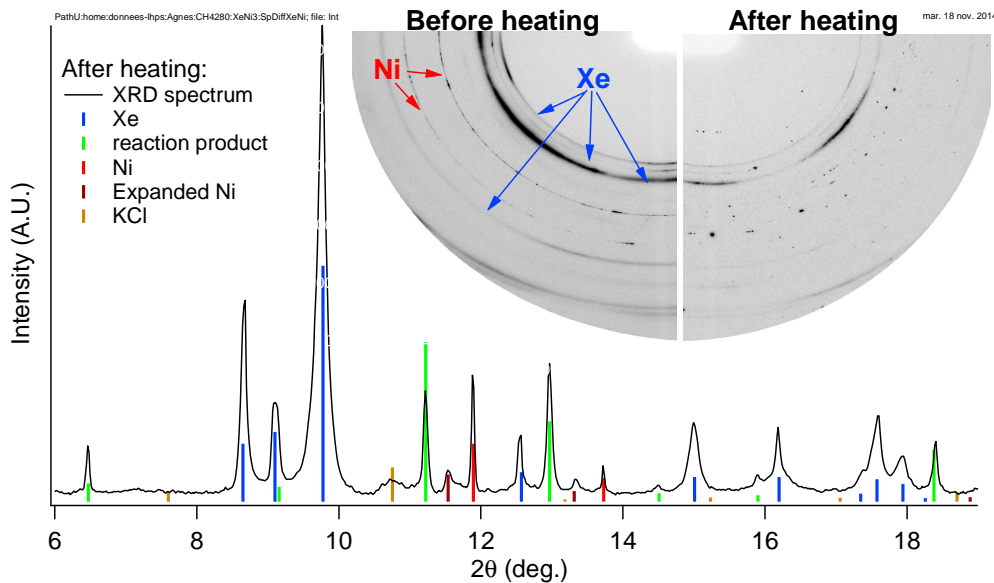
The properties of xenon change drastically in the studied pressure range. It becomes opaque in the visible range above ~150 GPa, due to its metallization[5,3].

The temperature has not been measured during heating; visual observation of the samples during laser-heating suggests that the surface temperature was higher than 3000K.

In the case of Xe-Ni, we have detected a reaction above ~152 GPa in two runs. Surprisingly, the reaction has not been observed in the first run, even though the pressure reached 156 GPa. The XRD spectra collected before and after heating for sample XeNi3 are presented in Figure 1. The reaction product is different from the one predicted in Ref. [1]. This product is stable up to 185 GPa at least. We have measured its equation of state on pressure decrease down to 100 GPa.

In the case of Xe-Fe, we have laser-heated the samples up to 200 GPa without observing any reaction. The reaction was predicted around 190 GPa. It is possible that it actually happens at a pressure higher than the pressure that we have reached. This might also be due to unefficient heating. In fact, we did not detect any high-temperature recrystallization of Xe or Fe in the Fe-Xe experiments. If the hot working temperature of iron is assumed to be 60% of its melting temperature (between 4500 K and 5000 K in the scanned pressure range), it means that we have not reached 3000K in bulk Fe in runs XeFe1 and XeFe2. The low heating efficiency might be due to the change of optical and transport properties in xenon due to its metallization. In run XeFe3, the anvils broke during laser-heating.

We wish to repeat these experiments with different sample conditionings which will allow a better thermal insulation of the samples.



**Fig. 1:** Integrated X-ray diffraction spectrum recorded in run XeNi3 at 152 GPa after laser-heating. The tick marks correspond to X-ray diffraction angles for xenon (in blue), nickel (in red), KCl (in yellow) and reaction products (in dark red and green).

#### References:

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