ESRF	**Experiment title:** High-Pressure synthesis of ReC_x by laser heating in the DAC	**Experiment number**: HS-2776
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Report:

Among the metallic elements, rhenium has one of the highest known bulk modulus, shear modulus, and melting point. Rhenium does not undergo a temperature-induced phase transition up to melting and remains stable to pressures of at least 216 GPa [1]. These properties make rhenium one of the strongest and most stable polycrystalline materials, suitable for applications such as gasket material in diamond anvil cells (DACs).

At high pressures the formation of rhenium carbides has been reported. Popova *et al.* stated the synthesis of an hexagonal ReC with MoC-type structure, lattice parameter a = 2.8403 Å and c = 9.8543 Å, and synthesis conditions above 6 GPa and 1073 K [2]; and the synthesis of a cubic polymorph with NaCl-type structure, a lattice parameter of 4.005 Å, and synthesis conditions above 17 GPa and 1300 K [3]. However, all these observations were performed on quenched samples using X-ray diffraction.

To investigate the reaction of rhenium with carbon, a rhenium foil (thickness ~20 μm) and powder graphite were laser heated in a diamond anvil cell. The starting materials were compressed in a DAC using a rhenium gasket (110 μm hole). Argon was used as a pressure-transmitting medium and pressure was determined using its equation of state measured at ambient temperature [4]. The uncertainities in pressure at high temperature were determined as $\Delta P = \pm 4$ GPa [5]. The starting materials were laser heated from one side at different pressures by a YAG laser. The temperature was measured at the center of the hot spot by analyzing the pyrometric signal; the uncertainities in measured temperatures fluctuated between 100-300 K [6]. Synchrotron X-ray radiation at a wavelength of 0.3738 Å and a MAR CCD detector were used. The images were processed and integrated with FIT2D [7]. LeBail refinements were carried out with Fullprof [8].

Diffraction data were collected at pressures of 4, 18-20 and 56-67 GPa prior to heating, during heating between 1800 and 3800 K and on temperature quenched samples. For each temperature run a new sample position was selected. From the data collected at around 4 GPa

the formation of a ReC_x phase was not detected up to the maximum temperature reached. However in the data collected at around 18-20 GPa, a hexagonal phase appeared at around 2260 K and it is stable up to the maximum temperature reached. At pressures around 56-67 GPa the hexagonal phase appeared at around 2800 K and, in contrast to what was observed at 18-20 GPa, rhenium reflections were observed up to the maximum temperature reached (Figure 1 *left*).

Main Results

A preliminary P-T rhenium-carbon phase diagram is shown in Figure 1 *right*. The hexagonal ReC_x is the stable phase at high-(P,T) conditions and no signs of a cubic polymorph or another phase were observed in all the data sets. The data collected here in conjunction with data from complementary multi-anvil cell experiments and from density functional theory calculations have now allowed us to (a) determine the structure of the rhenium carbide and (b) derive structure – property relations. Clearly the absence of data at high temperatures and pressures between 20-40 GPa limits our ability to derive a complete phase diagram. Beam time for measurements in this P,T-range will now be requested.

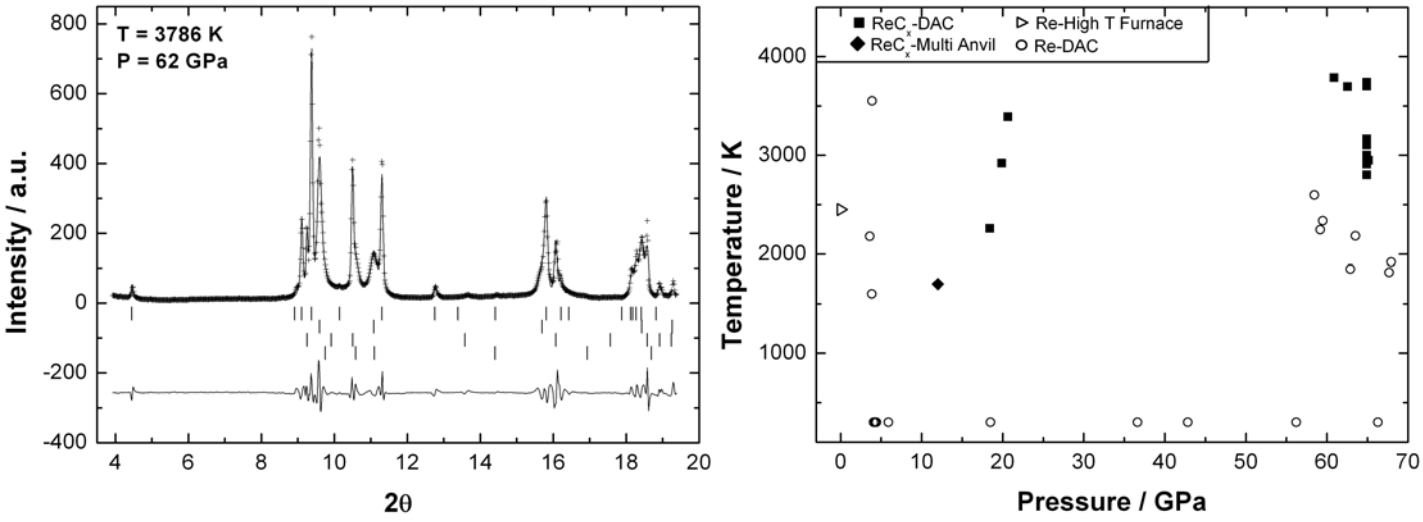

Figure 1. **Left**: X-ray diffraction pattern from the sample synthesized in a laser heated DAC. Observed (+), calculated (-) and the difference (at the bottom). Vertical marks correspond to the position of the allowed Bragg reflections of ReC_x, fcc-argon, hcp-argon and rhenium, from top respectively. **Right**: Preliminary P-T rhenium-carbon phase diagram.

References

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