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High-Pressure Synthesis

Novel Rhenium Carbides at 200 GPa**

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Abstract: Laser heating of rhenium in a diamond anvil cell to 3000 ± 300 K at about 200 GPa results in formation of two previously unknown rhenium carbides, hexagonal WC-type structured ReC and orthorhombic $TiSi_2$-type structured ReC_2. The shortest C–C distances [1.758(3) Å at 219(5) GPa and 1.850(4) Å at 180(7) GPa] found in honeycomb-like carbon nets in the structure of ReC_2 are quite unusual. The Re–C solid solution formed at multimegabar pressure has the carbon content of ≈ 20 at%.

Chemical compounds of 5d transition metals and carbon or other first-row elements, as B and N, often possess interesting properties attributed to strong covalent bonding.[1] Many of carbides, borides, and nitrides reveal very high melting points (for example, over 3500 K for ZrC, NbC, HfC, TaC),[2] large bulk moduli ($K_0 > 390$ GPa for Re_2C,[3] Re_2N,[4] ReN_2,[5] IrN_2,[6] OsB,[7] Os_2B_3,[7] OsB_2[7]), and very high hardness ($H_v > 35$ GPa for ReN_2,[5] WB_4[7]). Transition-metal carbides belong to a large group of industrially important materials.

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The rhenium-carbon system provides a striking example of the pressure effect on elements reactivity and the binary phase diagram. At ambient pressure, rhenium does not form stoichiometric carbides; carbon dissolves into rhenium up to 28.45 at% at the eutectic temperature (2778 K).[8] However, even very moderate pressure, just above 6 GPa (and high temperatures) was reported to promote formation of a Re–C compound.[9,10] Its correct chemical composition (Re_2C) and crystal structure of anti-MoS_2 type (hexagonal primitive, hP, space group $P6_3/mmc$) were established relatively recently on the basis of X-ray powder diffraction, Raman spectroscopy data and DFT calculations.[11,12] No other stoichiometric carbides apart of hP-Re_2C have been observed at pressures up to ≈ 70 GPa and temperatures ≈ 4000 K.[3]

The hP-Re_2C was found to be isostructural with Re_2N.[11] This analogy and the recently observed very complex and unexpected behavior of the Re–N system, featuring numerous nitrogen-rich compounds,[5,13] stimulated the study of potential reactions between Re and C at multimegabar pressures. Awareness of these reactions is also of a primary interest for the development of the methodology of ultra-high pressure high temperature experiments, in which Re gaskets are commonly used. The range of currently achievable static pressures has been extended to ≈ 1000 GPa due to implementation of double-stage diamond anvil cells (dsDAC) and to ≈ 600 GPa with toroidal type anvils (tDAC).[14–17] In order to achieve such extreme pressures, the linear size of samples and sample chambers should be drastically decreased. At pressures above ≈ 150 GPa, a pressure chamber's diameter (made, as a rule, of Re) is usually smaller than 50 μm, and in dsDACs above 300 GPa it is less than 10 μm. Meanwhile, the size of a laser beam in typical laser heating (LH) setups used in DAC experiments varies from 15 to 50 μm at FWHM.[18,19] As a result, irradiation of, at least the edge of a Re-gasket, by the laser beam during laser heating becomes unavoidable and may lead to a chemical reaction between Re and carbon of the diamond anvils. Therefore, correct interpretation of the results of experiments with laser-heating

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at ultra-high static pressures requires knowledge about possible products of rhenium and carbon interaction.

Here, we report on the in situ study of Re–C compounds formed due to chemical interactions between diamond anvils and the rhenium gasket after pulsed laser heating in DACs at about 200 GPa. The structures of all of the synthesized rhenium carbides, Re_2C, ReC_2, ReC, and $ReC_{0.2}$ were solved and refined using single-crystal X-ray diffraction (SCXRD) providing direct and unequivocal data to judge on both the atomic arrangement and chemical composition of the crystalline matter.

Two DACs have been prepared, dsDAC for Experiment #1 and conventional assembly for Experiment #2 (for details see Supporting Information, DAC preparation). Secondary anvils for Experiment #1 were made from nano-crystalline diamond (NCD) spheres of about 15–20 μm in diameter. The dsDAC in Experiment #1 was compressed up to ≈ 415 GPa (according to the diamond Raman shift on the secondary anvil),[20] then the sample (Re flakes) was laser heated until the first bright flash of light that held for less than a second (temperature was not measured). After the first heating attempt pressure dropped down to ≈ 200 GPa probably due to failure of the secondary anvils. However, the DAC remained intact and rhenium in the central area was again carefully laser heated in a pulsed mode (1 μs pulses, 25 kHz repetition rate, and maximum temperature of 3000 ± 300 K).[21] Pressure did not change upon the repeated laser heating (Supporting Information, Figure S1) and the DAC with the temperature-quenched material was investigated using powder and single-crystal X-ray diffraction (for details see the Supporting Information). A 2D diffraction map collected across the sample chamber revealed the presence of not only Re, but four additional phases (Figure 1). All of them have been identified and are described in detail below.
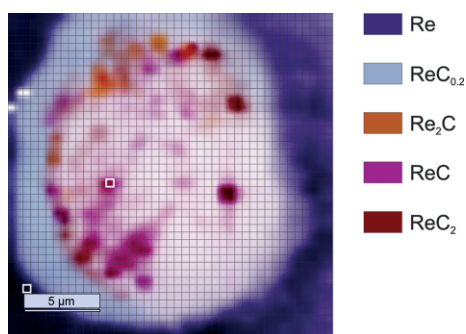

Figure 1. 2D map of X-ray diffraction patterns. The color intensity is proportional to the intensity of the following reflections: the (100) and (101) reflections of the Re gasket for the violet region; the (100) and (101) reflections of $ReC_{0.2}$ for the light blueish-gray region; the (105) reflection of Re_2C for the orange region; the (101) reflection of ReC with the WC-type structure for the purple region; the (002), (111), (020) reflections of ReC_2 for the dark red region. Characteristic diffraction images highlighted with white rectangles are shown in Figure S2.

The XRD patterns collected from the edge of the Re-gasket show typical continuous powder diffraction rings of rhenium (Supporting Information, Figure S2a) not exposed to the laser beam. The unit cell volume of Re determined from the Le Bail fit varies from 22.000(5) Å^3 to 21.978(7) Å^3 for different patterns

(Supporting Information, Figure S3). This corresponds to pressures of ≈ 230–240 GPa according to equation of state (EOS) from Dubrovinsky et al.,[14] or ≈ 190–195 GPa, according to Anzellini et al.[22] (Supporting Information, Table S1).

One of the crystalline phases we found within the laser-heated area is the hexagonal hP-Re_2C ($P6_3/mmc$, #194) (Figure 2a).[11,12,23] The parameters of the unit cell were found to be a = 2.5860(9) Å, c = 9.272(3) Å, V = 53.70(3) Å^3 (Supporting Information, Table S2, S3, Figure S4a). As written above, in this experiment the pressure, as determined from the Raman shift of the diamond anvil (Supporting Information, Figure S1), was of about 200 GPa, whereas the Re EOS gave us to 240 GPa. These values do not match the pressure of 172(13) GPa, determined for the given unit cell volume of hP-Re_2C according to the EOS reported by Juarez-Arellano et al.[3] The EOS of Re_2C in ref.[3] was determined on the basis of powder XRD from a sample in hard pressure transmitting medium that led to a significant uncertainty in the bulk modulus: K = 405(30) GPa (K' = 4.6).[3] Comparison of our data with literature motivated us to make an independent measurement to establish the EOS of hP-Re_2C on the basis of SCXRD. In the experiment described in Supporting Information, Experimental Procedures, the $P–V$ data were obtained up to 50 GPa from a single crystal of hP-Re_2C pressurized in a soft (Ne) pressure transmitting medium (Supporting Information, Table S11, Figure S5). The parameters of the 3rd order Birch–Murnaghan equation of state of hP-Re_2C [V_0 = 69.18(4) Å^3/unit cell, K = 375(15) GPa, K' = 5.0(1)] we obtained only slightly (within uncertainties) differ from the values reported by Juarez-Arellano et al.[3] According to our EOS hP-Re_2C synthesized in Experiment #1 was under the pressure of 180(7) GPa (Figure 3a). The structure of hP-Re_2C (Figure 2a, Supporting Information, Table S3) determined from SCXRD is in a good agreement with model described by Friedrich et al.[11] It is characterized by the stacking sequence AABB of layers of Re atoms (with four Re atoms per a unit cell). Carbon atoms occupy the 2d Wyckoff position in trigonal prisms formed by Re atoms with the Re–C distances of 1.997(1) Å (Figure 2e, Supporting Information, Table S4).

The structure of another phase, identified as ReC, has a hexagonal unit cell with the lattice parameters a = 2.5510(9) Å, c = 2.7048(11) Å, and V = 15.24(1) Å^3 (Figure 1b) (Supporting Information, Table S2, S5, Figure S4b). The structure solution and refinement revealed that hP-ReC belongs to the WC structure type ($P\bar{6}m2$, #187) with characteristic c/a ratio (≈ 0.94). Like in Re_2C, carbon atoms are located in trigonal prisms formed by Re atoms with the Re–C distances equal to 2.000(2) Å (Figure 2f, Supporting Information, Table S6).

One more carbide, ReC_2, with the orthorhombic structure found in Experiment #1 (Figure 2c, Supporting Information, Table S2, S7, Figure S4c) has the lattice parameters a = 3.3367(10) Å, b = 4.3155(16) Å, c = 5.6220(13) Å, and V = 80.95(4) Å^3 at 180(7) GPa, and a space group $Fmmm$ (#69). Remarkably, the same orthorhombic phase [a = 3.2880(9) Å, b = 4.2088(9) Å, c = 5.5645(8) Å, and V= 77.00(3) Å^3] was found in Experiment #2, in which iron oxide (FeO) in a Ne pressure medium was compressed to 219(5) GPa and pulsed-laser heated up to 2500 ± 300 K.[19] The relatively large beam (of about

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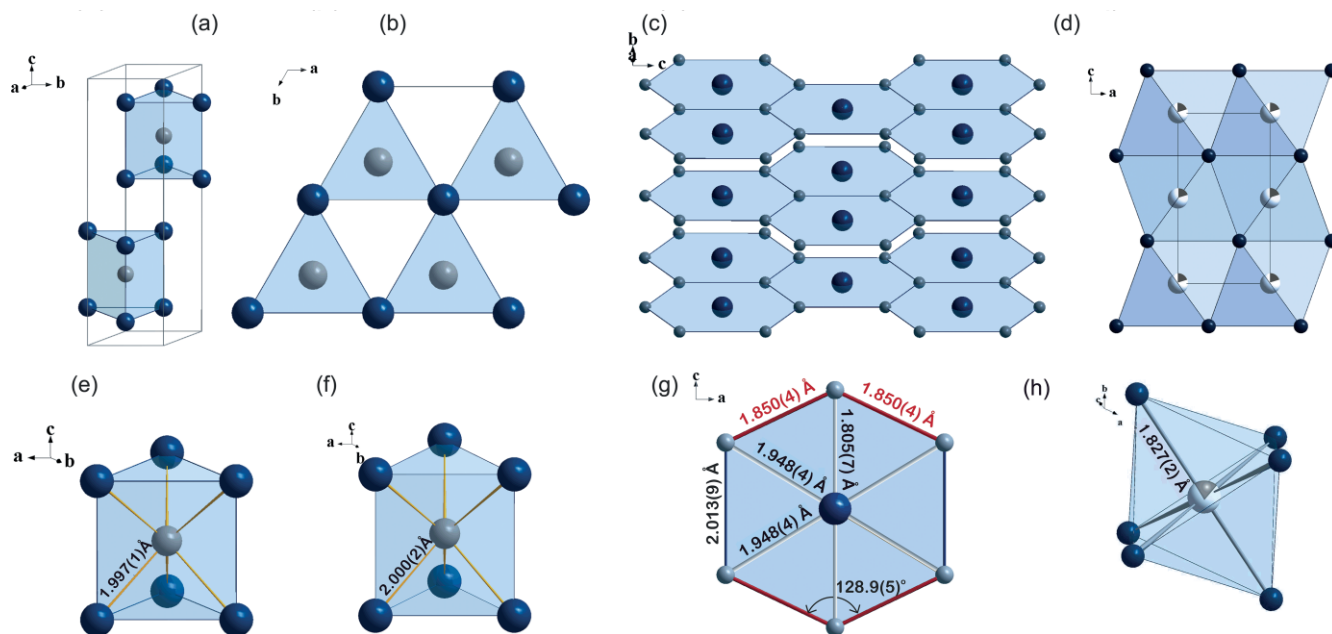European Journal of Inorganic Chemistry

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Figure 2. Crystal structures of Re–C compounds observed in Experiment #1 at 180(7) GPa. (a) hP-Re$_2$C, (b) WC-type structured ReC, (c) TiSi$_2$-type structured oF-ReC$_2$; and (d) B8-type structured interstitial solid solution ReC$_{0.2}$. Dark blue balls designate Re atoms; dark gray balls – carbon atoms; white balls with a dark gray sector symbolize a partial occupancy of the position by carbon atoms. Carbon coordination polyhedra in the crystal structures of Re$_2$C (e) and ReC (f) are strikingly similar with the Re–C bond length ≈ 2 Å (shown by yellow color). (g) A single hexagon in the honeycomb-like web of carbon atoms, which coordinate each Re atom in the structure of ReC$_2$. The shortest C–C distances of 1.850(4) Å are highlighted by red color. [h] A single octahedron formed by Re atoms in the structure of ReC$_{0.2}$, which incorporates carbon atoms with the occupancy of 20 %. The Re–C distances are relatively short: 1.827(2) Å.
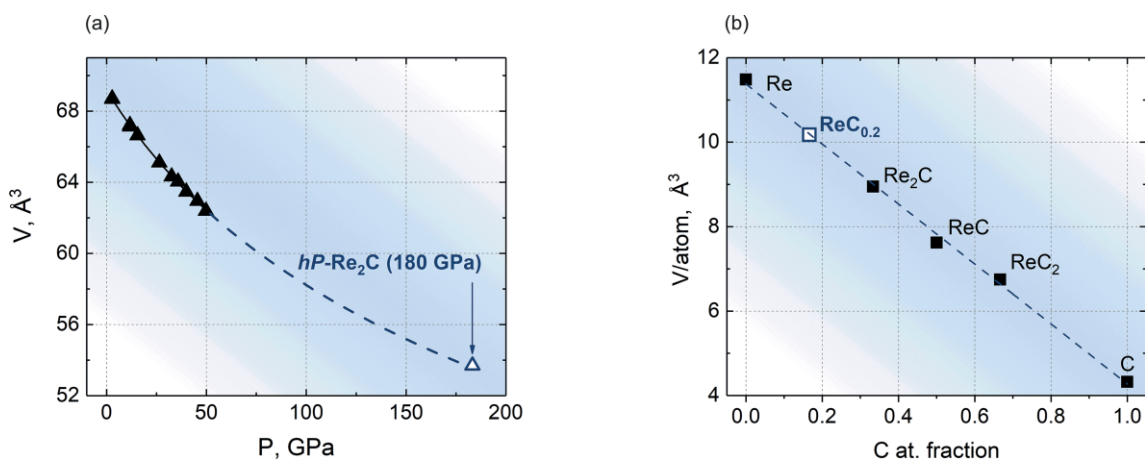

Figure 3. The unit cell volume of hP-Re$_2$C as a function of pressure (a) and the dependence of the unit cell volumes per atom on the atomic fraction of carbon for various Re$_x$C$_y$ compounds at 180(7) GPa (b). Black triangles are experimental data points obtained using SCXRD on compression of hP-Re$_2$C up to ≈ 50 GPa in a soft (Ne) pressure transmitting medium. Solid black line is a fit of the P-V experimental data using the 3rd order Birch–Murnaghan EOS [V_0 = 69.18(4) Å^3/unit cell, K = 375(15) GPa, K' = 5.0(1)] and the dashed line is its extrapolation. Open triangle [corresponds to the volume obtained for hP-Re$_2$C in one pressure point in Experiment #1 (see text)]. Black squares are experimental data points for observed Re–C compounds; open square designates ReC$_{0.2}$, in which the atomic fraction of carbon was determined in accordance with this empiric "V/atom vs. C at. fraction" relationship.

25 μm at FWHM) irradiated the Re gasket used in Experiment #2 and ReC$_2$ was found at the border of the pressure chamber.

The structure solution and refinement of the orthorhombic phase, in both Experiment #1 and #2, resulted in the chemical composition ReC$_2$. The structure shown in Figure 2c can be described as stacking of flat honeycomb-like nets of carbon atoms along the b direction with a translation (¹/₂, 0, ¹/₂). The "honeycomb" hexagons are not ideal (Figure 2g), they have two longer and four shorter sides (Supporting Information, Table S8). Rhe-

nium atoms are located in the center of each of carbon hexagons (Figure 2g). Thus, in the same ac-plane each Re has 6 closest carbon neighbors, four of which are at a distance of 1.948(4) Å and two at 1.805(7) Å, as determined at 180(7) GPa (Supporting Information, Table S8). Note that whereas the description in terms of stacking of the Re–C nets obviously helpful to give a clear geometrical presentation of the atomic arrangement in oF-ReC$_2$, the structure is not layered, as the shortest Re–Re distances between the "layers" (≈ 2.72 Å) are similar to

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those in non-layered structures of hP-Re_2C ($\approx$ 2.5–2.6 Å) and hP-ReC ($\approx$ 2.55–2.70 Å) at the same pressure.

It is worth noticing that in oF-ReC_2 the shortest C–C distances [1.758(3) Å at 219(5) GPa and 1.850(4) Å at 180(7) GPa] are up to 30 %, or 20 % larger than in sp^2- ($\approx$ 1.42 Å in graphite[24]) or sp^3- ($\approx$ 1.54 Å in diamond[25]) bonded carbon, correspondingly. This may suggest the absence of the chemical bonding between carbon atoms, but still, the latter should be proven by theoretical computations, which are out of scope of the present study. Some crystal-chemical analogy can be considered also, but it does not allow to make any definite conclusion regarding the chemical bonding in this particular case. Indeed, oF-ReC_2 is isostructural to one of the polymorphs of titanium disilicide (TiSi$_2$) (space group $Fmmm$, #69), (a = 4.428 Å, b = 4.779 Å, c = 9.078 Å, V = 192.1 Å^3).[26] The shortest Si–Si distance in oF-TiSi$_2$ at ambient conditions is $\approx$ 2.57 Å that is ca. 9 % larger than in diamond-structured Si ($\approx$ 2.35 Å).[27] Any direct analogy is difficult to draw, as there is no data on chemical bonding in oF-TiSi$_2$. Although ab initio simulations for one other polymorph of oF-TiSi$_2$ and a Zintl phase CaSi suggest a significant covalent interaction between Si atoms in zig-zag chains geometrically similar to the C–C chains in our oF-ReC_2 carbide, their Si–Si distances, of about 2.53 Å and 2.54 Å, correspondingly, are of only 8 % larger than those in diamond-structured Si.[28,29]

Remarkably, there is a strong diffuse scattering, especially evident in the reciprocal space, suggesting a disorder along the b-axis in the structure of oF-ReC_2 (Supporting Information, Figure S6a). Those defects might help to stabilize the structure. In fact, the titanium disilicide oF-TiSi$_2$,[26] which is isostructural to oF-ReC_2 (discussed above), is calculated to have the energy of about 0.3 eV/atom higher, than other titanium disilicide polymorphs.[26] However, further studies are required to elaborate this hypothesis.

In the phase map built on the basis of powder XRD data (Figure 1), the light blueish-gray region indicates the phase filling the outer rim of the pressure chamber at the border with the Re gasket. On the basis of only powder XRD, it could be interpreted exclusively as Re at about 100 GPa, according to the EOS of Anzellini et al.[22] or at about 115 GPa, according to the EOS of Dubrovinsky et al.[14] that would be inconsistent with the pressure of 180(7) GPa determined previously. In a few points of this region it was possible to collect single-crystal data sets (Supporting Information, Figure S4d). Indexing of the single-crystal data gave the same result as that of powder XRD data: a hexagonal unit cell with the lattice parameters a = 2.6028(12) Å, c = 4.161(2) Å, V= 24.41(2) Å^3, and a space group $P6_3/mmc$ (#194) (Supporting Information, Figure S4d, Table S2, S9). However, single-crystal data revealed the electron density, localized in the octahedral voids of the hcp-Re like in the B8 (NiAs)-type structure (Figure 2d) that suggests this phase to be a Re–C interstitial solid solution based on the B8-type structure (Figure 2d, h). Single-crystal diffraction data at hands are not sufficient to refine the site occupancies for carbon atoms, especially taking into account a huge difference in X-ray scattering factors of Re and C. Fortunately, we noticed that the unit cell volumes per atom for Re$_x$C$_y$ compounds [Re,[14] hP-Re$_2$C, hP-ReC, oF-ReC$_2$, and C (diamond)[30]] at 180(7) GPa, if plotted as a function

of carbon content, all appear along the common straight line (Figure 3b). Considering the volume of the B8 rhenium-carbon solid solution, we have estimated its composition as ReC$_{0.2}$.

A comparison of our data for Re–C compounds and recent reports on the Re–N system demonstrates obvious crystal-chemical similarities in the two systems for compositions Re/C ≥1. Two carbides, Re$_2$C and ReC, are isostructural to Re$_2$N and ReN. They are built up of CRe$_6$ or NRe$_6$ trigonal prisms, and even Re–C and Re–N interatomic distances at the same pressures are very similar.[4,5] Contrary, the structures of ReC$_2$ and ReN$_2$[5] have nothing in common and there are no signs of formation of rhenium polycarbides with more than two carbon atoms per a formula unit at least up to 180(7) GPa and 3000 ± 300 K, unlike to the rhenium-nitrogen system.[13] Such differences can be linked a tendency of nitrogen to form dinitrogen and poly-nitrogen anions (based on N4 units[13,31–33]) at high-pressures and high-temperatures (HPHT), whereas rhenium carbides do not reveal formation of polycarbon anions at such conditions.

Nevertheless, there is a number of experimental works and theoretical predictions that suggest progressive polymerization of carbon atoms at high pressures.[34–39] In all cases polymerization results in formation of short ($\approx$ 1.6 Å or shorter) C–C bonds for single bonded carbon atoms. For hexagonal carbides hP-Re$_2$C or hP-ReC at $\approx$ 200 GPa, the C–C distances are longer than 2.5 Å, which suggest the absence of chemical bonding between carbon atoms in the crystal structure. In case of rhenium dicarbide, oF-ReC$_2$, we observed formation of honeycomb-like carbon layers, in which the shortest C–C distances ($\approx$ 1.76–1.85 Å) are much longer than expected for sp^2- or sp^3-bonded carbon atoms in inorganic compounds; still, in alkanes the C–C bond length as long as 1.704 Å has been detected.[40]

To summarize, we have extended the knowledge about the chemical interaction between rhenium and carbon to $\approx$ 200 GPa. Two novel carbon-rich rhenium carbides – WC-type structured hP-ReC and TiSi$_2$-type structured oF-ReC$_2$ – were synthesized (hitherto only Re$_2$C was known in the Re–C system). The fact that rhenium and carbon can produce numerous compounds at HPHT conditions should be taken into account upon planning of experiments in LHDACs at multimegabar pressures.

CCDC 1976969 (for Re$_2$C), 1976970 (for ReC), 1976971 (for ReC$_{0.2}$), and 1976973 (for ReC$_2$) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from FIZ Karlsruhe.

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