	Experiment title: High Pressure Study of californium metal and americium/curium alloys	**Experiment number**: **HS-3016**
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Shifts:	**Local contact(s)**: **Mohamed MEZOUAR**	*Received at ESRF:*

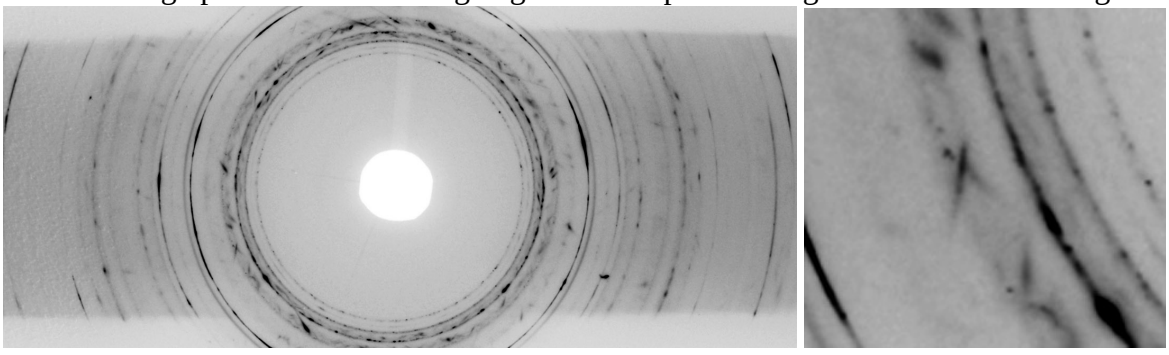
Names and affiliations of applicants (* indicates experimentalists):

Heathman S [*1],Heines P [*1], J.C. Griveau J C [*1], Yagoubi S [*1], Haire R G [2]

(1) European Commission JRC, Institute for Transuranium Elements, Postfach 2340, D-76125 Karlsruhe, Germany
(2) Oak Ridge National Laboratory, PO Box 2008, MS-6375, Oak Ridge, Tennessee 37831, USA

Report:

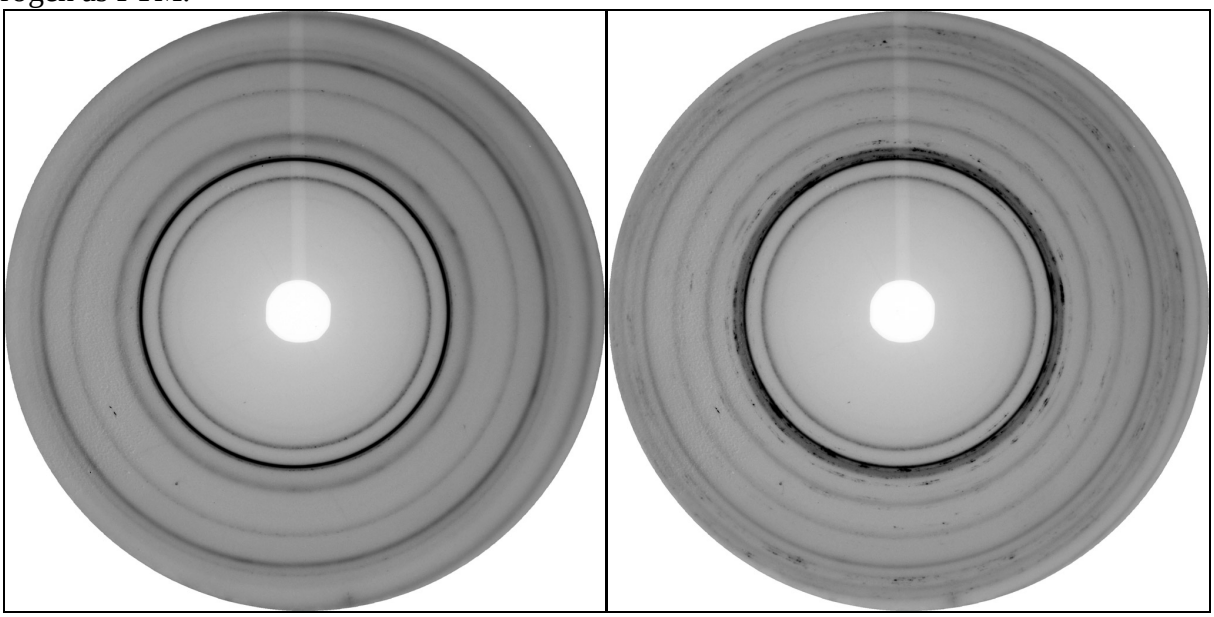
The purpose of this proposal was to complete high pressure x-ray diffraction experiments on californium metal (Cf-249) at the ID27 beamline. The cells prepared were 2 Cornell type and 2 Syassen/Holzapfel type. Diamonds with culets of 100 microns and steel T301 gaskets with 40-50 micron holes were used for the Cornells. For the other cells we used diamonds with 200 and 400 micron culets with Inconel gaskets drilled to 80 and 120 microns respectively. 1 microgram of californium metal (Cf-249) was loaded into each cell along with silicone oil or nitrogen as the pressure transmitting medium. 2 to 5 micron diameter ruby spheres or copper powder were loaded alongside the californium samples for pressure determination. Images of the diffraction spectra at 3 GPa below show an interesting diffraction effect representing a macroscopic cluster of crystal defects which arises from attraction among defects probably caused by radiation damage in the crystal lattice. At high pressure after undergoing structural phase changes this effect is no longer evident.



Cf at 3 GPa

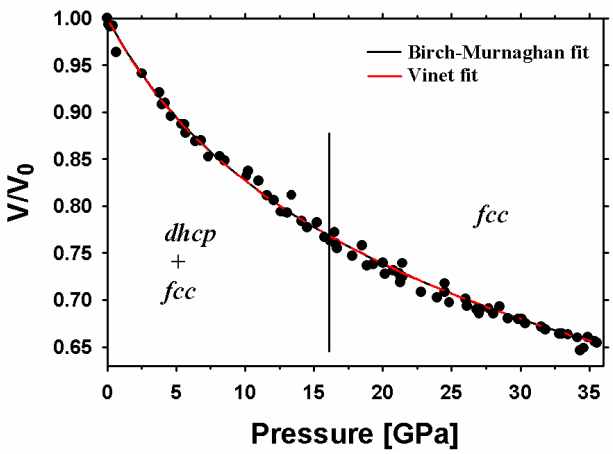
Structural phase transitions were observed with double hexagonal close packed and face centered cubic structures formed below 35 GPa. At between 35 and 40 GPa we observe a further transistion which represents a mixture of the two orthorhombic structures of the Am III and Am IV type.

The 2 images shown below at pressures of 37 GPa **(A)** and 45 GPa **(B)** were taken from Cf samples loaded with nitrogen as PTM.



A **B**

In addition to the obvious change of structure we also observe a re-crystallisation effect represented by the spotty lines in B arising from the large 10% volume collapse which occurs upon formation of the Am IV type (orthorhombic-pnma) phase as full de-localisation of the 5f electrons takes place.



Relative volume behaviour of Californium as a function of pressure.

The compressibility for Cf (shown above) was obtained by fitting the Birch - Murnaghan and Vinet equations of state to the low pressure phases (regions of localized *f* electrons) to obtain the bulk modulus B_0 and its pressure derivative B'_0. Both calculations gave similar values, which were 36.2(3) GPa for B_0 and 3.9(2) for B'_0 and 35.3(3) GPa for B_0 and 4.3(3) for B'_0, respectively. The theoretical bulk modulus was calculated to be 37.4 GPa, in excellent agreement with the experimental results. These californium moduli are in line with values for the pure Am-Cf metals, which range from 25 to 43 GPa but considerably smaller than the moduli of the Th - Pu metals, which have additional bonding from their itinerant *f* electrons and are less compressible.

We thank the staff of beamline ID27 and the safety group at the ESRF for their help during synchrotron experiments.