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Report:

We have performed angle-dispersive x-ray diffraction measurements on hydrostatically compressed polycrystalline hcp cobalt to 90 GPa. At 65-75 GPa we document an inversion in the pressure derivative of the axial ratio c/a with no discontinuity in the volume and lattice parameters compression curves. These data indicate previously unrecognized interactions amongst structure, electronic density at the Fermi level, magnetism and elasticity and are likely responsible for anomalies in the elastic and vibrational properties of hcp Co at high pressure.

The presence of the metastable fcc phase of cobalt is major hinderance to the accurate fitting of Co powder diffraction patterns [1]. Based on our previous experience with uniaxially compressed polycrystalline cobalt, where we observed a pure hcp phase after a non-hydrostatic compression to less than 2 GPa, in the present experiments, we obtained a pure hcp sample - despite the natural mixture of the two phases at room conditions - by loading the diamond anvil cell (DAC) with a previously non-hydrostatically compressed powder. Specifically,99.999\% purity cobalt powder from MV Laboratories was non-hydrostatically compressed into a DAC to 5 GPa and then quenched in liquid nitrogen. The recovered powder was loaded, using neon as pressure transmitting medium, into a 90 μm diameter hole drilled into a rhenium gasket pre-indented to a thickness of 28 μm (initially 200 μm), together with some platinum and a ruby chip for pressure determination. We employed two membrane type DACs, both equipped with beveled diamond anvils (150 μm flat beveled from 300 μm culet at 8°.

We used a monochromatic beam (λ=0.4121 Å) focused down to less than 15x15 μm FWHM and a MAR345 image plate detector. Sample to detector distance, detector tilt and pixel size ratios were calibrated using a Si standard and FIT2D software. More than 50 diffraction pattern where collected in the 0-90 GPa pressure range, throughout the entire stability field of the hcp phase. Particular care was taken to stabilize the pressure and to minimize the drift during the measurements. Pressure was measured using both the Pt EOS and by ruby luminescence, before and after each collection of the Co diffraction, and the observed pressure drifts were well below 0.5 Kbar (typically 0.1-0.2 Kbar). The cobalt diffraction patterns do not show any contamination from scattering of the pressure markers or the gasket at any pressure. Rietveld structural refinements were performed using GSAS. The obtained compression curve is reported in Fig. 1, together with the weighted third-order Birch-Murnaghan fit to the experimental data, which yields K_0=203±4 GPa and K'=3.7±0.2, in good agreement with previous determinations (K_0=199±6 GPa, K'=3.6±0.2) [1,2].

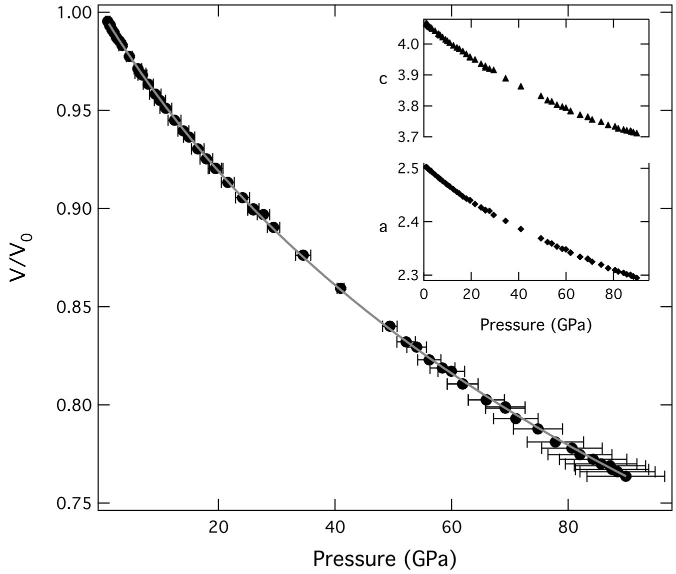

Fig. 1: Isothermal compression curve of hcp Co at ambient temperature. The indetermination on the volume are smaller than the symbols; the errors on pressure include the indetermination arising from the difference amongst the ruby determination and two different Pt EOS, together with an estimation of pressure gradient within the cell. The solid curve is a third-order Birch-Murnaghan fit to the experimental data, yielding K_0=203±4 GPa and K'=3.7±0.2.
Inset: pressure evolution of the lattice parameters a and c (values in Å).

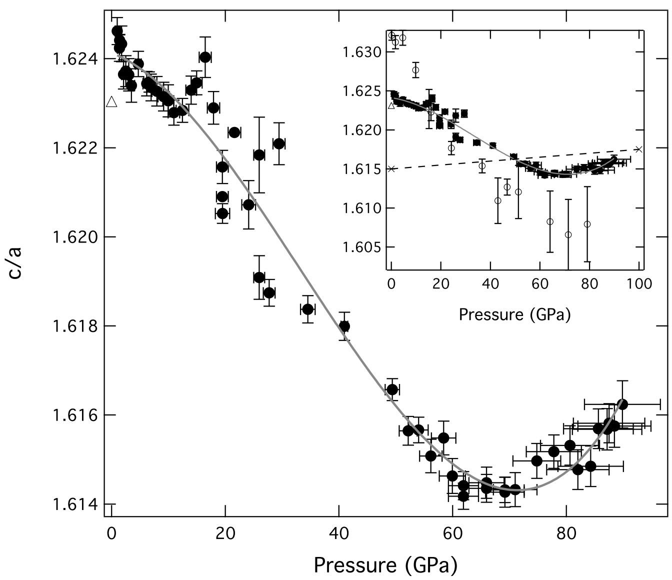

Fig. 2: Pressure evolution of the axial ratio c/a. The solid line is a simple polynomial fit to the experimental data, as a guide for the eyes. The values in the 18-35 GPa pressure region display increased scatter and have larger error bars due to a partial overlapping of Ne and Co diffraction lines. The open triangle shows the axial ratio at ambient pressure from literature.
Inset: comparison of previous high-pressure works with this study. Open symbols are data from [1]; the data from [2] have a variance that does not allow them to be plotted at this scale. The dashed line is from ab initio calculations [3].

However, we observe a markedly different behavior in the axial ratio c/a from what was suggested by previous experimental work on foils [2] and *ab initio* calculations [3,4]. Our new data display a monotonic decrease in the axial ratio with pressure up to 65-70 GPa, where the slope of c/a *vs*. pressure becomes flat, and then, above 75 GPa, c/a increases (Fig. 2). A continuous decrease of the axial ratio with pressure was previously observed in diffraction experiments on powders [1], but the limited investigated pressure range (up to 78 GPa) and the larger error bars, likely prevented the detection of the change in the slope.

References

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