

Supplementary Information

Mechanochemical Synthesis of Cr_3C_2 : Investigating the Role of Pressure and Temperature

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Thermal treatment experiments

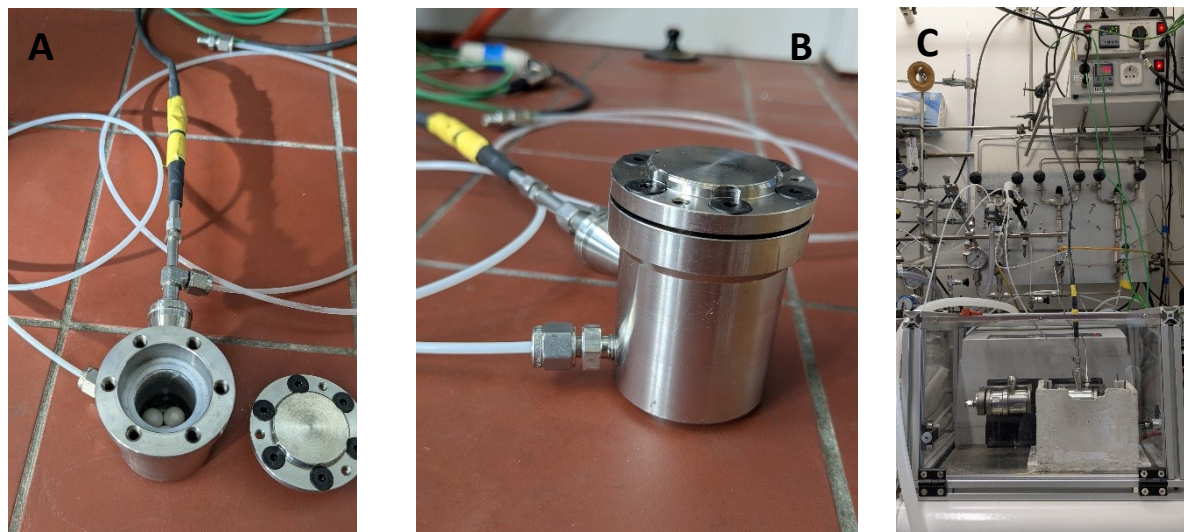


Figure S1: (a) Top view of the custom gas-flow shaker mill jar, showing the zirconia inlet fitting. The two white polyamide tubes are used for controlled gas inflow and outflow, while the black wire is connected to a temperature controller for real-time monitoring. (b) The fully assembled stainless-steel jar, tightly sealed and ready for milling, with all connections in place. (c) Experimental setup depicting the milling jar mounted in the MM400 shaker mill (Retsch), including attached gas lines, a temperature controller, and the heating jacket installed beneath the jar for thermal treatment during milling.

Pressure-induced experiments



Figure S2: (a) The inlet area of custom built pressurisable stainless-steel milling (b) Top view of disassembled setup including the inner jar and the outer jacket. (c) Fully assembled and sealed stainless-steel pressure jar. (d) Experimental setup displaying the pressurized milling jar connected to the external pressure line and gauge system, used to purge the gas pressure into the jar.

Specific surface area of the samples determined by BET analysis

Sample ID	SBET [m ² g ⁻¹]
S1	27.83
S2	28.65
S3	29.75
S4	31.20
S5	29.70
S6	31.68
S7	33.52
S8	28.15
S9	30.89
S10	27.88
S11	26.52
S12	28.96
S13	28.65
S15	33.12
S16	26.41
S17	4.12

Table S1: The BET surface area values of the mechanochemically synthesized Cr₃C₂ samples.

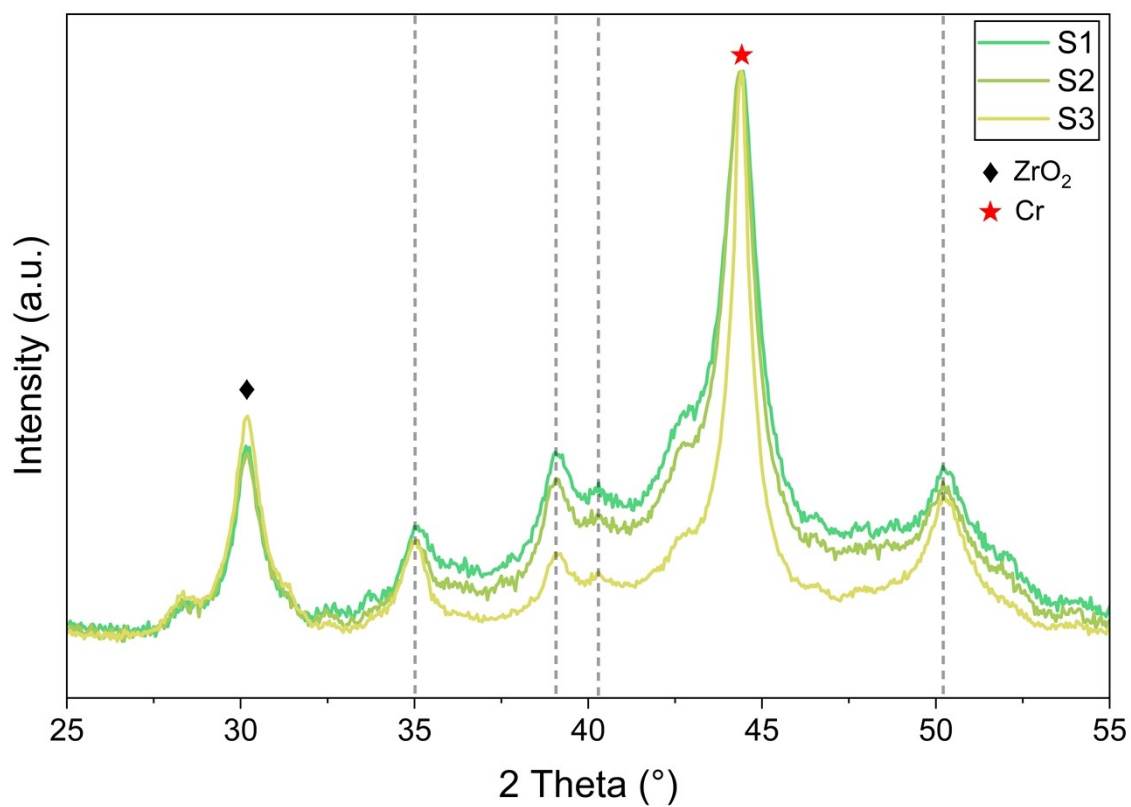


Figure S3: X-ray diffraction patterns of powders S1 to S3 milled at 6h under room temperature, 100°C, and 175 °C respectively. The reference diffraction peaks of the Cr₃C₂ phase (PDF 00-035-0804) are marked by dashed lines, unreacted chromium is indicated by red stars, and zirconia impurities originating from the milling media are marked by black diamonds.

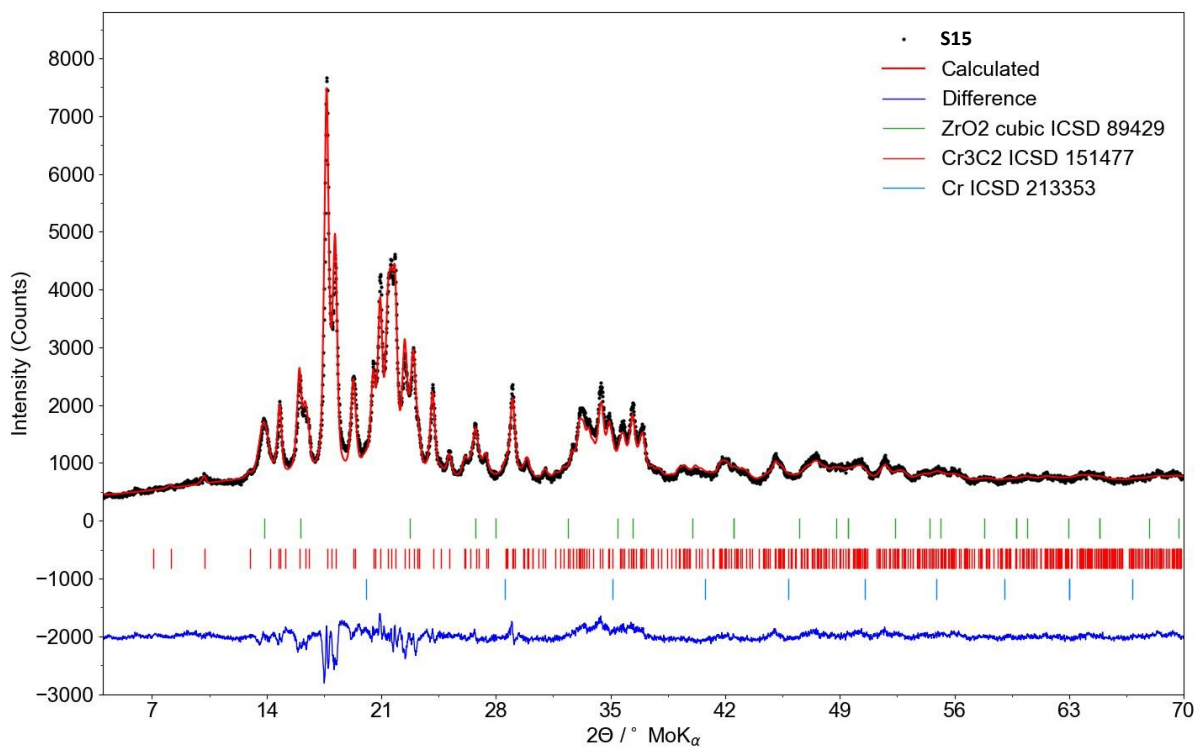


Figure S4: Rietveld-refined powder X-ray diffraction patterns of powder S15 milled at 48h under room temperature under 20 bar pressure.

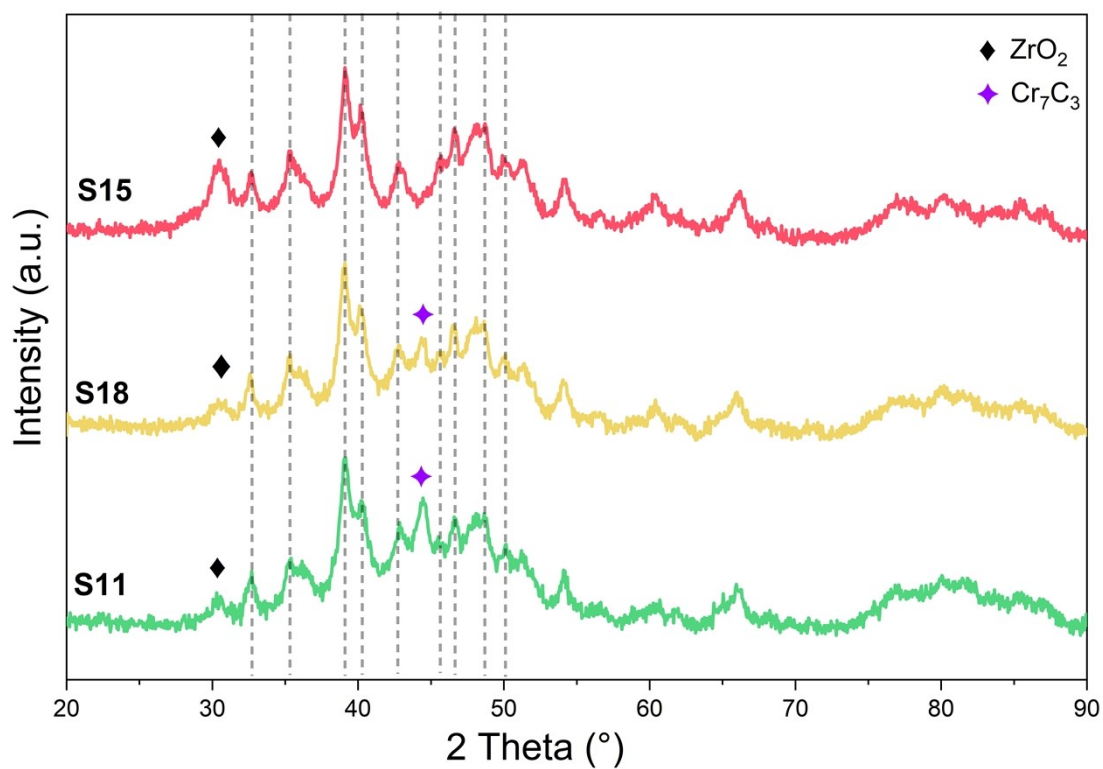


Figure S5: X-ray diffraction patterns of samples S11, S15, and S18 obtained after planetary milling at 20 bar for 32 h, 48 h, and 40 h, respectively. The referenced diffraction peaks of the Cr_3C_2 phase (PDF 00-035-0804) are marked by grey dashed lines, while those corresponding to the Cr_7C_3 phase (PDF 00-036-1482) are indicated by purple symbols.