

Rapid Microwave Synthesis and Characterisation of the Layered Carbide, Al_4C_3 .

Jennifer L. Kennedy,^{a,b} Timothy D. Drysdale^{*a} and Duncan H. Gregory^{*b}

^a School of Engineering, Rankine Building, University of Glasgow, Glasgow G12 8QQ; E-mail: Timothy.Drysdale@Glasgow.ac.uk.

^b WestCHEM, School of Chemistry, Joseph Black Building, University of Glasgow, Glasgow G12 8QQ; E-mail: Duncan.Gregory@Glasgow.ac.uk.

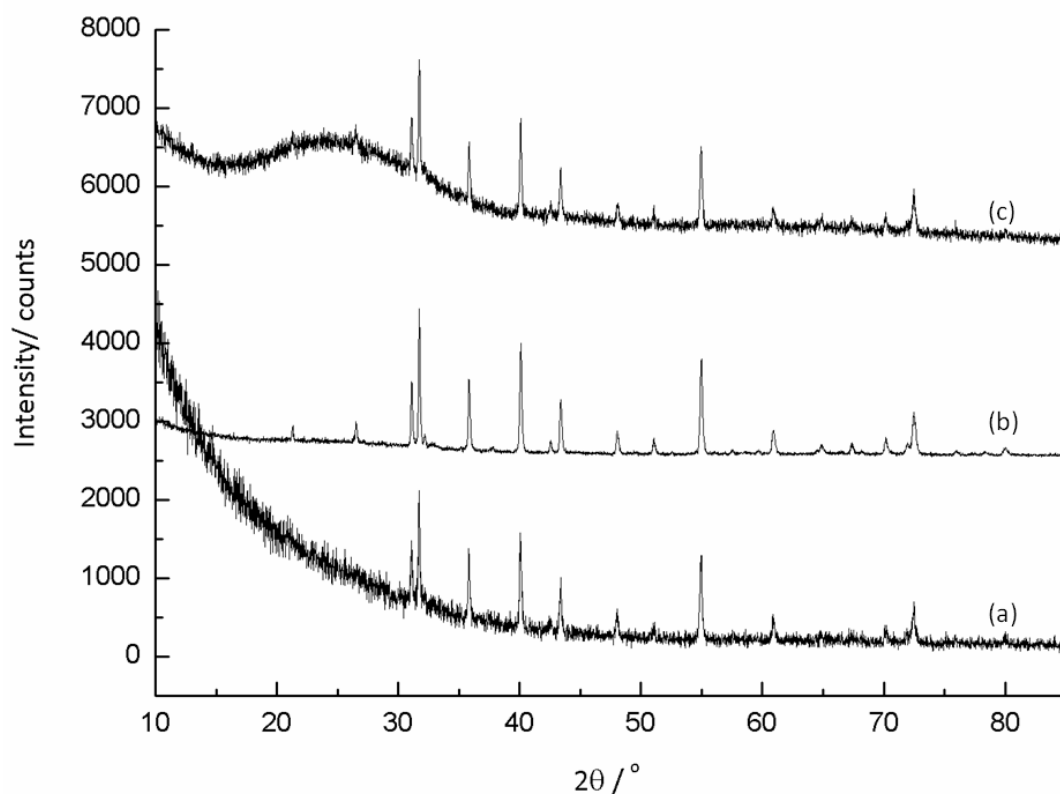


Fig. S1 PXD patterns of post-TGA Al_4C_3 with thermal analysis carried out under : (a) air, (b) argon and (c) nitrogen. Note the graphite reflection at $2\theta \sim 27^\circ$ is no longer present in the pattern from the air-heated product (a).

Table S1: Atomic parameters from Rietveld refinement.

Atom Type	Atom Site Label	X	Y	Z	Site occupancy	Thermal Displacement Type	Uiso	Multiplicity
Al	Al1	0	0	0.29407(5)	1.0	Uiso	0.0171(5)	6
Al	Al2	0	0	0.12953(4)	1.0	Uiso	0.0150(5)	6
C	C1	0	0	0	1.0	Uiso	0.0081(7)	3
C	C2	0	0	0.21700(13)	1.0	Uiso	0.0081(7)	6

Table S2: Selected atomic distances (Å) and angles (°) from Rietveld Refinement

Atomic Distances (Å) and Angles (°) in Microwave Synthesised Al ₄ C ₃			
Al(1)- Al(1)	2.7511(17)	Al(1)-Al(1)-C(2)	135.530(34)
Al(1)-Al(2)	2.9679(13)	C(1)-Al(1)-C(1)	101.013(34)
Al(1)-C(1)	2.1629(5)	C(1)-Al(1)-C(2)	116.991(28)
Al(1)-C(2)	1.9268(34)	Al(2)-Al(2)-Al(2)	77.17(5)
Al(2)- Al(1)	2.9679(13)	Al(2)-Al(2)-C(2)	46.067(35)
Al(2)- Al(2)	2.6762(16)	Al(2)-Al(2)-C(2)	53.65(10)
Al(2)- C(2)	2.1870(34)	Al(2)-Al(2)-C(2)	118.17(8)
Al(2)- C(2)	1.9554(6)	C(2)-Al(2)-C(2)	99.72(11)
C(1)- Al(1)	2.1629(5)	C(2)-Al(2)-C(2)	117.21(6)
C(2)- Al(1)	1.9268(34)	Al(1)-C(1)-Al(1)	101.013(34)
C(2)- Al(2)	2.1870(34)	Al(1)-C(1)-Al(1)	78.987(34)
C(2)- Al(2)	1.9554(6)	Al(1)-C(1)-Al(1)	180.0
		Al(1)-C(2)-Al(2)	180.0
Al(1)-Al(1)-Al(1)	74.70(5)	Al(1)-C(2)-Al(2)	99.72(11)
Al(1)-Al(1)-C(1)	50.506(17)	Al(2)-C(2)-Al(2)	80.28(11)
Al(1)-Al(1)-C(1)	107.48(6)	Al(2)-C(2)-Al(2)	117.21(6)

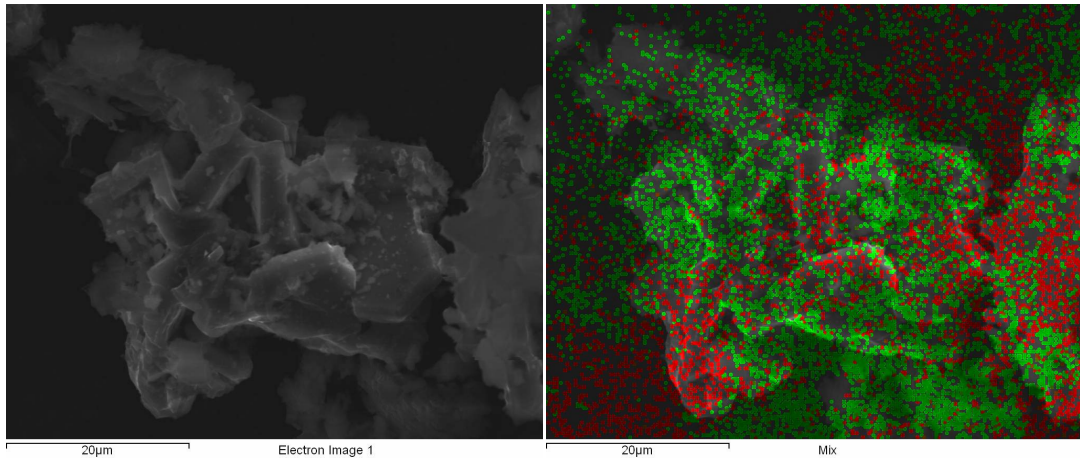


Fig. S2a SEM micrograph and element map; green = Al, red = C, for MW synthesised Al_4C_3

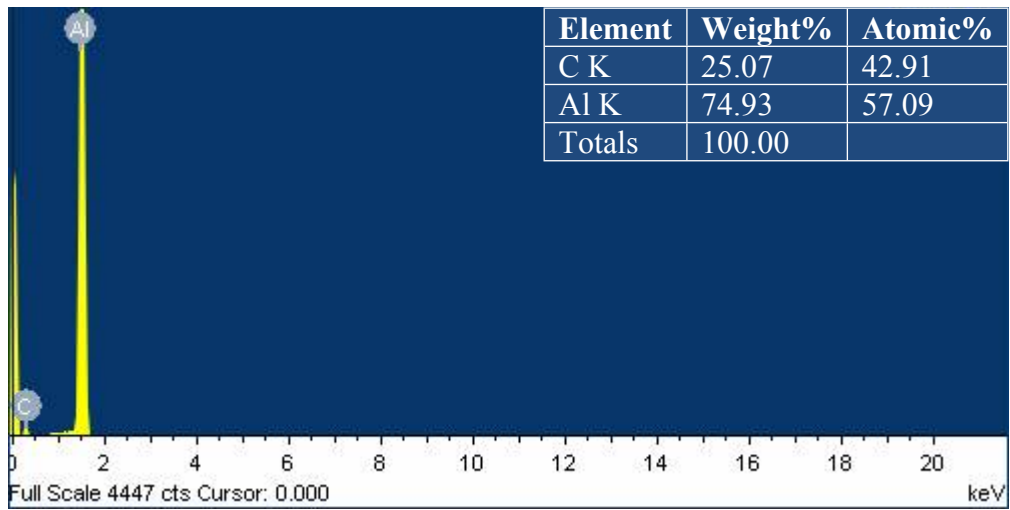


Fig. S2b EDX Spectrum for the MW synthesised Al_4C_3 sample above

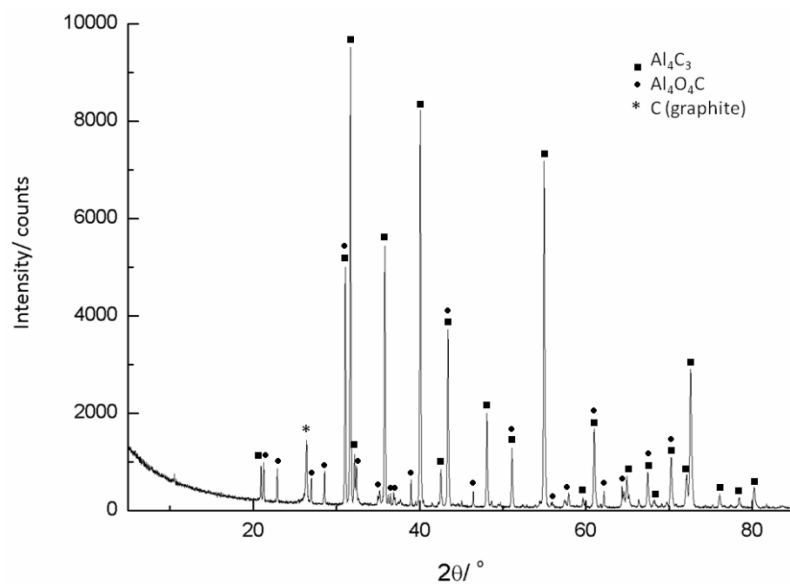
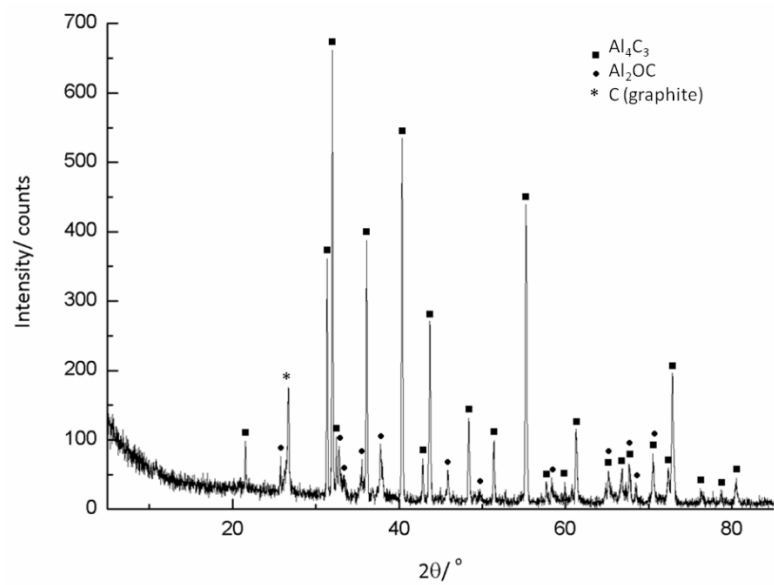


Fig. S3 *Ex-situ* PXD patterns from typical 4 Al + 3 C reactions performed in air for 30 min using graphite powder as a susceptor in the MMC reactor.