Supplemental Information

Unlocking the Secrets of Porous Silicon Formation: Insights into Magnesiothermic Reduction Mechanism using In-situ Powder X-ray Diffraction Studies

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Data analysis

XRD reflection area calculation. The scans from in-situ XRD were uploaded into a MatLab workspace where the MgO reflections were integrated using the trapz function to calculate the approximate area under the MgO reflection. The trapz function performs a numerical integration of a region by breaking the area of interest into trapezoids and measuring the area of those trapezoids. The initial MgO reflection area at low temperatures is assumed to be due to crystallization of the native oxide layer on the surface of the Mg particles. The crystallization of the native oxide layer can be seen in control experiments where both sMg and LMg were heated to 650°C (Figure S6 and S8). For most of the heating stage the native oxide layer slowly crystallizes until a point where the Mg fully melts, and the Mg reflections disappear. The derivative (rate of change) of the MgO reflection area over time was plotted to identify where the transition from native oxide crystallization to the formation of new MgO occurred. The start of the reaction (formation of MgO) was identified to occur when the value of the derivative of MgO reflection area was greater than the average of the baseline plus 3x the standard derivative of the baseline in the derivative plot. This statistically indicates the formation of new MgO that occurs through reaction 1. A summary of the values is shown in Table S1 and S2.

The same process was used to statistically determine the formation of Mg_2Si. Since there is no native Mg_2Si present in the sample, the reflection area itself was used for analysis rather than the derivative of the reflection area. The reflection areas were measured between 39 - 41 ° 2θ (Cu Kα). The start of Mg_2Si formation was identified to occur when the value of the Mg_2Si reflection area was greater than the average of the baseline plus 3x the standard derivative of the baseline in the plot. A summary of the values is shown in Table S3. As there were nearby reflections at higher angles, the baseline was subtracted from the patterns in order to determine where the Mg_2Si reflection disappeared. The disappearance of the reflection statistically occurred when the value of the Mg_2Si reflection area was less than the average of the new baseline plus 3x the

standard derivative of the new baseline in the baseline subtracted Mg_2Si reflection area plot. A summary of the values is shown in Table S4.

Reaction Onset Temperature Determination. In-situ VT XRD experiments whereby sMg and LMg were heated separately under argon revealed slight growth in the MgO (200) reflection between 200-400°C. This was presumed to be due to crystallization of the native oxide layer on the Mg particle surface. With this in mind, to obtain a true reaction onset temperature for reaction 1, the sMg or LMg datasets were subtracted from the dataset of the reaction of interest. This avoids identifying the crystallization of the native oxide as an indication of the reaction onset.

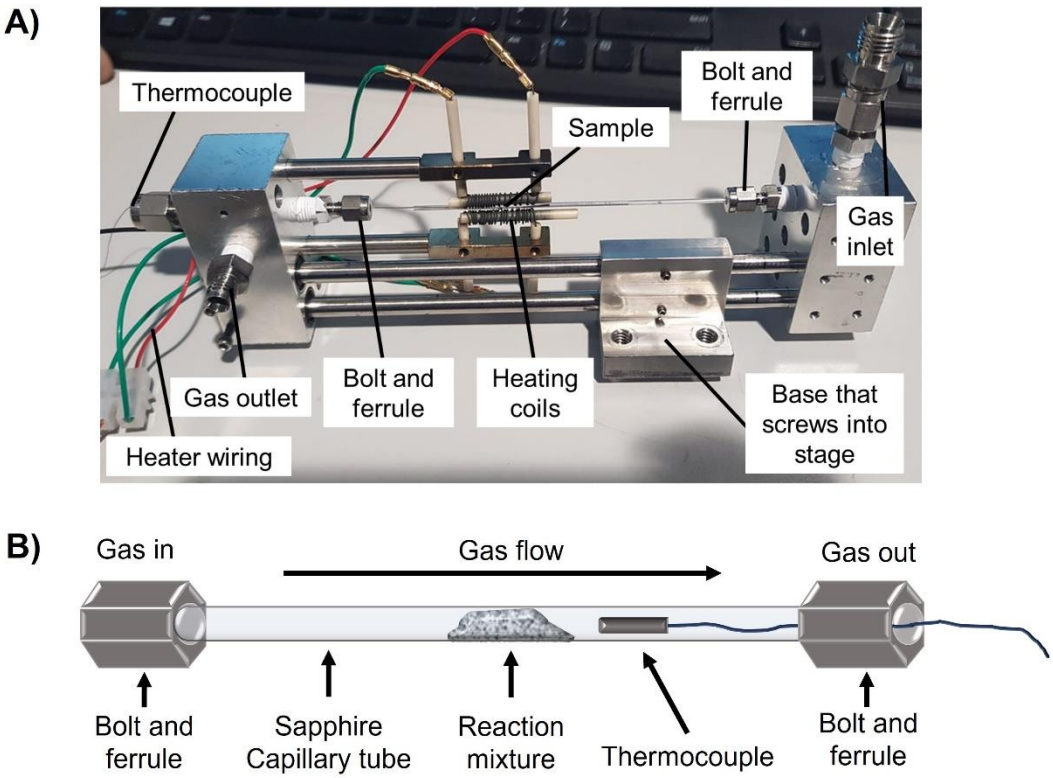


Figure S1: A) Sample holder and heating stage used for in-situ XRD experiments. B) Schematic of relative position of reaction mixture and thermocouple inside the sapphire capillary tube.

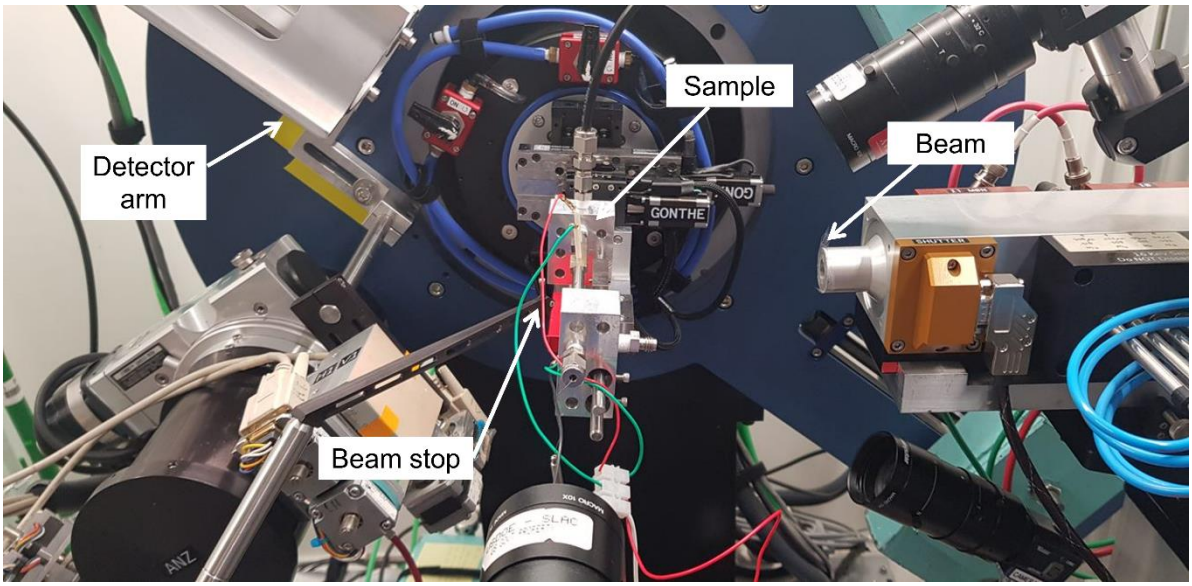


Figure S2: Beam-line setup for synchrotron experiments.

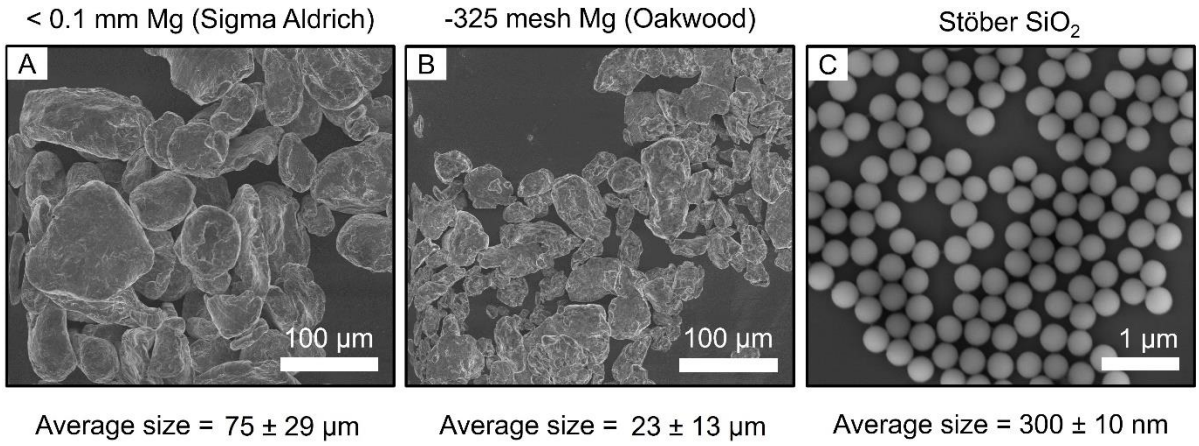


Figure S3: SEM images of Mg reagents along with average particle size measured lengthwise on ImageJ. A) LMg (<0.1 mm Mg from Sigma Aldrich), B) sMg (-325 mesh Mg from Oakwood Chemicals) and C) 300 nm Stöber SiO_2. Images and sizing in panel A and B are reproduced from reference 39 with permission from the author.

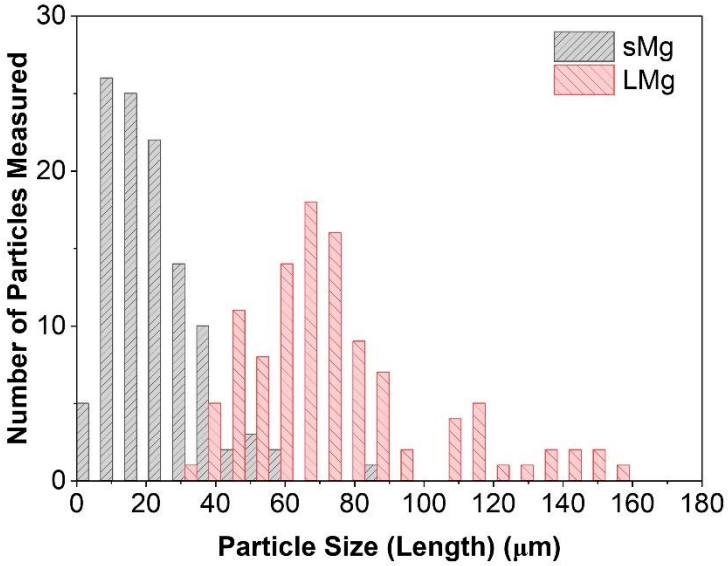


Figure S4: Histogram of size distribution of sMg and LMg particles. The diameter of 113 particles were measured lengthwise for each sample.

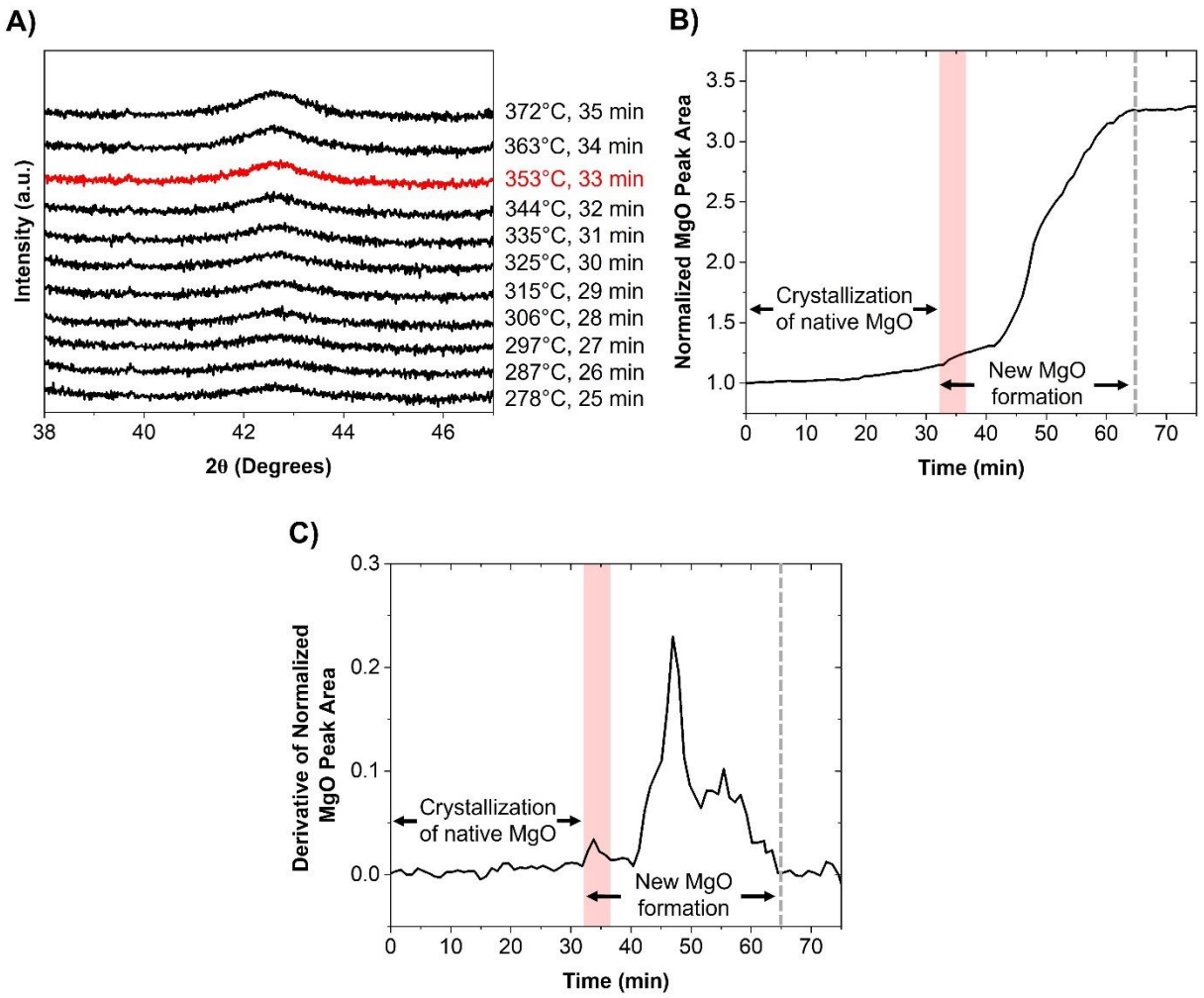


Figure S5: A) Waterfall plot of MgO reflections in sMg/SiO_2 system at 650°C, B) calculated MgO reflection area over time integrated from 40 to 44° 2θ for sMg sample at 650°C and C) calculated rate of change of the MgO reflection area over reaction time for sMg sample at 650°C. Red shading indicates when MgO begins to form via reaction 1. Grey hashed line indicates when the MgO reflection area plateaus. 2θ axis is in terms of Cu kα wavelength.

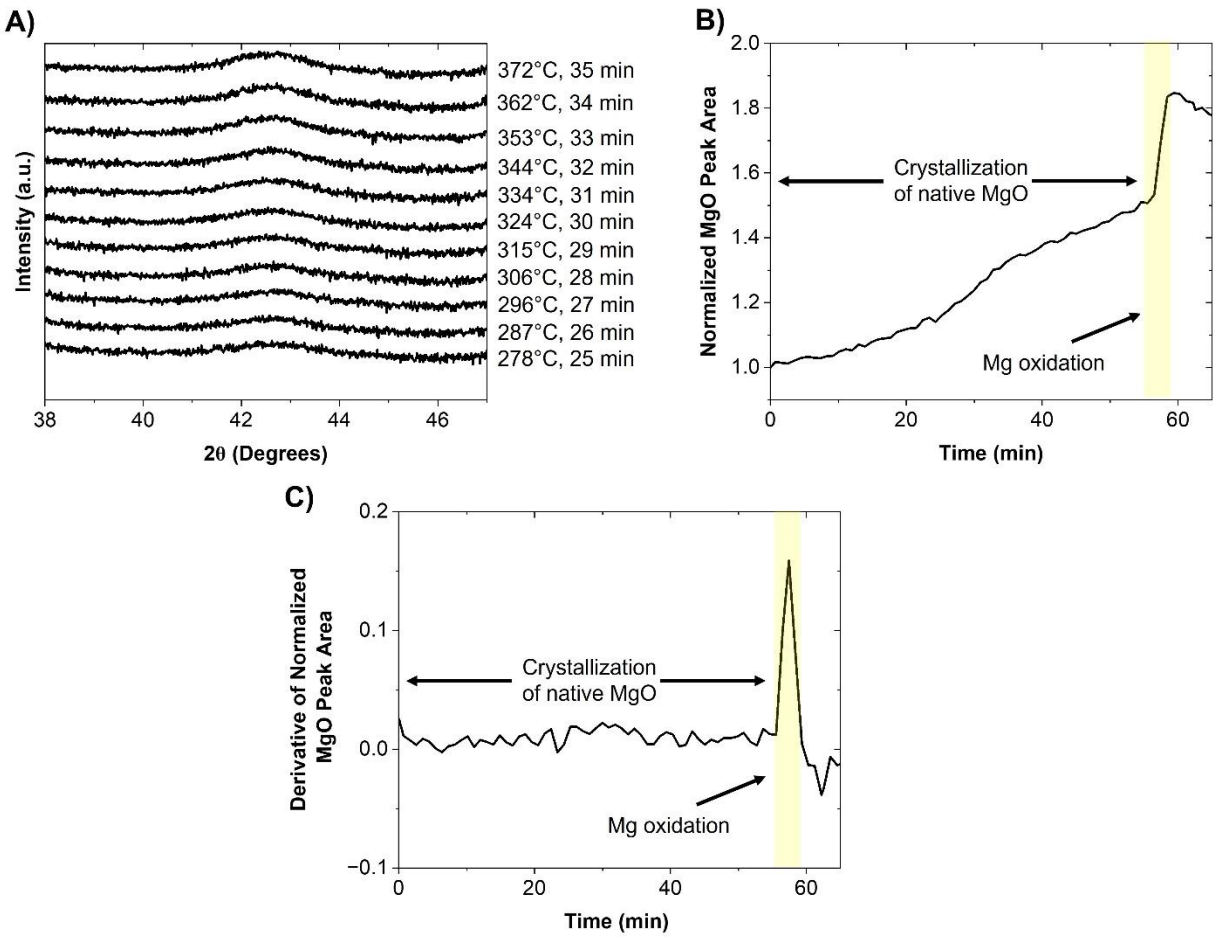


Figure S6: Control experiment for sMg by itself heated to 650°C at a ramp rate of 10°C/min. A) Waterfall plot of MgO reflection in sMg control sample at 650°C B) Calculated MgO reflection area over time integrated from 40 to 44° 2θ for sMg control sample at 650°C and C) calculated rate of change of the MgO reflection area over reaction time for sMg control sample at 650°C. Yellow shading indicates when MgO begins to form via the oxidation of Mg. 2θ axis is in terms of Cu kα wavelength.

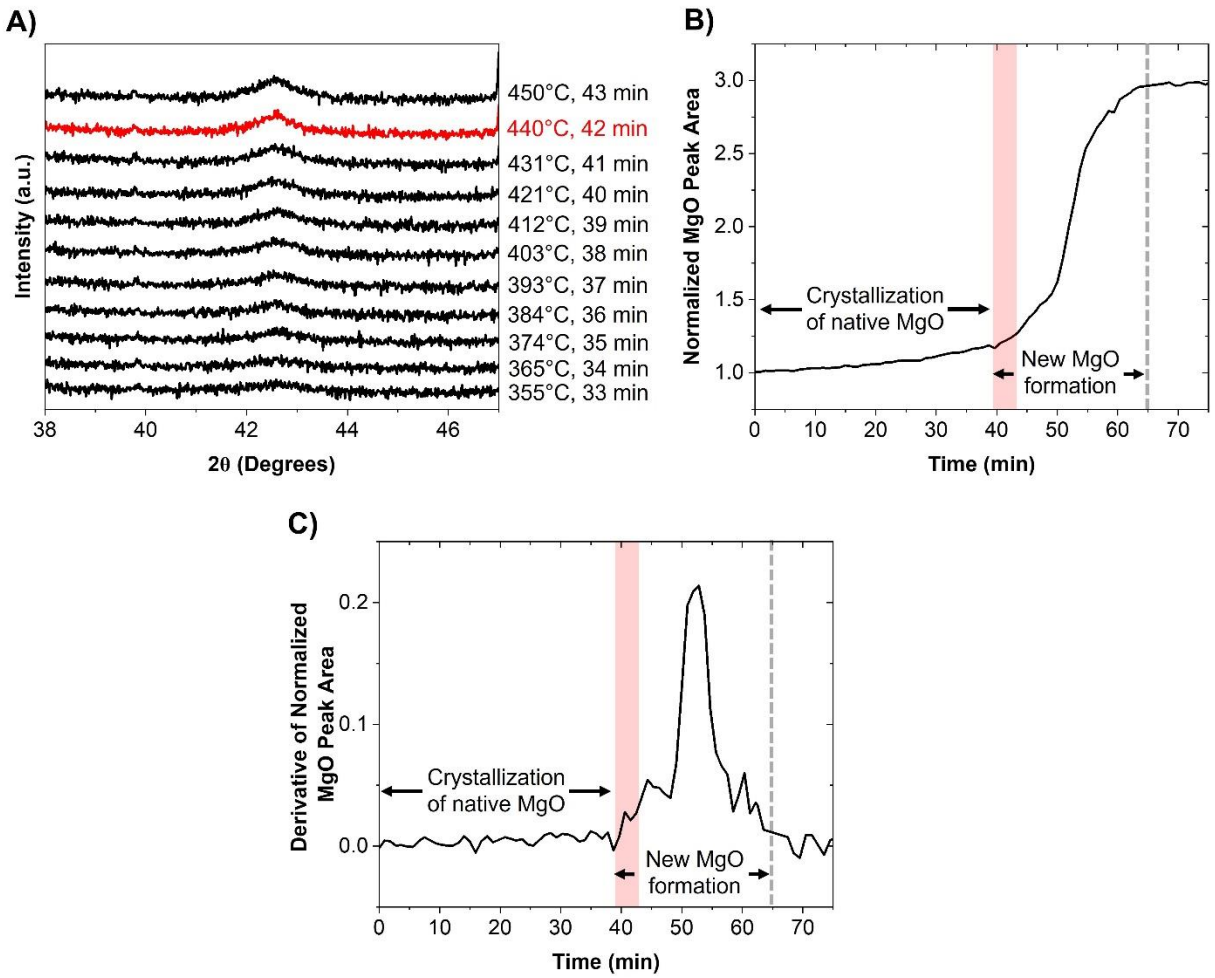


Figure S7: A) Waterfall plot of MgO reflection in LMg samples at 650°C B) Calculated MgO reflection area over time integrated from 40 to 44° 2θ for LMg sample at 650°C and C) calculated rate of change of the MgO reflection area over reaction time for LMg sample at 650°C. Red shading indicates when MgO begins to form via reaction 1. Grey hashed line indicates when the MgO reflection area plateaus. 2θ axis is in terms of Cu kα wavelength.

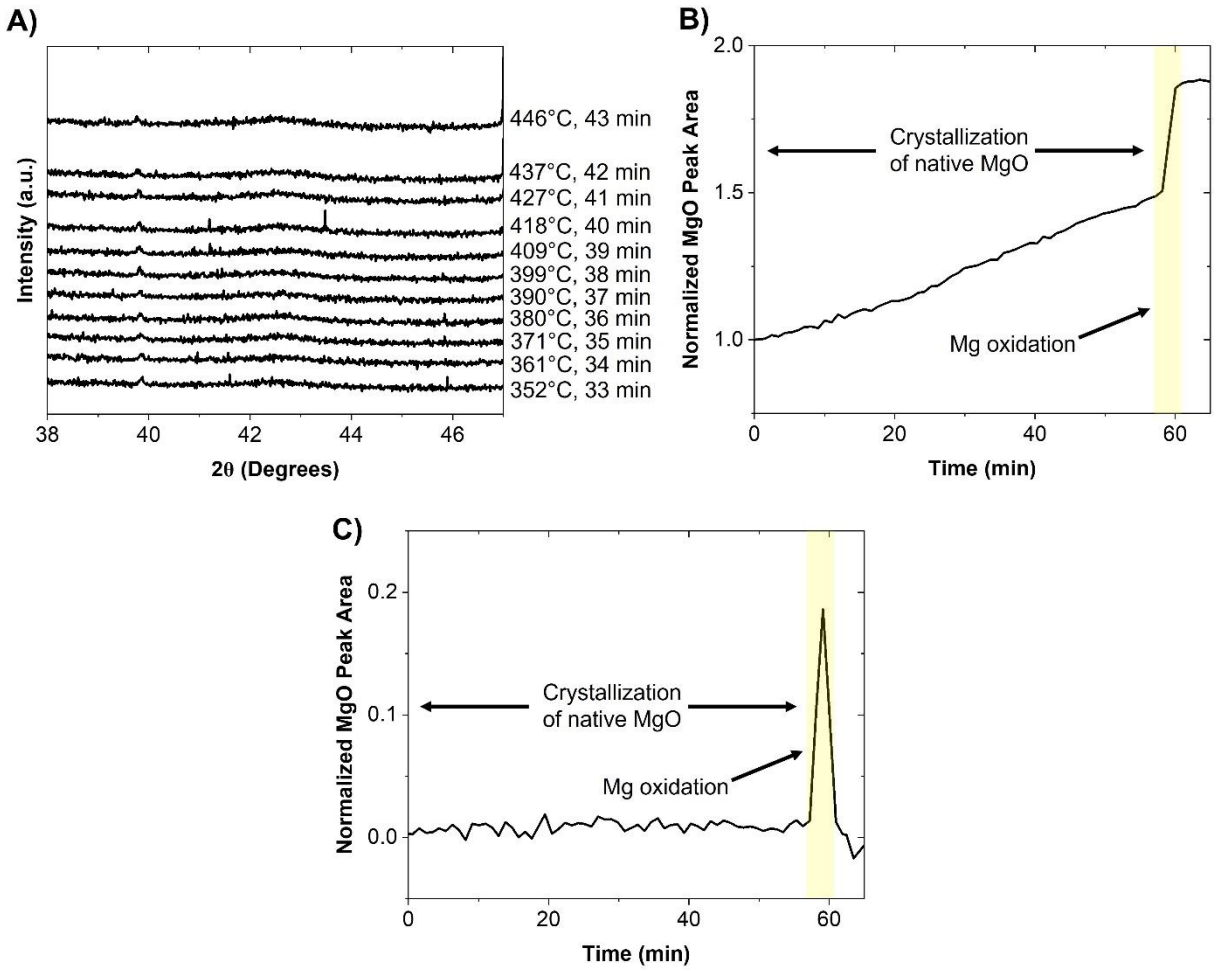


Figure S8: Control experiment for LMg by itself heated to 650°C at a ramp rate of 10°C/min. A) Waterfall plot of MgO reflection in LMg control sample at 650°C B) Calculated MgO reflection area over time integrated from 40 to 44° 2θ for LMg control sample at 650°C and C) calculated rate of change of the MgO reflection area over reaction time for LMg control sample at 650°C. Yellow shading indicates when MgO begins to form via the oxidation of Mg. 2θ axis is in terms of Cu kα wavelength.

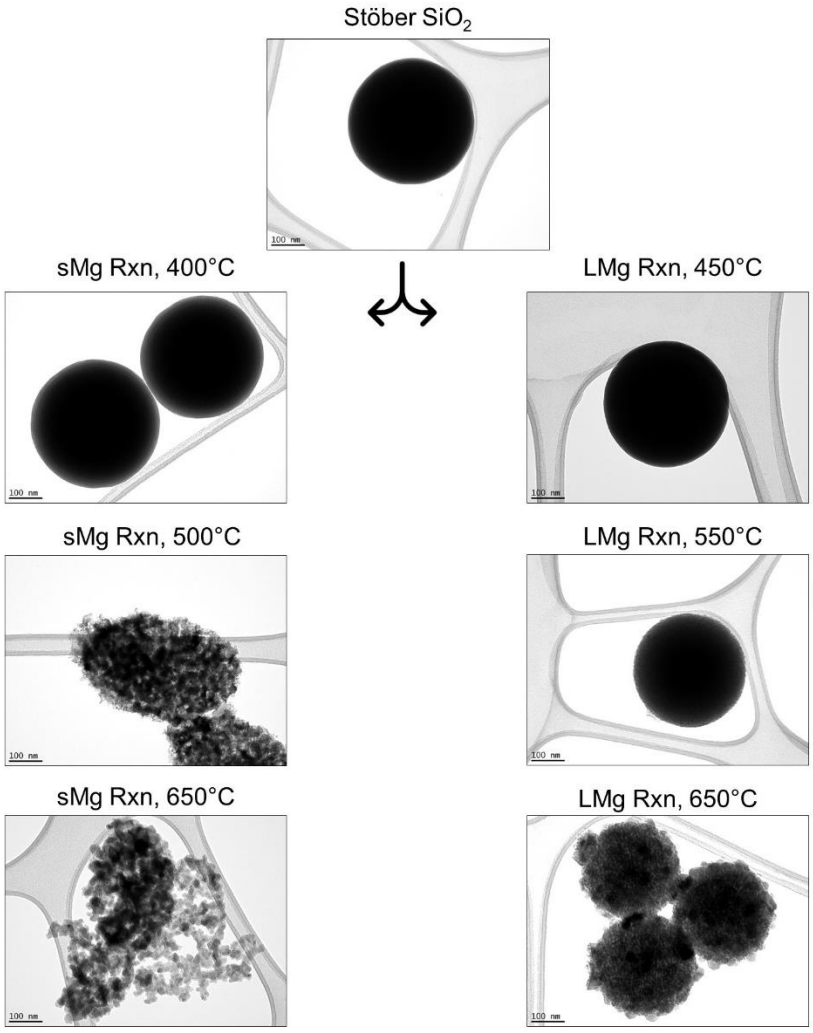


Figure S9. TEM images of SiO_2 or MgTR reaction products collected at the given reaction temperatures listed above followed by treatment with HCl (performed at laboratory scale).

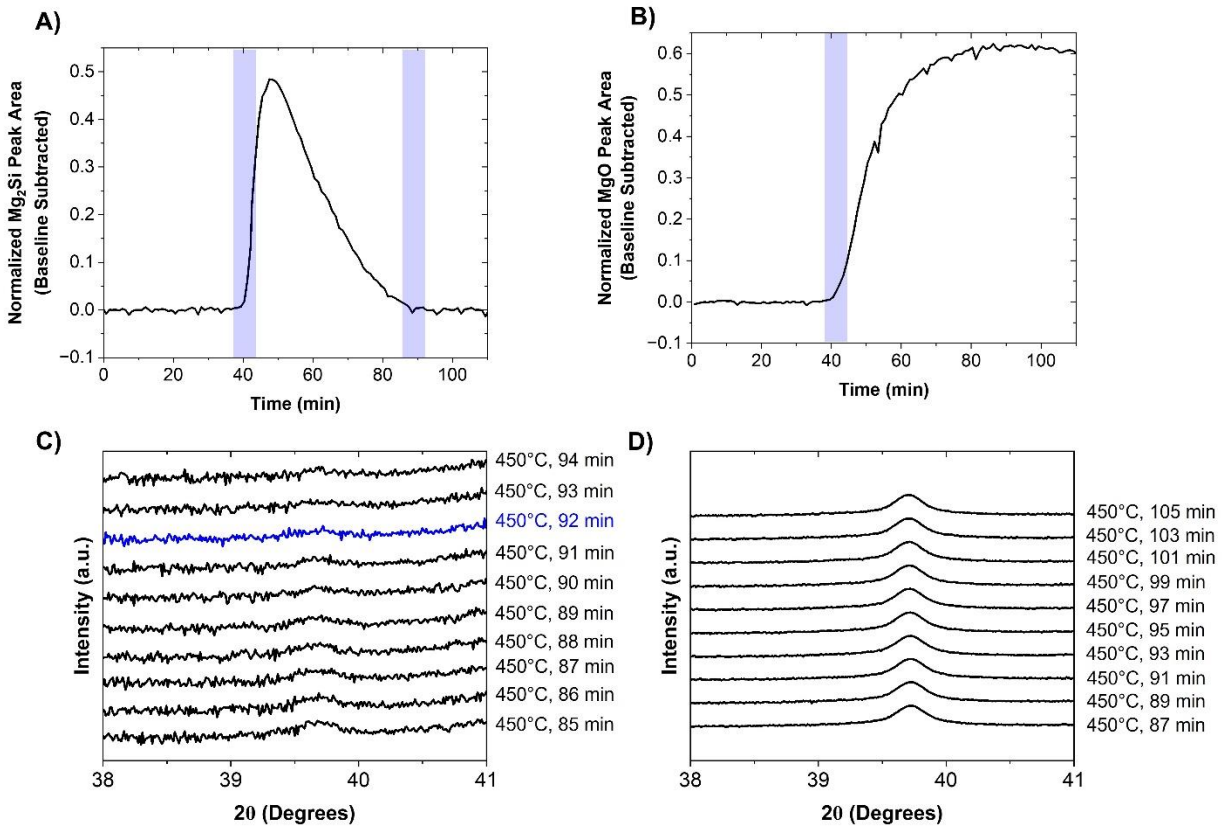


Figure S10: The change in Mg_2Si reflection area over time for 450°C samples involving A) sMg and B) LMg. The disappearance of Mg_2Si for samples involving C) sMg and D) LMg. Reaction events involving Mg_2Si are traced in blue. 2θ axis is in terms of Cu kα wavelength.

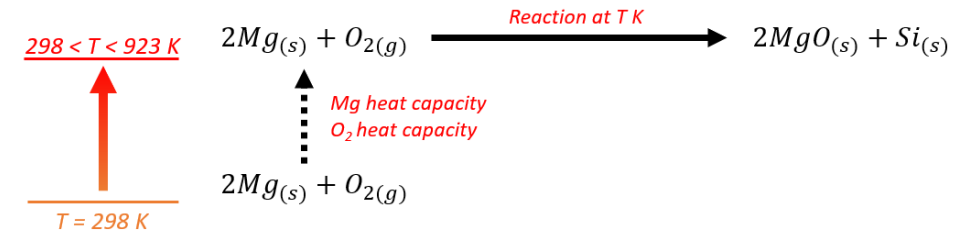
Thermodynamic calculations

Values for reaction enthalpy, entropy and Gibbs free energy were calculated for temperatures between 298 and 1300 K using the Shomate equations reported on the NIST Chemistry WebBook. Data for reactions 1 and 3 were not available and were calculated using the formation enthalpy and entropy of each compound present in the reaction and applying Hess's law. Calculations were corrected for phase changes where appropriate with values obtained from NIST.

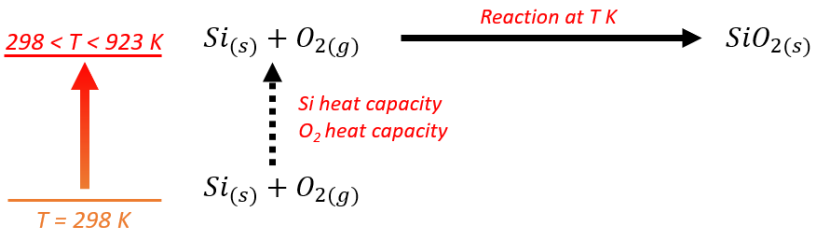
Shomate equations for calculating 1) the change in enthalpy from 298K up to a specified temperature T and 2) entropy (NIST Chemistry WebBook).

$$H - H_{298} = A * T + \frac{B * T^2}{2} + \frac{CT^3}{3} + \frac{DT^4}{4} - \frac{E}{T} + F - H \qquad \textbf{Equation S2}$$

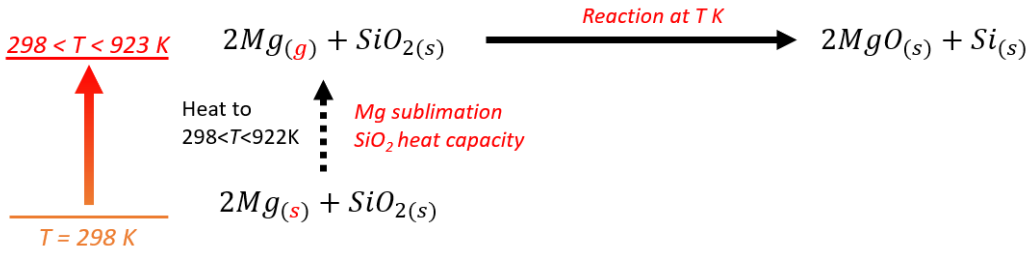
$$\Delta S = A * \ln(T) + BT + \frac{CT^2}{2} + \frac{DT^3}{3} - \frac{E}{2T^2} + G \qquad \textbf{Equation S3}$$



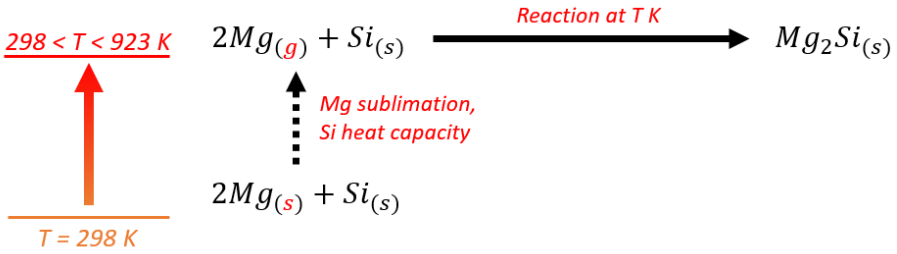
Process 1



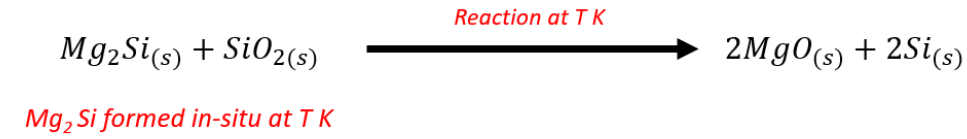
Process 2



Process 3



Process 4



Process 5

Schemes showing the physical and chemical processes for which thermodynamic calculations were performed in this work.

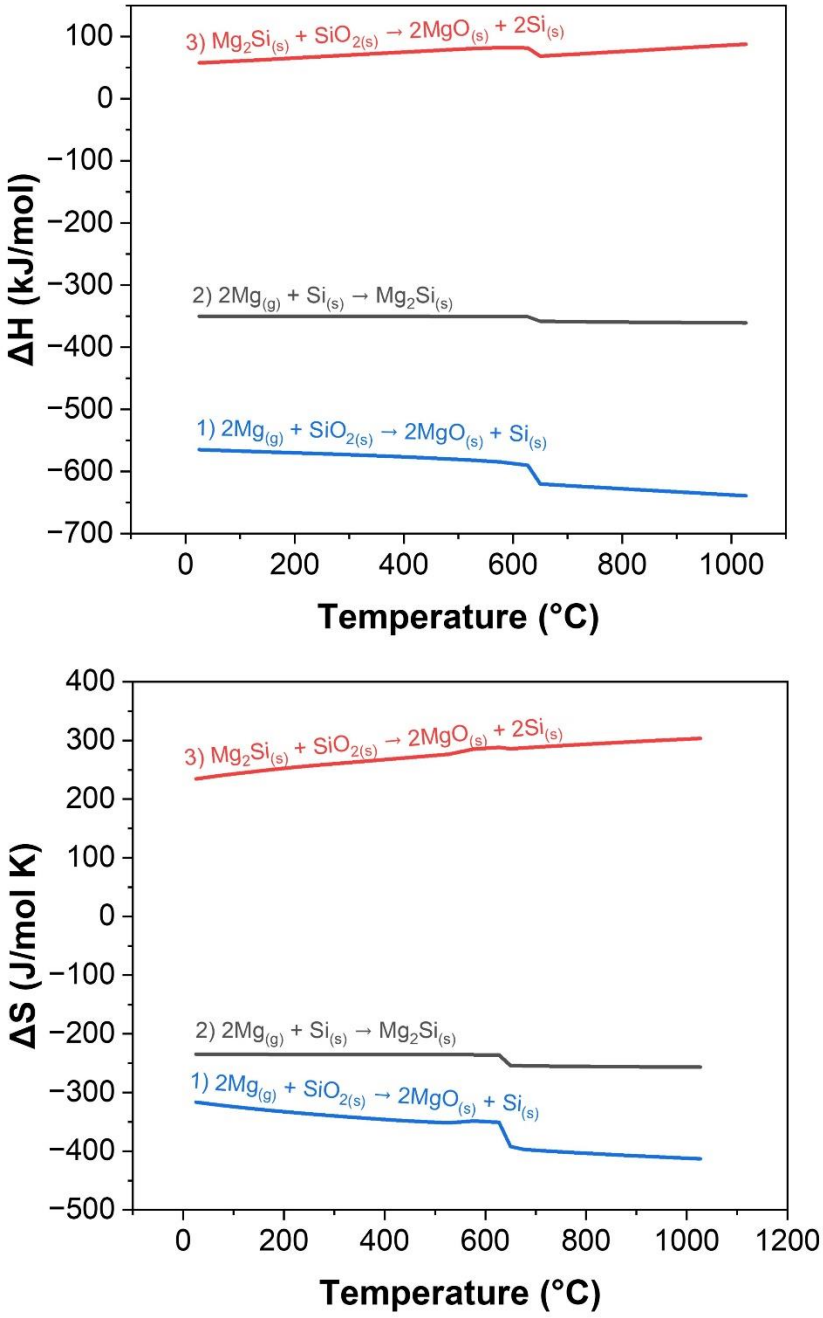


Figure S11: A) Reaction enthalpies and B) reaction entropies between 25° and 1027°C.

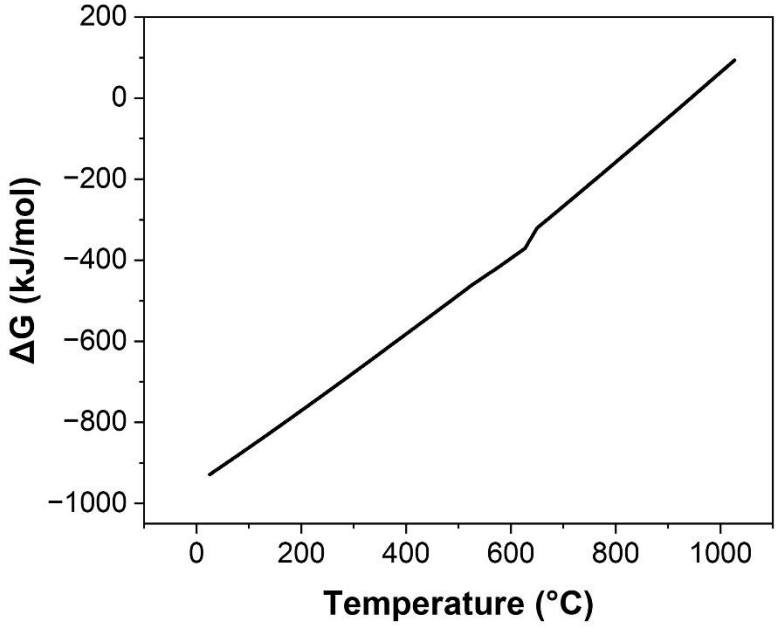


Figure S12. ΔG of reaction 4 between the range of 25 and 1027°C.

Table S1: List of values for the derivative of normalized MgO reflection area for sMg samples reacted at 650°C, 450°C as well as the sMg control sample heated to 650°C. Reflections indicating MgO growth were identified when the value for the derivative of normalized MgO reflection area was greater than the average baseline plus 3x the standard deviation of the baseline. These values are highlighted in yellow within the table.

sMg (reaction) 650°C		**sMg (control) 650°C**	
Average baseline + 3X STD		**Average baseline + 3X STD**	
0.0503		0.0171	
Derivative of MgO Reflection Area	**Temperature (°C)**	**Derivative of MgO Reflection Area**	**Temperature (°C)**
0.0004	26	0.0136	24
0.0010	32	0.0061	32
0.0014	42	0.0019	42
0.0000	51	0.0048	51
0.0001	61	0.0034	60
0.0018	70	0.0004	70
0.0010	80	-0.0014	79
0.0004	89	0.0013	89
-0.0005	99	0.0021	98
-0.0007	108	0.0041	108
0.0007	117	0.0058	117
0.0008	127	0.0010	127
0.0012	137	0.0042	136
0.0008	146	0.0033	145
0.0010	155	0.0021	155
0.0010	165	0.0062	164
-0.0013	174	0.0032	174
-0.0005	184	0.0017	183
0.0019	193	0.0058	193
0.0011	203	0.0068	202
0.0032	212	0.0032	211
0.0033	221	0.0018	221
0.0015	231	0.0071	230
0.0021	240	0.0091	240
0.0018	250	-0.0015	249
0.0024	259	0.0021	259
0.0018	268	0.0103	268
0.0026	278	0.0102	278
0.0019	287	0.0081	287
0.0011	297	0.0069	296
0.0020	306	0.0096	306
0.0032	316	0.0120	315
0.0034	325	0.0099	325

0.0034	335	0.0112	334
0.0025	344	0.0095	344
0.0069	353	0.0067	353
0.0102	363	0.0094	363
0.0067	372	0.0065	372
0.0058	382	0.0023	381
0.0043	391	0.0022	391
0.0043	400	0.0059	400
0.0048	410	0.0078	410
0.0046	419	0.0067	419
0.0025	429	0.0012	428
0.0073	438	0.0020	438
0.0185	448	0.0082	447
0.0254	457	0.0046	457
0.0291	466	0.0021	466
0.0328	476	0.0043	476
0.0479	485	0.0032	485
0.0686	495	0.0052	494
0.0582	504	0.0050	504
0.0339	513	0.0057	513
0.0258	523	0.0075	523
0.0224	532	0.0033	532
0.0192	542	0.0017	543
0.0243	551	0.0092	552
0.0242	561	0.0069	561
0.0232	570	0.0066	571
0.0305	579	0.0537	580
0.0224	589	0.0860	590
0.0209	598	0.0430	599
0.0230	607	0.0024	609
0.0175	617	-0.0073	618
0.0092	627	-0.0076	628
0.0093	637	-0.0072	637
0.0091	647	-0.0043	647
0.0056	650	-0.0034	650

Table S2: List of values for the derivative of normalized MgO reflection area for LMg samples reacted at 650°C, 450°C as well as the LMg control sample heated to 650°C**.** Reflections indicating MgO growth were identified when the value for the derivative of normalized MgO reflection area was greater than the average baseline plus 3x the standard deviation of the baseline. These values are highlighted in yellow within the table.

LMg (reaction) 650°C		**LMg (control) 650°C**	
Average baseline + 3X STD		**Average baseline + 3X STD**	
0.0050		0.0121	
Derivative of MgO Reflection Area	**Temperature (°C)**	**Derivative of MgO Reflection Area**	**Temperature (°C)**
-0.0007	26	0.0015	25
-0.0005	33	0.0013	31
0.0015	43	0.0039	40
0.0013	52	0.0020	50
0.0002	62	0.0022	59
0.0003	71	0.0039	69
-0.0001	80	0.0027	78
-0.0002	90	0.0054	88
0.0014	100	0.0032	97
0.0024	109	-0.0011	106
0.0016	118	0.0059	116
0.0007	128	0.0053	125
0.0004	137	0.0059	135
0.0001	147	0.0043	144
0.0001	156	0.0006	154
0.0009	165	0.0066	163
0.0027	175	0.0043	173
0.0012	184	0.0000	182
-0.0017	194	0.0024	191
0.0015	204	-0.0005	201
0.0028	213	0.0053	210
0.0010	233	0.0100	220
0.0024	242	0.0016	230
0.0020	251	0.0036	239
0.0015	261	0.0063	248
0.0019	270	0.0052	258
0.0005	280	0.0062	267
0.0001	299	0.0058	277
0.0029	308	0.0047	286
0.0035	318	0.0090	295
0.0024	327	0.0077	305
0.0033	337	0.0078	314
0.0031	346	0.0064	324

0.0025	355	0.0027	333
0.0012	365	0.0040	343
0.0017	374	0.0055	352
0.0040	384	0.0027	361
0.0033	393	0.0067	371
0.0020	403	0.0082	380
0.0037	412	0.0039	390
-0.0011	421	0.0054	399
0.0028	431	0.0056	409
0.0092	440	0.0019	418
0.0069	450	0.0059	428
0.0089	459	0.0049	437
0.0132	469	0.0031	446
0.0176	478	0.0070	456
0.0159	488	0.0053	465
0.0157	497	0.0073	474
0.0138	506	0.0063	484
0.0130	516	0.0046	494
0.0223	525	0.0043	503
0.0418	534	0.0047	512
0.0644	544	0.0040	522
0.0688	553	0.0028	531
0.0691	563	0.0037	541
0.0612	572	0.0035	550
0.0370	582	0.0023	559
0.0248	591	0.0056	569
0.0209	600	0.0074	578
0.0197	610	0.0049	587
0.0097	619	0.0072	597
0.0142	629	0.0558	607
0.0200	638	0.0988	616
0.0084	647	0.0527	625
0.0062	650	0.0066	634

Table S3 : List of values for the normalized Mg_2Si reflection area for sMg and LMg samples reacted at 650°C. Reflections indicating Mg_2Si growth were identified when the value for the normalized Mg_2Si reflection area was greater than the average baseline plus 3x the standard deviation of the baseline. These values are highlighted in yellow within the table.

sMg (reaction) 650°C		**LMg (reaction) 650°C**	
Average baseline + 3X STD		**Average baseline + 3X STD**	
0.4984		0.6605	
Mg_2Si Reflection Area	**Temperature (°C)**	**Mg_2Si Reflection Area**	**Temperature (°C)**
0.4434	26	0.5659	26
0.4484	32	0.5674	33
0.4453	51	0.5684	43
0.4487	61	0.5742	52
0.4529	70	0.5781	62
0.4530	80	0.5763	71
0.4506	89	0.5749	80
0.4563	99	0.5759	90
0.4559	108	0.5727	100
0.4514	117	0.5796	109
0.4553	127	0.5789	118
0.4581	137	0.5858	128
0.4592	146	0.5873	137
0.4553	155	0.5826	147
0.4580	165	0.5801	156
0.4600	174	0.5928	165
0.4442	184	0.5912	175
0.4650	193	0.5937	184
0.4589	203	0.5941	194
0.4619	212	0.5967	204
0.4680	221	0.5967	213
0.4698	231	0.5985	222
0.4686	240	0.5930	233
0.4618	250	0.6009	242
0.4686	259	0.6009	251
0.4713	268	0.6070	261
0.4685	278	0.6087	270
0.4738	287	0.6054	280
0.4730	297	0.6069	289
0.4744	306	0.6054	299
0.4677	316	0.6151	308
0.4750	325	0.6103	318
0.4747	335	0.6171	327
0.4796	344	0.6174	337
0.4758	353	0.6188	346

0.4802	363	0.6198	355
0.4786	372	0.6233	365
0.4690	382	0.6228	374
0.4799	391	0.6247	384
0.4779	400	0.6265	393
0.4805	410	0.6264	403
0.4822	419	0.6288	412
0.4827	429	0.6310	421
0.4823	438	0.6320	431
0.4896	448	0.6342	440
0.5192	457	0.6412	450
0.5678	466	0.6519	459
0.6108	476	0.6688	469
0.6756	485	0.6784	478
0.8111	495	0.7002	488
0.9579	504	0.7158	497
1.0000	513	0.7299	506
0.9974	523	0.7548	516
0.9592	532	0.7796	525
0.9194	542	0.8247	534
0.8668	551	0.8949	544
0.8177	561	0.9579	553
0.7655	570	0.9857	563
0.7155	579	1.0000	572
0.6749	589	0.9772	582
0.6361	598	0.9355	591
0.6018	607	0.8898	600
0.5766	617	0.8505	610
0.5560	627	0.8270	619
0.5387	637	0.7857	629
0.5235	647	0.7682	638
0.5179	650	0.7500	647
0.5172	650	0.7352	650
0.5112	650	0.7237	650
0.5088	650	0.7189	650
0.5072	650	0.7156	650
0.5088	650	0.6989	650
0.4882	650	0.7042	650
0.5053	650	0.6991	650
0.5018	650	0.6983	650
0.5021	650	0.6933	650
0.4993	650	0.6949	650
0.4997	650	0.6916	650

0.4973	650	0.6840	650
0.4934	650	0.6794	650
0.4918	650	0.6843	650
0.4970	650	0.6791	650
0.4947	650	0.6760	650
0.4961	650	0.6746	650
0.4962	650	0.6752	650
0.4990	650	0.6650	650
0.4889	650	0.6653	650
0.4952	650	0.6627	650
0.4959	650	0.6633	650
0.4953	650	0.6565	650
0.4954	650	0.6569	650
0.4897	650	0.6455	650
0.4961	650	0.6431	650
0.4913	650	0.6403	650
0.4910	650	0.6333	650
0.4953	650	0.6282	650
0.4929	650	0.6265	650
0.4904	650	0.6242	650
0.4923	650	0.6282	650
0.4913	650	0.6231	650
0.4900	650	0.6297	650
0.4919	650	0.6339	650
0.4924	650	0.6331	650
0.4916	650	0.6348	650
0.4923	650	0.6305	650
0.4933	650	0.6389	650
0.4953	650	0.6412	650
0.4949	650	0.6357	650
0.4970	650	0.6423	650
0.4951	650	0.6386	650
0.4957	650	0.6379	650
0.4983	650	0.6386	650
0.4932	650	0.6401	650
0.4995	650	0.6439	650

Table S4: List of values for the baseline subtracted, normalized Mg_2Si reflection area for sMg and LMg samples reacted at 650°C and 450°C**.** As per Table S3, reflections indicating Mg_2Si growth were identified when the value for the normalized Mg_2Si reflection area was greater than the average baseline plus 3x the standard deviation of the baseline (shown in yellow). The disappearance of the Mg_2Si reflection is indicated when the value for the baseline subtracted, normalized Mg_2Si reflection area was greater than the average baseline plus 3x the standard deviation of the baseline. These values are highlighted in red within the table.

sMg (reaction) 650°C		**sMg (reaction) 450°C**		**LMg (reaction) 650°C**	
Average baseline + 3X STD		**Average baseline + 3X STD**		**Average baseline + 3X STD**	
0.0102		0.0024		0.0049	
Mg_2Si Reflection Area	**Temperature (°C)**	**Mg_2Si Reflection Area**	**Temperature (°C)**	**Mg_2Si Reflection Area**	**Temperature (°C)**
0.0018	26	0.0006	25	0.0000	26
0.0058	32	-0.0081	32	0.0002	33
0.0000	51	0.0001	41	-0.0024	43
0.0022	61	0.0000	50	0.0016	52
0.0050	70	0.0005	61	0.0038	62
0.0037	80	-0.0014	70	0.0000	71
0.0000	89	0.0025	79	-0.0019	80
0.0046	99	-0.0004	88	-0.0014	90
0.0029	108	-0.0107	97	-0.0051	100
-0.0027	117	-0.0027	107	0.0012	109
0.0000	127	0.0000	117	0.0000	118
0.0021	137	-0.0004	126	0.0040	128
0.0025	146	0.0001	135	0.0027	137
-0.0020	155	0.0060	145	-0.0047	147
0.0000	165	0.0007	154	-0.0100	156
0.0012	174	-0.0043	163	0.0000	165
-0.0154	184	0.0016	173	-0.0026	175
0.0046	193	0.0000	182	-0.0012	184
-0.0023	203	0.0021	192	-0.0018	194
0.0000	212	0.0009	202	-0.0002	204
0.0061	221	-0.0025	211	-0.0012	213
0.0080	231	-0.0007	220	-0.0004	222
0.0068	240	-0.0076	230	-0.0069	233
0.0000	250	-0.0014	239	0.0000	242
0.0038	259	0.0000	249	-0.0012	251
0.0035	268	-0.0019	258	0.0037	261
-0.0024	278	0.0015	267	0.0042	270
0.0000	287	0.0046	277	-0.0003	280
-0.0011	297	0.0028	287	0.0000	289
0.0000	306	-0.0099	296	-0.0037	299
-0.0070	316	0.0062	305	0.0040	308

0.0000	325	0.0000	315	-0.0029	318
-0.0017	335	0.0030	324	0.0018	327
0.0020	344	0.0052	334	0.0000	337
-0.0031	353	0.0000	343	0.0000	346
0.0000	363	0.0030	352	-0.0005	355
-0.0023	372	-0.0065	362	0.0015	365
-0.0127	382	0.0011	371	-0.0004	374
-0.0026	391	0.0031	380	0.0000	384
-0.0054	400	0.0014	390	0.0017	393
-0.0035	410	0.0039	400	0.0015	403
-0.0026	419	0.0044	409	0.0037	412
-0.0029	429	0.0066	418	0.0058	421
-0.0041	438	0.0177	428	0.0068	431
0.0025	448	0.0622	437	0.0088	440
0.0314	457	0.1353	446	0.0157	450
0.0792	466	0.2202	450	0.0262	459
0.1215	476	0.3161	450	0.0431	469
0.1855	485	0.3999	451	0.0525	478
0.3203	495	0.4478	450	0.0742	488
0.4663	504	0.4614	450	0.0897	497
0.5077	513	0.4842	450	0.1036	506
0.5042	523	0.4832	450	0.1285	516
0.4653	532	0.4789	450	0.1531	525
0.4248	542	0.4676	450	0.1981	534
0.3714	551	0.4524	450	0.2682	544
0.3215	561	0.4359	450	0.3310	553
0.2685	570	0.4158	450	0.3587	563
0.2179	579	0.3966	450	0.3729	572
0.1764	589	0.3748	450	0.3500	582
0.1369	598	0.3613	450	0.3081	591
0.1019	607	0.3377	450	0.2623	600
0.0759	617	0.3187	450	0.2229	610
0.0545	627	0.2999	450	0.1993	619
0.0364	637	0.2756	450	0.1578	629
0.0204	647	0.2697	450	0.1402	638
0.0140	650	0.2484	450	0.1218	647
0.0125	650	0.2405	450	0.1069	650
0.0057	650	0.2287	450	0.0953	650
0.0024	650	0.2079	450	0.0903	650
0.0000	650	0.1974	450	0.0869	650
0.0030	650	0.1724	450	0.0701	650
-0.0162	650	0.1674	450	0.0753	650
0.0022	650	0.1551	450	0.0701	650

0.0000	650	0.1406	450	0.0691	650
0.0014	650	0.1254	450	0.0640	650
-0.0002	650	0.1167	450	0.0654	650
0.0012	650	0.1045	450	0.0621	650
0.0000	650	0.0882	450	0.0543	650
-0.0033	650	0.0802	450	0.0496	650
-0.0042	650	0.0758	450	0.0544	650
0.0017	650	0.0653	450	0.0490	650
0.0000	650	0.0605	450	0.0458	650
0.0028	650	0.0509	450	0.0443	650
0.0044	650	0.0456	450	0.0447	650
0.0086	650	0.0282	450	0.0343	650
0.0000	650	0.0287	450	0.0345	650
0.0047	650	0.0253	450	0.0318	650
0.0038	650	0.0204	450	0.0322	650
0.0015	650	0.0162	450	0.0254	650
0.0000	650	0.0121	450	0.0256	650
-0.0059	650	0.0070	450	0.0141	650
0.0004	650	0.0037	450	0.0115	650
-0.0046	650	0.0038	449	0.0086	650
-0.0051	650	0.0056	450	0.0020	650
-0.0010	650	0.0000	450	-0.0022	650
-0.0035	650	-0.0015	450	-0.0030	650
-0.0062	650	0.0002	450	-0.0043	650
-0.0045	650	-0.0095	450	0.0005	650
-0.0057	650	-0.0037	450	-0.0055	650
-0.0071	650	0.0033	450	-0.0003	650
-0.0054	650	0.0013	450	0.0024	650
-0.0051	650	0.0000	450	0.0000	650
-0.0061	650	-0.0001	450	0.0012	650
-0.0055	650	0.0040	450	-0.0036	650
-0.0048	650	-0.0078	450	0.0043	650
-0.0029	650	0.0013	450	0.0060	650
-0.0035	650	0.0023	450	0.0000	650
-0.0016	650	0.0045	450	0.0057	650
-0.0037	650	0.0000	450	0.0011	650
-0.0032	650	0.0003	450	-0.0004	650
-0.0008	650	-0.0008	450	-0.0007	650
-0.0061	650	-0.0139	450	0.0000	650