Supplementary Information (SI) for RSC Mechanochemistry.

ELECTRONIC SUPPLEMENTARY INFORMATION

Resonant Acoustic Mixing-Induced Polymorphic Transformation of Glycine Observed by Solid-State NMR

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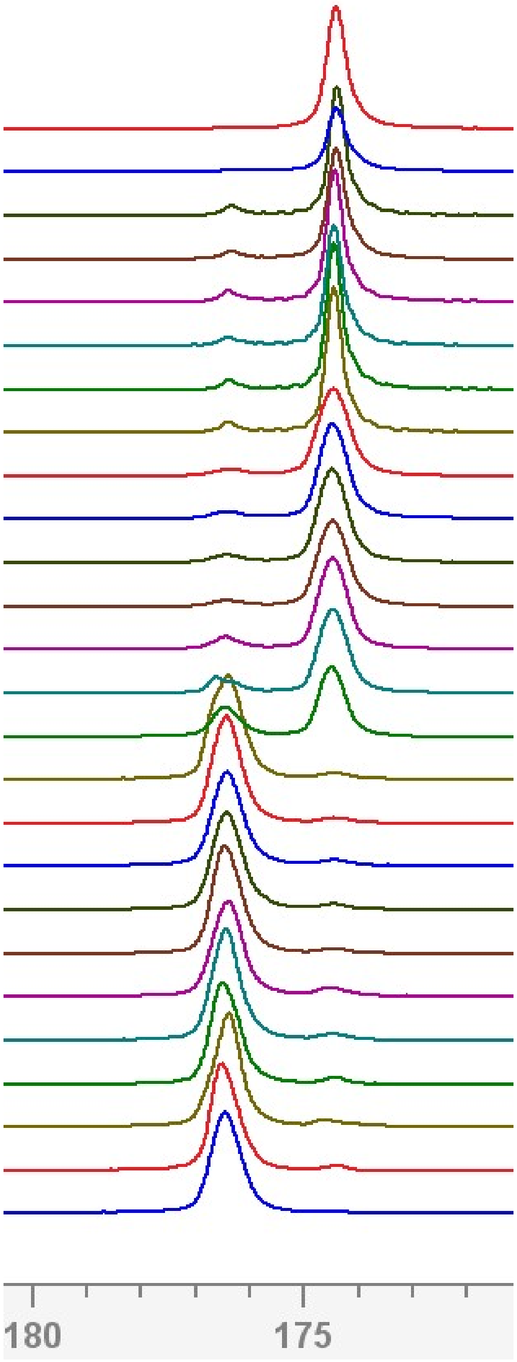

Figure S1. ^{13}C CP/MAS NMR spectra of glycine from Sigma-Aldrich with recrystallization and grinding at various timepoints after RAM at high level (glass vial filled with 1.54 g of glycine), recorded using a Bruker Avance III 200 NMR spectrometer with ν_{rot} = 3000 Hz. Time scale goes from zero to 750 minutes in increments of 30 minutes, from bottom to top. Scale in ppm.

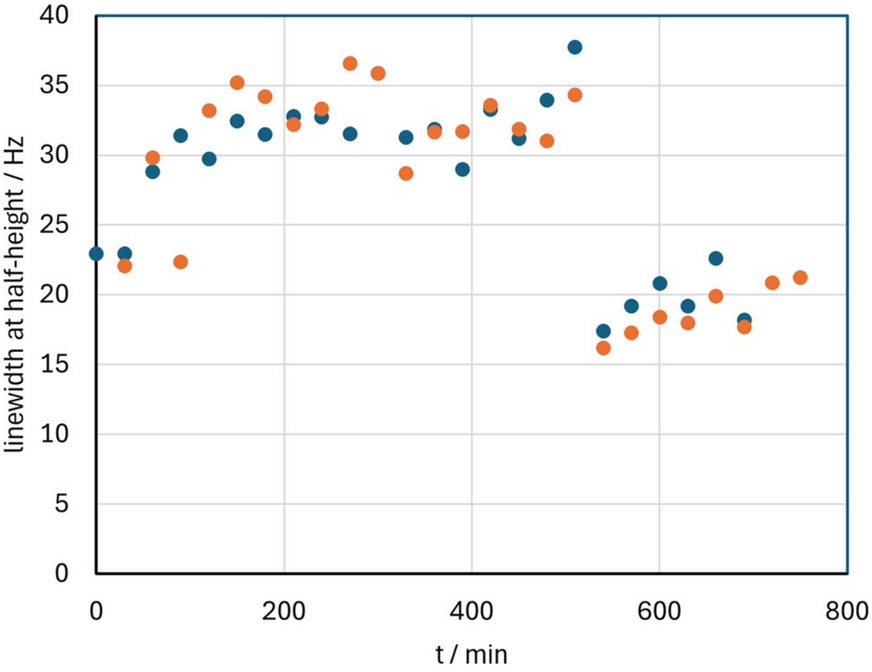

Figure S2. Linewidths at half-height vs time for the spectra shown in Figure S1 (glass vial filled with 1.54 g of glycine). Dark blue data points are for the α-glycine carbonyl peak and orange data points are for the γ-glycine carbonyl peak. Spectra were acquired with a 68 ms acquisition time, TD = 2k, SI = 16k, and 1 Hz of exponential apodization. Both peaks broaden slightly from ~22 Hz at time ~0 to a maximum of ~35 Hz at ~510 min; this is followed by a resharpening of both peaks during the final phase of the transformation. We speculate that the initial broadening reflects the introduction of some crystal defects as a result of RAM, fuelling the polymorphic transformation; and that the resharpening reflects the completion of the transformation.

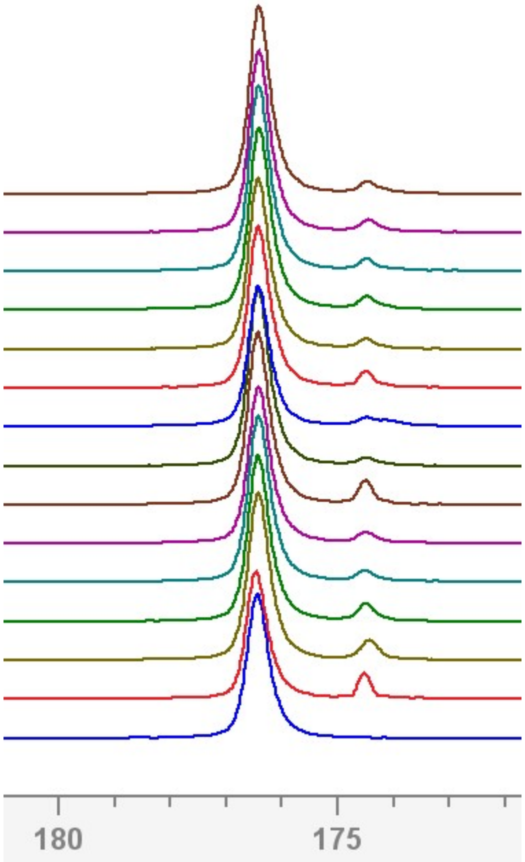

Figure S3. ^{13}C CP/MAS NMR spectra of glycine from Sigma-Aldrich with recrystallization and grinding at various timepoints after RAM at high level (glass vial filled with 7.30 g of glycine), recorded using a Bruker Avance III 200 NMR spectrometer with v_{rot} = 3000 Hz. Time scale goes from zero to 420 minutes in increments of 30 minutes, from bottom to top. Scale in ppm.

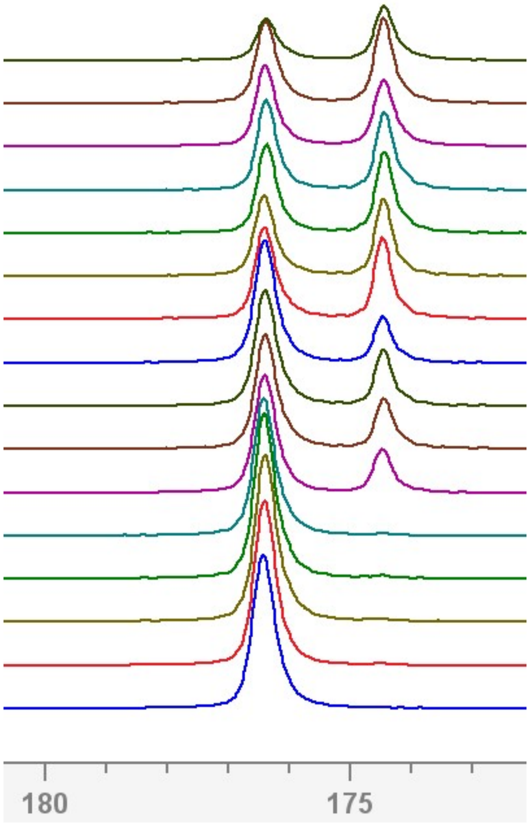

Figure S4. ^{13}C CP/MAS NMR spectra of glycine from Sigma-Aldrich with recrystallization and grinding at various timepoints after RAM at high level (glass vial filled with 1.70 g of glycine), recorded using a Bruker Avance III 200 NMR spectrometer with $\nu_{\mathrm{rot}} = 3000$ Hz. Time scale goes from zero to 900 minutes in increments of 60 minutes, from bottom to top. Scale in ppm.

Table S1. Peak areas of the carbonyl peak of α-glycine and γ-glycine as reported by the Bruker TopSpin application for the solid-state ^{13}C CP/MAS NMR spectra produced during the time-course experiment shown in Figure S1 (1.54 g glycine).

Timepoint (min)	Peak area of α-glycine carbonyl peak (176.4 ppm)	Peak area of γ-glycine carbonyl peak (174.4 ppm)
0	3007287.84	0
30	3200335.27	160510.59
60	3403955.38	202046.41
90	3039722.99	215329.7
120	3337786.08	197123.79
150	2843835.7	252656.19
180	3240739.02	166743.8
210	2920043.69	178942.59
240	2804553.39	184378.18
270	3212690.41	172480.62
300	3111309.4	193872.75
330	901551.43	2106707.28
360	482549.16	2508126.12
390	379763.17	2728378.57
420	188236.66	2568434.45
450	232595.91	2807087.09
480	218295.45	2840763.48
510	200231.93	2626158.78
540	314341.48	4328370.03
570	307265.55	4399103.75
600	264765.53	3622110.45
630	342336.46	3948004.21
660	245760.67	3316589.64
690	295580.09	3871246.36
720	45148.28	1905139.18
750	0	3673116.57

Table S2. Peak areas of the carbonyl peak of α-glycine and γ-glycine as reported by the Bruker TopSpin application for the solid-state ^{13}C CP/MAS NMR spectra produced during the time-course experiment shown in Figure S3 (7.30 g glycine).

Timepoint (min)	Peak area of α-glycine carbonyl peak (176.4 ppm)	Peak area of γ-glycine carbonyl peak (174.4 ppm)
0	3989809.7	0
30	3513111.3	712572.8
60	4627635.38	550339.09
90	4599964.58	505418.56
120	4587259.84	319196.17
150	4334762.06	309635.52
180	4764984.06	678707.44
210	4914010.05	240950.67
240	3884090.62	257094.79
270	4458730.2	465084.38
300	4738438.81	302180.66
330	5023495.64	378740.45
360	5125155.56	357104.98
390	5026344.38	361445.84
420	5196335.31	357923.14

Table S3. Peak areas of the carbonyl peak of α-glycine and γ-glycine as reported by the Bruker TopSpin application for the solid-state ^{13}C CP/MAS NMR spectra produced during the time-course experiment shown in Figure S4 (1.70 g glycine).

Timepoint (min)	Peak area of α-glycine carbonyl peak (176.4 ppm)	Peak area of γ-glycine carbonyl peak (174.4 ppm)
0	4676523.48	0
60	5027285.61	72961.75
120	5092796.50	87242.70
180	5027909.84	105934.14
240	4200212.66	79181.17
300	3595371.92	1337447.14
360	3491499.43	1531689.96
420	3540819.05	1718943.42
480	3745675.19	1413316.79
540	2794406.30	2481042.30
600	2450231.25	2349994.70
660	2719772.95	2475404.82
720	2762457.84	2377211.75
780	2473639.38	2013189.38
840	2522982.67	2599728.26
900	1273481.91	1659080.06

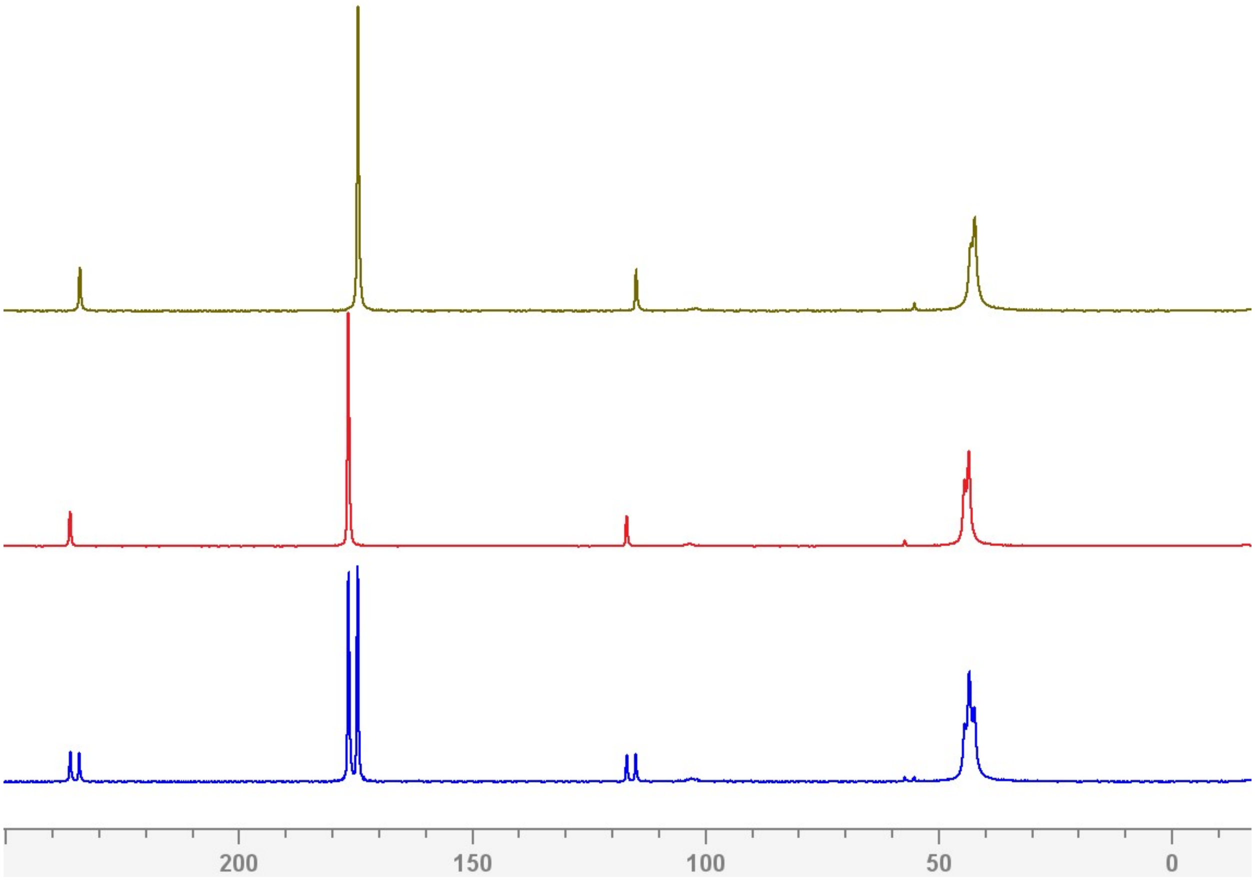

Figure S5. Representative ^{13}C CP/MAS NMR spectra of glycine recorded using a Bruker Avance III 200 NMR spectrometer with a rotor spinning speed of ν_{rot} = 3000 Hz. Top: 100% γ-glycine; middle: 100% α-glycine; bottom: mixture of the two polymorphs. Scale in ppm.