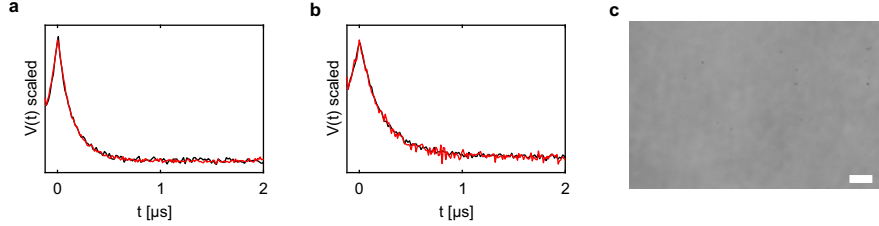


	dispersed			condensed				$T_{0,2}^{(1)}$ [μs]	$T_{0,2}^{(2)}$ [μs]	Λ_0	λ_1	λ_2	η
	$\langle r \rangle_{\text{dis}}$ [Å]	Γ_{dis} [Å]	k_{dis} [μs ⁻¹]	$\langle r \rangle_{\text{cond}}$ [Å]	Γ_{cond} [Å]	k_{cond} [μs ^{-d/3}]	d						
lower	$\langle r \rangle_{\text{low}}$	Γ_{low}	0	10	2	0	2	2.20	2.00	0	0	0	0.468
upper	$\langle r \rangle_{\text{up}}$	Γ_{up}	0.09	80	50	1	4	2.25	2.05	1	1	1	0.572

Supplementary Table 1: Table with overview of parameter ranges employed for the analysis of the biphasic DEER measurements. The parameter range of the mean $\langle r \rangle_{\text{dis}}$ and width Γ_{dis} of the biphasic dispersed state were constrained to the lower and upper 95 % confidence interval (indexed with “low” and “up”, respectively) obtained for the monophasic dispersed state measured at 0.6 M urea concentration (Extended Data Fig. 9c-d). $T_{0,2}^{(1)}$ and $T_{0,2}^{(2)}$ are the refocusing times of the additional modulated dipolar pathway of the first and second 5-pulse DEER trace, respectively. The parameter η corresponds to the fraction of protein in the dispersed phase and was constrained to $\pm 10\%$ of the fraction determined by NMR.



Supplementary Figure 1: Control experiments for the biphasic dispersed state measurement of FUS NTD. Primary DEER data of the FUS NTD mutants a) A10C S29C R1 and b) A105C G128C R1 in the monophasic dispersed state at 0.6 M urea concentration (black) and the supernatant collected after centrifugation of an agarose-free biphasic sample (red) scaled to the same modulation depth. The primary DEER data overlap perfectly confirming our hypothesis that the biphasic dispersed state retains the distance distribution of the monophasic dispersed state. c) FUS NTD under the same buffer conditions as the biphasic sample at 5 μM protein concentration shows no phase separation. Representative image from three independent experiments. Scale bar: 30 μm