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Exploring crystallisation in "no man's land'

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The region in the water's phase diagram called "no-man's land" (160K - 232 K) [1,2] has incited vast research. It is within its depths that the origin of water's anomalous properties is thought to hide behind a veil of crystallisation [3,4]. However, there are other open questions in this region regarding the early stages of crystallisation itself, particularly that of the structure of ice. The main hurdle to overcome has been that water spontaneously crystallises as either bound of "no-man's land" is approached.

We succeeded in navigating this region by applying ultra-fast heating using an infrared laser on amorphous ices, followed by femtosecond X-ray scattering measurements at different delay times. After the observation of a liquid-liquid transition[5], the curtain fell and crystallisation started, allowing us to probe the early stages of crystallisation[6]. We observed that the crystallising phase is stacking disordered ice (Isd) with a high cubicity which decreased over time. We note that a growing small portion of hexagonal ice (Ih) was also present, suggesting that already within our timeframe, Isd starts annealing into Ih.

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