Graphene oxide (GO) and reduced graphene oxide (rGO) are important materials in a wide range of fields. The modified Hummers methods, for synthesizing GO, and subsequent thermal reduction to rGO, are often employed for production of rGO. However, the mechanism behinds these syntheses methods are still unclear. We present an in situ X-ray diffraction study of the synthesis of GO and thermal reduction of GO. The X-ray diffraction revealed that the Hummers method includes an intercalation state and finally formation of additional crystalline material. The formation of GO is observed during both the intercalation and the crystallization stage. During thermal reduction of GO three stages were observed: GO, a disordered stage, and the rGO stage. The appearance of these stages depends on the heating ramp. The aim of this study is to provide deeper insight into the chemical and physical processes during the syntheses.

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