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**CRYSTALLINITY OF ANNEALED NAFION MEMBRANES STUDIED BY SAXS AND AFM: EFFECT ON PEFC PERFORMANCE**

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The relationship between electrical and morphological properties of annealed Nafion samples is investigated by X-ray diffraction (XRD), small angle X-ray scattering (SAXS), atomic force microscopy (AFM), and impedance spectroscopy. The experimental results reveal that membranes submitted to a heat treatment at high temperature ( $T \sim 130 - 140^\circ\text{C}$ ) and low relative humidity (RH), display higher crystallinity and irreversibly shrunk hydrophilic domains. Such an effect was not observed in samples heat treated in the same temperature range but at high RH. The increase in crystallinity and shrinking of the hydrophilic domains reduce the ionomer conductivity. These features were reflected in the polymer electrolyte fuel cell (PEFC) tests in which the measured performance was lower for Nafion samples annealed at low RH and high T than that of samples annealed at high RH. Such results are relevant for understanding the limitations of Nafion membranes at harsh PEFC operating conditions as well as for the development of high-performance ionomer membranes.