Cryomilled aluminum alloy parts have recently generated interest as a method to produce nanocrystalline, high strength, and low density structural parts. Degassing is a critical step in the manufacturing sequence of cryomilled aluminum powder in order to remove volatiles (primarily moisture) entrapped in the powders. For cryomilled aluminum alloy powders, degassing subjects the powder to elevated temperatures for extended durations causing microstructural and chemical changes such as removal of volatiles, grain growth, and precipitation of dispersoids. In this work, the effect of degassing temperature on the grain size, dispersoid precipitation and impurity concentrations of nanocrystalline AA5083 was explored. Cryomilled AA5083 powders were degassed at 200°C, 300°C, 350°C, 410°C and 500°C at a ramp rate of 68.3°C/hr for a soak time of 8 hours at 2·10⁻⁵ torr. Grain size was determined through both X-ray diffraction (XRD) and transmission electron microscopy (TEM). Dispersoids were examined by XRD, scanning electron microscopy (SEM) and TEM equipped with high angle annular dark field (HAADF) detector and energy dispersive spectroscopy (EDS). Inert gas fusion and thermal conductivity analysis was conducted to determine the oxygen, nitrogen and hydrogen concentrations as a function of degassing temperature. Al₆(MnFeCr) was the largest dispersoid formed and was observed to precipitate on all samples degassed at or above 300°C. Hydrogen concentration was strongly influenced by degassing temperature for an 8 hour soak. Chemical modeling of the degassing process was conducted through the fitting of Crank’s kinetic model for the degassing of a sphere.