Subwavelength-scale nanostructures hold great promise for the development of new, useful optical devices. One of the challenges in realizing such devices is the creation of the desired nanoscale patterns in materials such as silicon with the required precision. Recently, a new resist material for electron-beam lithography, Hydrogen Silsesquioxane (HSQ), has emerged as a solution to this challenge. The purpose of this project is to create a recipe for a 100 nm layer of HSQ to be applied to a silicon substrate by means of spin-coating. After formulation of a recipe to achieve the desired 100 nm thickness, tests were performed to determine which electron beam dosages produced the best features in terms of resolution and contrast. Grating patterns were created with varying periods while maintaining a constant 50% fill factor. Characterization of the gratings was performed to determine the optimal dosage for these features. The optimal dose was found to be 95 \( \mu \text{C/cm}^2 \). After determining an ideal recipe, the HSQ was used as an etch mask to create hybrid HSQ-silicon gratings with nanoscale features. This capability will enable the development of a number of new optical devices based on nanostructures, for a range of interdisciplinary applications.