We observed charge relaxation processes directly in separation process after contact charging with various plastic films. In experiments, a plastic thin film was fixed on a stainless steel specimen support, then the film was made contact with a mirror polished stainless plate. Then the specimen support separated by a piezo drive at a separation speed of 100 µm/s. When the separation distance reached 100 µm gap, the film approached-contacted at the same speed again. Behind the film, the electrostatic potential of the specimen support was recorded with an ultra-high input impedance surface voltage meter (Trec MODEL 820). In addition, the separation distance of the film and the stainless plate was measured with a high precision capacitance-type displacement sensor. As separated, due to the reduction of capacitance between the film and target plate, the surface potential difference increased. Occasionally, sometimes, sharp partial drops were observed. This is due to gas discharge and charge relaxation. For each potential drop the amount of charge relaxation was small, which means that the potential did not reach to null even after such gas discharge and the surface potential increased again with residual charge after the relaxation. Even a few times of the potential drop were observed in one separation process. This kind of potential drop was not observed in the re-approach process after separation. The discharges seemed to take place randomly, but after an accumulation of such data with multiple measurements, the starting points of discharges were consistent as a linear relationship between surface potential and gap. In this work, several kinds of PFA, with different surface resistance maintained with additives, and Nylon films were used as sample. Results showed that films with surface resistance less than $10^{12}$ Ohm did not receive enough charge to relax (gas discharge did not take place). In the case of some PFA samples showed gas discharge, the discharge property (discharge limit potential as a function of gap) for the samples were consistent for all the samples.