The doctoral thesis deals with an experimental study of several diagnostic techniques intended for plasma diagnostics and diagnostics of thin films during reactive sputter deposition. A relatively novel probe diagnostic technique called Floating harmonic probe for measurement of the ion density and electron temperature in technological low-temperature plasma is studied. A Langmuir probe is commonly used, however, its application in conditions where non-conducting films are being deposited can be problematic or unreliable. The floating harmonic probe measurement technique deals with this inherent problem of the Langmuir probe. The Floating harmonic probe results are compared to those obtained by a classical Langmuir probe in non-reactive DC continuously driven discharge, and its applicability in reactive regime during deposition of iron oxide thin films is proved. The work deals also with a modification of the Floating harmonic probe called Phase Delay Harmonic Analysis Method which is intended for diagnostics of pulsed driven discharges. The second part of the thesis is devoted to a new proposed method for in-situ diagnostics of thin films. This method monitors the capacitance and resistance of a thin film during deposition up to the frequencies in the kHz range. This new method could be used for diagnostics of dielectric properties of thin films or could be suitable for studying or control of reactive deposition processes.