The subject of this thesis is an electrodeposition of Ni-W alloy and characterization of microstructure and properties of the deposits. In Chapter 3 background such as theoretical comments and literature reviews which provided suggestions for the way to tackle this subject, is described. The experimental results of the present work are given in the chapters 4-9. In Chapter 4 development of a new electrolyte for Ni-W alloys is described. In the chapters 5-9 the properties of the Ni-W alloys such as residual stress, microstructure, hardness and thermal stability are investigated. Furthermore, a paper on the fabrication of forming tool by electroforming of nickel based alloys is added as an appendix to this thesis. This serves as an example of an application of the material developed in this work. Details of the experimental procedures and the theories of unusual techniques such as cross-section observation with focused ion beam microscopy, compositional analysis with glow discharge optical emission spectroscopy and line broadening analysis of X-ray diffraction etc are given in Chapter 3. In Chapter 4 the effect of the complexing agents citrate, glycine and triethanolamine (TEA) on the electrodeposition of Ni-W layers from electrolytes based on NiSO\textsubscript{4} and Na\textsubscript{2}WO\textsubscript{4}, is investigated. High W content and current efficiency could be realized by using electrolytes containing all of the three complexing agents. The results show that small amounts of glycine in a citrate-triethanolamine based electrolyte positively influence fractional current for Ni and Ni-W alloy deposition. In Chapter 5 the residual stress in Ni-W layers electrodeposited from electrolytes based on NiSO\textsubscript{4} and Na\textsubscript{2}WO\textsubscript{4}, is investigated. The results show that the type of complexing agent and the current efficiency have an influence on the residual stress. In all cases, an increase in tensile stress in the deposit with time after deposition was observed. The increment depends on the applied current density. In Chapter 6 Ni-W layers electrodeposited from electrolytes based on NiSO\textsubscript{4}, Na\textsubscript{2}WO\textsubscript{4} citrate, glycine and triethanolamine are characterized with glow discharge optical emission spectroscopy (GD-OES) and X-ray diffraction analysis (XRD). XRD showed the occurrence of an anomalous phase in the deposits, associated with the presence of an appreciable amount of carbon as identified with GD-OES. The anomalous phase is metastable and vanishes upon annealing at 550°C in air. In Chapter 7 the influence of the age of an electrolyte, containing glycine as complexing agent, on the microstructure of a Ni-W electrodeposits is investigated. The microstructure of the electrodeposits was characterized with scanning electron microscopy (SEM), X-ray diffraction (XRD) analysis and glow discharge optical emission spectroscopy (GD-OES). It was observed that an anomalous Bragg peak at 2θ=41.5° occurs for layers deposited from the aged electrolyte and not for layers deposited from the fresh electrolyte; the intensity of the Bragg peak increases with the age of the electrolyte. Simultaneously, the presence of carbon is observed with GDOES in layers deposited from the aged electrolyte. The carbon dissolution in the Ni-W alloy deposit is associated with the formation of a new phase in the electrodeposited, giving rise to the anomalous Bragg peak. In Chapter 8 hardness, grain size and thermal stability of nickel and Ni-W alloy layers deposited from electrolytes containing equal amounts of citrate, glycine and triethanolamine are investigated. The hardness of the deposits was investigated in the as-deposited layer as well as after annealing for 1 hour at temperatures up to 550°C. The grain size and microstrains were determined for several crystal orientations by pseudo-Voigt single line analysis of the corresponding X-ray line profiles. The micro Vickers hardness of the nickel deposits depends on the thermal history of the sample. Depending on the microstructure and composition of the electrodeposits hardness increases or decreases with annealing temperature (for 1 hour). The results are discussed in terms of the possible strengthening mechanisms for nano-crystalline electrodeposits. In Chapter 9 the evolution of residual stress in Ni-W layers electrodeposited from electrolytes based on NiSO\textsubscript{4} and Na\textsubscript{2}WO\textsubscript{4} upon storage at room temperature is investigated. An increase in tensile stress in the Ni-W electrodeposited upon storage at room temperature was observed; the increase in tensile residual stress is larger for higher the current densities during electrodeposition. For one current density the electrical resistivity of a Ni-W layer was measured as a function of storage time. The evolution of stress increase and reduction of electrical resistivity are equal for the first three hours.