Abstract: The correlation of vacancy concentration with microhardness of Fe-Al alloys was studied on samples quenched from 1000 °C and subsequently annealed at lower temperatures. Using X-ray diffraction the lattice parameter and crystal structure were determined for samples of Fe-Al alloys. By measurements of positron lifetime was revealed the high concentration of vacancies in quenched samples and subsequent annealing caused significant decrease in vacancy concentration while in samples with Al content above 39% also the decrease of microhardness was measured. Measurements of coincidence Doppler broadening of annihilation peak helped to distinguish the annihilations coming from positron trapped or delocalized annihilated by electrons of both atoms, Fe and Al. Comparison of measured results with theoretical quantum-mechanics calculations performed in this diploma thesis determined the most probable defect type as a vacancy on A-sublatice of B2 structure.