Niobium films on the single crystal substrate have been studied. The films of thicknesses less than 5 nm, 5 nm, 15 nm and 400 nm were deposited onto sapphire (001) surface by mean of the cathodic arc in UHV conditions [1-3]. Energy of incoming Nb$^{3+}$ ions in the range of tens electronvolts and substrate temperature about 200°C are distinctive characteristics of that deposition method. The aim of performed XRD studies was to recognize the structural forms of Nb film formed in the early stages of growth. The measurements were preformed at the wiggler beamline W1 using the photon energy 8048 eV corresponding to the wavelength $\lambda = 0.15406$ nm (Cu K$_{\alpha 1}$ characteristic line).

Fig. 1. X-ray $\omega$-2$\theta$ patterns (1), and 2$\theta$ scans in grazing incidence geometry with incidence angles 1°(2) and 5° (3), respectively, for the samples of thickness less than 5 nm, 15 nm, and 400 nm.

Sharp maxima observed in diffraction patterns in Fig. 1 originate from the single crystal sapphire substrate. The thinnest film shows only one niobium originated feature in $\omega$-2$\theta$ pattern. This is a broad diffraction profile at $2\theta \approx 35.5^\circ$, what is very close to the position of 110 diffraction line of the Nb. No diffraction lines corresponding to the Nb were observed for
that film in the grazing incidence scan. It indicates that the Nb film is very thin and ordered so as the (110) lattice planes are parallel to the substrate surface.

A shape of the feature observed in the angular position corresponding to Nb 110 maximum depends on the film thickness (Fig. 2). It results from the superposition of Nb 110 reflections originating from two different forms of Nb. The broaden one described above, and a narrow one. The narrow peak appears for the films having a thickness larger than 15 nm. This is the Nb 110 reflection typical for a bulk niobium. Although bulk–like reflection appears in ω-2θ pattern, it is absent in grazing incidence one. That indicates the preferred orientation of large crystallites the same as the orientation of early grown layer. This observation points to a thickness of 15 nm as that, where the large crystallites start to grow. In Fig. 2 the upper pattern shows that, for a thick film, phases containing small and large crystallites exist together in oppose to two the thinnest films. For a 400 nm thick sample, large crystallites phase is clearly depicted in the diffraction patterns. The grazing incidence patterns for this sample show all reflections allowed for Nb what indicates on the appearance of thin, polycrystalline Nb layer.

According to our studies, the growth of Nb layer is realized in three stages: the first one is the growth of very thin and preferably (110)-oriented layer. In the next stage an increasing of the layer thickness lead to the almost single crystal layer with (110) lattice planes parallel to the substrate surface. In the last stage, observed for the thickest layer, the appearance of thin polycrystalline Nb layer, probably on the surface, have been observed.

References