Highlighting of a crystalline and amorphous TiO₂ bilayer by Transmission Electron Microscope investigations: conventional TEM coupled with EDX and ASTAR mappings

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TiO₂ has been deposited on TiNi superelastic substrate in order to prevent any Ni diffusion for its further potential use in orthodontics. TiO₂ has been synthetized by a sol-gel technique; then a thermal treatment has been applied in order to partially crystallize the thin film. The objective of this work is to put in evidence if the TiO₂ film contains amorphous and crystalline parts. For this purpose, the material has been characterized by TEM microscopy on a transversal cross-section thin lamella priory prepared by double column FIB-SEM microscopy. The identification of the crystalline and amorphous regions in the thin film is based on EDX and ASTAR mappings together with TEM observations and Virtual Dark-Field images (reconstructed from electron diffraction patterns). These observations permit to put in evidence that the TiO₂ thin film is indeed composed of a crystalline layer, located towards the substrate ~50 nm thick, and of an amorphous and porous superior layer ~50 nm thick. The crystalline TiO₂ layer presents both anatase and rutile structure with very small grains. This peculiar microstructure, both crystalline and amorphous, permits to improve the mechanical properties of the thin film.