Transmission electron microscope measurement of the hydroxyapatite layers coated by droplets eliminated type pulsed-laser deposition scheme

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The high-crystalline hydroxyapatite layer was coated by a droplet-eliminated pulsed-laser deposition scheme on zirconia substrates [1]. Raman spectroscopy measurement showed that the coating layer changed drastically from amorphous to crystalline phase at annealing temperature Ta= 360 °C. Moreover, narrowing and spectral upshift of the Raman spectral line over the threshold indicated the increase of the crystallite size with increase Ta. An asymmetrical shape of the Raman spectral line just over the threshold also indicated the evidence came from the nm-size crystals [2]. This same asymmetric spectral shape was reported concern about the nano crystalline Si, diamond, Ge, etc. A transmission electron microscope (TEM) can observe the nm-size image and evaluate the crystal phase from the electron diffraction measurements.

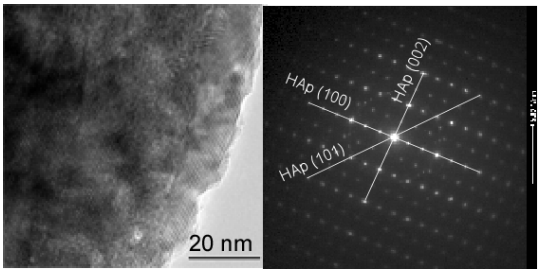

Figure 1 HR-TEM image of coating layer and electron diffraction pattern for Ta= 400 °C.

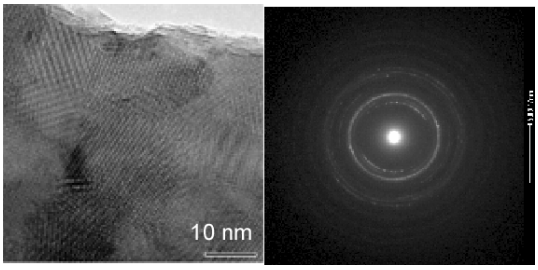

Figure 2 HR-TEM image of coating layer and electron diffraction pattern for Ta= 550 °C.

The coating layers for different Ta were scratched in ethanol and their grains were observed by TEM. Typical TEM images of the scratched grains and their electron diffraction patterns for Ta= 400 and 550 °C were shown in Fig. 1 and Fig. 2, respectively. The diameter of the electron transmission was set at 0.8 μm. These patterns showed that the coating layer shown in Fig. 1 is single-crystal with larger crystallite size, on the other hand, the coating layer shown in Fig. 2 became a polycrystal with smaller crystalline size. These differences might be caused by the growth rate of the crystal layer at different Ta. The disagreement on the crystallite size estimation between Raman spectroscopy and TEM analysis is under consideration.

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[2] I.H. Campbell and P.M. Fauchet, Solid State Comm. **58**, 10 739-741 (1986).