High resolution transmission electron microscopy study of a nanocrystalline layer on the surface of biomedical CoCrMo

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With its superior wear and corrosion resistance, CoCrMo alloys have been widely used for metal-on-metal total hip replacements (THRs). However, the use of the metal-on-metal implants has recently been seriously called into question and several alerts have been issued by the Medicines and Healthcare products Regulatory Agency (MHRA) due to concern on metal ion release [1]. Most recently, Smith et al [2] analyzed the data from the National joint Registry of England and Wales and found failure rates of metal-on-metal THRs are much higher than other bearings, such as metal-on-polymer and ceramic-on-ceramic THRs. They recommend that metal-on-metal bearing surfaces should not be used in stemmed THRs. Giving the fact that some of the first generation of metal-on-metal THRs lasted for more than 20 years [3], it is important to understand wear of metal-on-metal THRs, hence to reduce the wear rate and metal ion dissolution. A nanocrystalline layer has been reported on the topmost surface of both in vivo and in vitro CoCrMo THRs [4, 5] and the understanding of this nanocrystalline layer could be essential to improve the material. The current work provides a detailed study of sub-surface damage of biomedical CoCrMo after reciprocating wear test, including high resolution transmission electron microscopy (HRTEM) of the nanocrystalline layer.

CoCrMo samples were obtained from Smith & Nephew as high carbon as-cast CoCrMo (F75). Prior to wear testing, CoCrMo samples were mechanically polished. Reciprocating wear test was performed using a ball-on-disc apparatus UMT tribometer (Centre for Tribology Inc., USA). Testing was carried out in bovine serum under test conditions of 4N load, 0.2 m/s sliding speed, 10 mm distance against an alumina ball counterpart (4 mm diameter). Site-specific TEM cross-section samples along the wear track and from the as-polished surface were prepared by focused ion beam (FIB) in situ lift-out method (Quanta 200 3D with Ominprobe, FEI, the Netherlands). Carbon deposition was applied on the region of interest to prevent Ga⁺ implantation and sputter erosion of the top portion of the surface. Routine bright field TEM was performed on a Tecnai 20 (FEI, the Netherlands) operating at 200kV, while HRTEM was undertaken on a Jeol 2010F (Jeol, Japan) operating at 200kV.

Bright field TEM image of a longitude cross-section from 4N reciprocating wear track shows a fine nanocrystalline layer below the surface of thickness ~300nm, Fig.1a, on top of a structure showing general deformation (Fig.1b). The nanocrystalline layer was not observed in TEM cross-section samples from the as-polished surface using the same sample preparation technique, demonstrating that the nanocrystalline layer was formed during sliding contact. Fig.1b shows a deformed structure comprising strongly textured plates associated with the formation of ε-martensite. The same feature was observed in the as-polished surface, but the depth of this layer was greater in the worn sample and the density of the ε-martensite plates was greater than for the mechanically polished sample.

HRTEM images of the nanocrystalline layer just below the surface and in the centre of the layer are shown in Fig.2a and Fig.2b, respectively. The size of nanocrystallites was in the range of 5nm. FFTs of the HREM images indicated a predominantly hcp structure, although analysis also indicated the possibility of fcc phase (i.e. the parent CoCr structure) also present. Thus, it is suggested that the nanocrystalline layer forms through some high strain shear process from the prior ε-martensite structure.
References


**Figure 1.** (a) Bright field TEM image of longitude TEM cross-section from 4N reciprocating wear track shows a layer of nanocrystallines on the topmost surface, as indicated. (b) Magnified image shows textured plates on the cross-section.

**Figure 2.** HRTEM images of the nanocrystalline layer just below the surface (a) and in the centre of the layer (b). Squares indicate the region thin enough to enable lattice fringe imaging.