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Very Fine-Grained Cu-0.4Mg Alloy Improving Intrauterine Device

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The non-hormonal copper-containing intrauterine device (Cu-IUD) is one of the most widely used contraceptives for human birth control [1] due to its safety, high effectiveness, affordability, and reversibility [2]. However, the burst release of cupric ions (Cu^{2+}) in the first few days of implantation prevents the continuation of the Cu^{2+} release in the conventional coarse-grained (CG) Cu-IUD. To maintain constant release of Cu^{2+} in the uterine over the lifespan of the Cu-IUD and to improve cell and tissue biocompatibility, a newly designed very fine-grained (VFG) Cu-0.4Mg alloy was developed and tested. The purpose of this study is to investigate the microstructure of very fine-grained Cu-0.4Mg alloy in the role of suppressing the burst release of Cu^{2+} in Cu-IUD using electron microscopy.

To investigate the corrosion behavior of the Cu and hence the release rate of Cu^{2+} in the intrauterine device, two kinds of Cu containing materials, pure CG Cu rods (99.99%) and VFG Cu-0.4Mg rods, were selected for this study. While pure CG Cu rods (99.99%) were purchased from a commercial company, the VFG Cu-0.4Mg rods were laboratory fabricated via 8-pass equal channel angular pressing (ECAP) method. Thin discs cut from these two Cu-containing rods were mechanically ground and polished. TEM specimen was prepared by further thinning a pre-thinned disc using a FIB (Tescan GAIA3) by *in-situ* lift-out technique whereas a pre-thinned discs was further polished using a broad ion beam (Gatan PECS II) for electron backscatter diffraction (EBSD) study. The TEM study was carried out using a JEOL 2100F TEM/STEM equipped with Bruker EDS and Gatan EELS/GIF systems at the accelerating voltage of 200 kV. A ThermoFisher (FEI) Helios G4 PFIB UXe DualBeam FIB/SEM equipped with an EBSD analyzer "CHANNEL 5" was employed for EBSD investigation.

TEM micrographs show a large grain-size difference between pure CG Cu and VFG Cu-0.4Mg alloy (Fig. 1). TEM images of CG Cu depicted heterogeneous grain size, ranging from 1 to 20 μm with random orientations. Many bend contours due to the bending/bulking of those very large Cu grains can easily be observed (Fig. 1a). On the other hand, the VFG Cu-0.4Mg alloy after 8-pass ECAP revealed elongated and relatively uniform grains with an average size of 300 nm that are preferentially oriented along the extrusion direction as compared to CG Cu (Fig. 1b). Electron diffraction analysis showed that angles between (020) and (200), and (200) and (220) in [001] zone axis were 45° each in CG Cu which is typical for the pure Cu, while 45.3° and 44.9°, respectively in VFG Cu-0.4Mg alloy. The very small angles change between (020), (200), and (220) in [001] zone axis of VFG Cu-0.4 Mg alloy indicate lattice distortion that might have resulted from the effect of ECAP extrusion and/or the additional Mg in the alloy. EBSD results were consistent with the data obtained from the TEM study.

To understand the release behaviors of the metallic ions in these two Cu materials, long term immersion tests in simulated uterine fluid (SUF) up to 300 days were conducted (Fig. 2). Both CG Cu and VFG Cu-0.4 Mg alloy exhibited a rapid Cu^{2+} release within the first month followed by a steady release rate after 50 days' immersion. Despite the high release rate of Cu^{2+} (76.63 μg/day) in VFG Cu-0.4Mg on the 1st day, the release rate after 30 days' immersion remained rather constant at 15.88 μg/day. This suggests that the addition of the more electrochemically active alloy element Mg with the high fraction grain boundary in VFG Cu-0.4 alloy might inhibit the release rate of Cu^{2+}. In addition, Mg in VFG Cu-0.4Mg might also play an important role in enhancing the biocompatibility and comfort of the Cu-IUD adoption [3].

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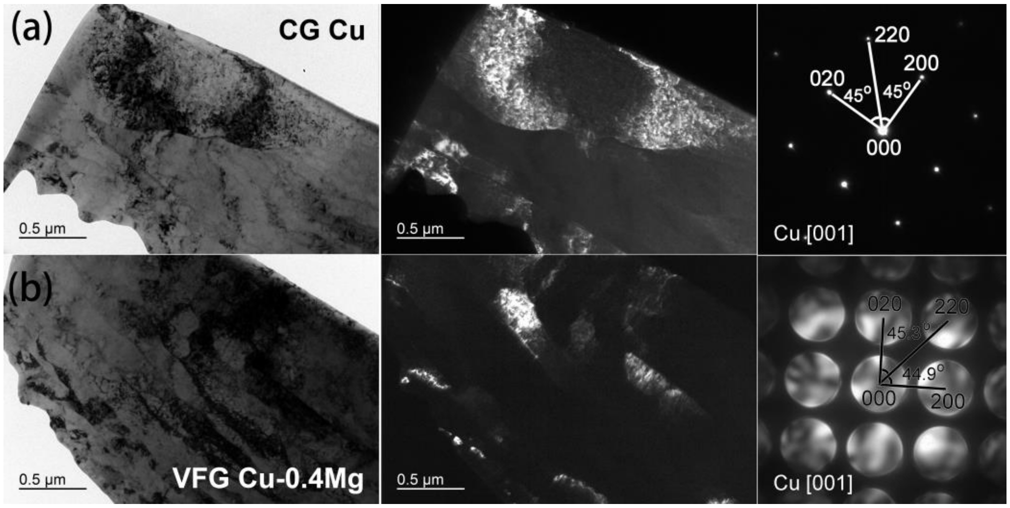

Figure 1. (a) TEM bright-/dark-field (left/central) micrographs, and SAD pattern (right) of CG Cu showing heterogeneous and randomly distributed large grain size with a typical FCC diffraction pattern. Note grains are so large that reveals bending/buckling as shown by bend contours. (b) TEM bright-/dark-field (left/central) micrographs, and a micro-diffraction pattern of VFG-0.4 Mg alloy revealing preferentially oriented very fine-grained materials with large amounts of defects after 8-pass ECAP extrusion.

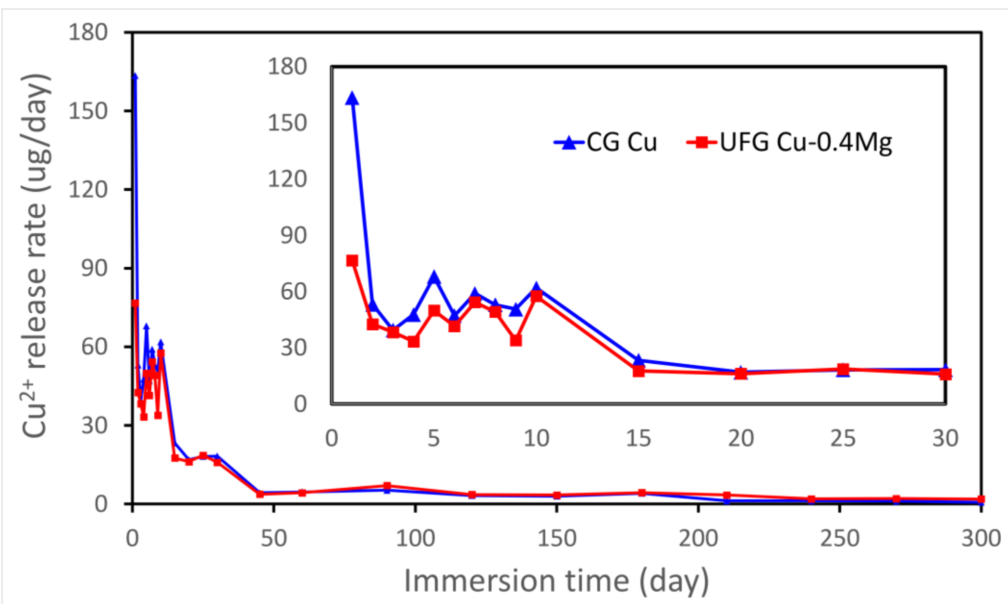

Figure 2. Comparison of cupric ion release rate of coarse-grained Cu and ultrafine-grained Cu-0.4Mg alloy incubated in SUF at 37 °C for 300 days.

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