Morphological properties of TiO_2 / $Fe_{50}Co_{50}$ composite films

V.S.K. Chakravadhanula[1], Amit Kulkarni[2], A. Lotnyk[1], V. Duppel[3], V. Zaporotchenko[2], L. Kienle[1], and F. Faupel[2]

1. Faculty of Engineering, Institute for Material Science, Synthesis and Real Structure, Christian Albrechts University of Kiel, Kaiserstr. 2, D-24143 Kiel, Germany
2. Faculty of Engineering, Institute for Material Science, Chair for Multicomponent Materials, Christian Albrechts University of Kiel, Kaiserstr. 2, D-24143 Kiel, Germany
3. Max Planck Institute for Solid State Research, Heisenbergstr. 1, D-70569 Stuttgart, Germany

cv@tf.uni-kiel.de
Keywords: nanocomposites, HRTEM, in-situ heating, cosputtering, FeCo

The metal-dielectric nanocomposites are of extensive research interest which enable them to be the potential building blocks for different miniaturized devices. Composite films of TiO_2 as an insulator and iron cobalt based ferromagnetic components with different metal volume fractions (MVF) were prepared by co-sputtering [1].

High resolution transmission electron microscopy (HRTEM) analysis reveals that the structural ordering in the TiO_2/FeCo nanocomposites depends on the MVF. Amorphous nanocomposites were formed for lower MVF whereas crystallites were present at higher MVF. HRTEM analysis was done with a Philips CM 30ST microscope (LaB_6 cathode, 300 kV, C_S = 1.15 mm). SAED (selected area electron diffraction) was carried out using a diaphragm which limited the diffraction to a circular area of 2500 Å in diameter. All images were recorded with a Gatan Multiscan CCD camera and evaluated (including Fourier filtering) with the programs Digital Micrograph 3.6.1 (Gatan) or Crisp (Calidris). Chemical analyses by EDX were performed in the nanoprobe mode or by spectral imaging (scanning mode) with a Si/Li detector (Noran, Vantage System). Heating experiments were performed in-situ within a Tecnai F30 G^2 by using a Double-tilt Tantalum heating stage holder (Gatan GmbH).

In the case of nanocomposites with lower MVF, according to SAED patterns, all components are fully amorphous in both the freshly prepared films and aged films which were at least partially oxidized, cf. Figure 1 (a) and (b). The diffracted intensity is on concentric circles; however, intensity for d ~ 2.0 Å is strongly excited correlating with the high-intensity peak of FeCo (110, [2]). Note that such intensity varies strongly when transmitting distinct regions of the films, thus suggesting the presence of distinct amounts of amorphous FeCo clusters inside the selected areas.

In aged films, the SAED pattern of Figure 1b, contains only very broad diffuse intensity concentrating on a concentric circle with the diameter of 2.7 Å. This value corresponds with the high-intensity peak (104, [3]) of an ilmenite-type structure. The most intense peak expected for amorphous TiO_2 is not excited thus supporting the presence of a quaternary ceramic main component.

In both freshly prepared and aged samples, EDX analyses indicates an equiatomic ratio of Fe : Co in all cases. In contrast to the aged films, the ratio O/Ti scatters strongly but is preferably strongly reduced with average values O : Ti ~ 1.8 – 2.5 in the freshly prepared films. Hence, the chemical nature of the freshly prepared films corresponds to a composite structure made of amorphous FeCo and TiO_2. In aged films, the atomic ratio of O : Ti was significantly larger than the expected value of 2.0. Typical values measured on central parts of the films are in the range of O : Ti ~ 3.3 – 3.8 due to a reduced relative content of Ti.

DOI: 10.3217/978-3-85125-062-6-447 ,

Hence, Fe and Co must be incorporated in an oxidized form inside the material. Assuming Fe and Co in tri- and bivalent states, the criterion of charge balance is approximately met by the determined average composition which is close to $Co_{0.56}Fe_{0.56}Ti_{0.8}O_3$.

At a temperature of 450 °C of the heating stage a separation of the bright field contrast by the formation of nanosized crystallites with strong diffraction contrast was observed (Figure 2). Furthermore, particles with less crystalline perfection start to segregate see arrow in Figure 2. The stripes in bright-field contrast frequently indicate the presence of multiple grains and crystal defects (Figure 2(b)). These crystallites are clearly facetted (Figure 2(c)) and SAED patterns confirm their highly crystalline state. The pattern of Figure 3(a) was recorded on a single-grain nanocrystallite along zone axis [110]. The d-values of d(111) = 2.05 Å and d(200) = 1.78 Å correlate well with reference values [4] of 2.046 Å and 1.772 Å for the cubic high temperature modification of cobalt. The arrangement of the grains frequently appears random thus producing diffracted intensity on concentric circles (Figure 3(b)), with the diameters expected for cubic cobalt (d(111) = 2.05 Å; d(200) = 1.78 Å, d(220) = 1.26 Å, d(311) =1.07 Å). Also, HRTEM studies suggest the local formation of TiO_2 species, e.g. anatase (d(101) = 3.566 Å).

1. F. Faupel et al., Contrib. Plasma Phys. 47, No. 7, (2007) p537.
2 P. Bayliss, Trans. Am. Soc. Met. **29**, (1941) p415.
3. J.M. Newsam, Y. Endoh, Y. Ishikawa, H. Takei, J. of Physics C **19**, (1986) p1273.
4. E.A Owen, D.Madoc Jones, **38**, (1992), p127.

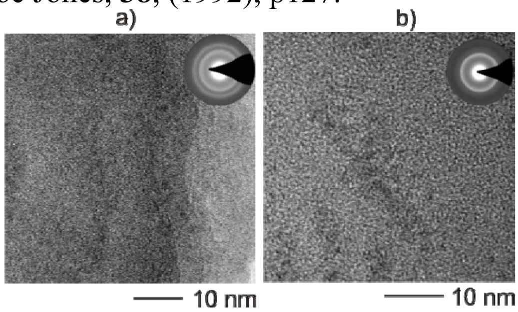

Figure 1. SAED pattern (inset) and bright-field images of an amorphous film TiO_2 : FeCo (a), and an amorphous and ceramic film(b).

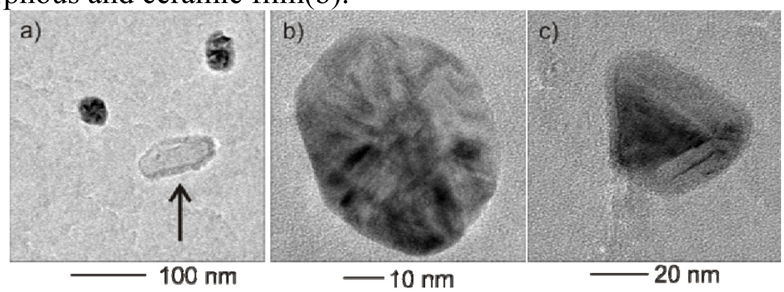

Figure 2. Microstructure of the initially amorphous film TiO_2 : FeCo after in-situ heating with enlarged sections of single Co nanocrystals (b, c).

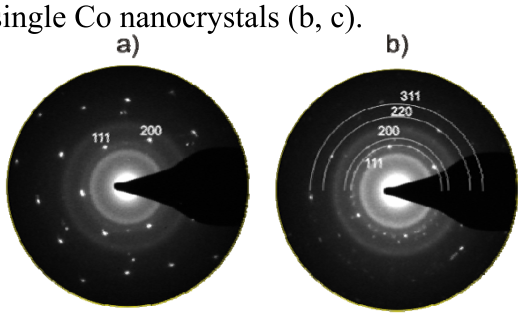

Figure 3. SAED patterns recorded on a single grain nanocrystal, (a), [110]) and on a multiple grain particle of fcc-cobalt.

DOI: 10.3217/978-3-85125-062-6-447 , © Verlag der TU Graz 2009