s10.m2.o3 Evidence from HRTEM Image Processing Analysis of Nanocrystalline Iron-Titanium Oxide Powders. A.M. Tonejc, I. Djerdj and A. Tonejc, *Faculty of Science, Department of Physics, Bijenicka 32, 10002 Zagreb, Croatia.*
Keywords: nanosized TiO_2; HRTEM; Fourier filtered images.

In the present work we investigated nanosized iron-doped TiO_2 with Ti/Fe ratio=1:0.15 synthesised by modified sol-gel method[1]. In the previous work[2] those samples were analysed by transmission electron microscopy (TEM), selected area electron diffraction (SAED) high-resolution electron microscopy (HRTEM), SEM, energy dispersive X-ray analysis (EDS) and X-ray diffraction (XRD). HRTEM was performed by using JEOL JEM 2010 200 kV microscope, having point resolution of 0.19 nm.

Here we report on the image processing analysis of HRTEM photographs. According to XRD in as prepared sample (S1) very broad diffraction maxima of anatase as the dominant phase were observed. XRD showed additional maxima that were ascribed to PEG. In HRTEM image, the material appeared very homogeneous with respect to the distribution of grains and pores having average sizes of 3.3 nm and 3.4 nm, respectively[2]. In the HRTEM, presence of PEG as the lattice image was not observed. The SAED pattern reveal nanocrystalline rings of anatase while PEG was barely observed. In spite of this, in the Fourier transform (FT) of HRTEM image the PEG spots were indexed. The obtained Fourier filtered images reveal lattice images of PEG and anatase that constitute the observed grain. The same planes filtered from HRTEM image were observed in XRD pattern of as prepared sample (S1).

XRD pattern of annealed sample (S2) contained diffraction lines of anatase as the dominant phase, faint lines of rutile and brookite. PEG was not observed. SAED patterns and FT of HRTEM images revealed except anatase, rutile, brookite, as well as Fe_2O_3 maghemite-q that precipitated during thermal treatment of S1. In both samples an amorphous phase was observed. Fourier filtering reveals at the atomic level the intergrown planes, indexed in SAED and identified in corresponding regions of nanocristalline grains and grain boundaries.

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[2] Tonejc A.M., Djerdj I., Gotic M., Tudja M., Popovic S., Tonejc A., Music S. "TEM, HRTEM, SEM and X-ray diffraction study of iron doped TiO_2.", Proc. 4th Multinac. Congress on Elect. Microscopy, Veszprem, Hungary, (1999), 375-376.

s10.m2.o4 Residual Stress Analysis of Thin Films. J.-D. Kamminga[a], M. Leoni[b], L.J. Seijbel[a], U. Welzel[b], P. Lamparter[b] and E.J. Mittemeijer[a,b]. [a] *Laboratory of Materials Science, Rotterdamseweg 137, 2628 AL Delft, The Netherlands.* [b] *Max Planck Institute for Metals Research, Seestrasse 192, 70174 Stuttgart, Germany.*
Keywords: thin layers, stress, grain interaction.

Diffraction is widely used to measure stresses in polycrystalline materials. Commonly, the so-called $\sin^2\psi$ method is applied to interpret the lattice strains measured by diffraction in terms of stress in the material.

Recently, new methods have been proposed for the calculation of the stress from the lattice strains. These methods use the mechanical elastic constants, instead of the so-called diffraction elastic constants, that are generally used for the $\sin^2\psi$ analysis. Due to the relatively large accuracy at which the mechanical elastic constants can be calculated from the single crystal elastic constants, the accuracy of the stress obtained on this basis is considerably increased.

When *thin films* are considered the above methods may fail, because a prerequisite for these methods - i.e. macroscopic isotropy - does not occur. For example, a fiber texture or a columnar microstructure violates macroscopic isotropy.

For layers with a columnar microstructure, a method for diffraction stress analysis has been proposed based on grain interaction parameters that suit the columnar microstructure well. Assuming *(i)* the *stress* components normal to the specimen surface to be zero for each crystallite and *(ii)* a biaxial state of *strain* equal for each crystallite, the diffraction responses have been calculated for both randomly textured layers, and layers with a fiber texture. The stress can then be straightforwardly obtained.

In this work, experimental results will be presented for both layers with a random and layers with a fiber texture. The stress in the various layers will be determined using methods of stress analysis appropriate to the microstructure of the particular layer investigated. The first evidence of direction dependent grain interaction will be presented.