

MS26 P01

Magnetite and Native Gold Nanoparticles From a Submarine –Hydrothermal Source Associated to the Peña Colorada Iron-Ores, Mexico. Alva-Valdivia, L. M.^{a,c}, Rivas-Sánchez, M. L.^a, Arenas-Alatorre, J.^b, Perrin, M.^c ^aLaboratorio de Paleomagnetismo, Instituto de Geofísica, Universidad Nacional Autónoma de México, 04510 México, D. F. ^bInstituto de Física, Universidad Nacional Autónoma de México, ^cLaboratoire Tectonophysique, UMR CNRS- Université Montpellier II. E-mail: luis.alva@gm.univ-montp2.fr

Keywords: magnetite, gold, nanoparticles

Physical-chemical and mineralogical characterization studies permitted identification and analyses of magnetite and gold nanoparticles. This revelation provided enough information to infer the geological environment favorable for production of nanoparticles associated to a redox-aqueous ecosystem where the bacteria action acquires great importance. We used optical microscopy, Mössbauer spectroscopy, differential gravimetric thermoanalysis and high resolution transmission electron microscopy for the study of magnetite and gold nanoparticles. We infer the origin of magnetite and native gold nanoparticles formed in a submarine-hydrothermal environment of shallow to depth water.

MS26 P02

In – situ hot – stage transmission electron microscopy of $\text{Pb}(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3$: Ljubomira Ana Schmitt^a, Ralf Theissmann^b, Jens Kling^a, Hans Kungl^c, Michael Hoffmann^c, Hartmut Fuess^a, ^aInstitute of Materials Science, Darmstadt University of Technology, Darmstadt, Germany. ^bInstitute of Nanotechnology, Research Centre Karlsruhe, Karlsruhe, Germany. ^cInstitute of Ceramics in Mechanical Engineering, University of Karlsruhe, Karlsruhe, Germany. E-mail: ljuba@st.tu-darmstadt.de

Keywords: Transmission electron microscopy, ferroic domain structures, ferroelectrics.

The influence of maximum temperature, heating- and cooling rate on the domain configuration of $\text{Pb}(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3$ with $x = 0.40, 0.45, 0.46, 0.47, 0.48$ and 0.55 was analysed by transmission electron microscopy (TEM). Investigations were carried out to establish a basis for further hot-stage experiments including the observation of changes in domain morphology as a function of temperature. The investigations revealed a temperature dependent appearance and disappearance of nano- and microdomains. The appearance of domains in the nano scale range during cooling, denoted as domain miniaturisation, and the time dependent recovering of the former domain structure, revealed that under specific experimental conditions the domain configuration is reversible.

Samples with compositions around the morphotropic phase boundary showed formation of nanodomains within the microdomains [1]. The appearance of nanodomains in $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ and $\text{Pb}(\text{Zr}_{0.53}\text{Ti}_{0.47})\text{O}_3$ was already described by Goo et al. [2] and Lucuta and Teodorescu [3]. Evidence of further twinning, however, within the nanodomains for composition $\text{Pb}(\text{Zr}_{0.54}\text{Ti}_{0.46})\text{O}_3$, designated as substructural twinning, is reported for the first time. The observations are consistent with the theory of Sapriel [4], Fousek and Janovec [5].

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MS26 P03

Using of the electron microscopy in the study of alloys

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Keywords : electron microscopy; long range order; short range order; phases; alloys

Commercial alloys like high speed steels and white cast irons have complex microstructures due to the variety of alloying elements that enter usually in their constitution. These alloys include c

carbides forming elements such Cr, Mo, W, V... which improve high hardness, and (or) other additions like Si, Al, Cu, Ni... which reinforce the matrix.

We present here a part of our study carried on ternary alloys FeMC (M:V,Cu) and quaternary alloys FeVXC (X:Cu,Al). In the first stage of our study we have employed the diffraction of Xray to determinate the nature of the phases formed in our alloys. It's turned out that this technique is limited for identify all the present phases cause of the strong matrix effect.

The microstructures are examined by scanning electron microscopy (fig. 1) and analysed by and microprobe. If these two methods gave good results for morphologies and for chemical analyses of phases rising from liquid reactions, it is not the same for those appearing at solid state.

To get round this insufficiency we employed transmission electron microscopy, which allow us to identify the precipitates and to highlight the presence of ordered structures at long and short distance in carbides. The following phases are revealed: primary and eutectic VC_{1-x} carbides, eutectic and proeutectoid cementite, ferrite and pearlite. Copious precipitation of Cu appears in (both in the ternary and quaternary) cementite shells, in the proeutectoid cementite and in the matrix (fig. 2). This precipitation may occur by interphase boundary diffusion during cooling. The VC_{1-x} carbides are sub-stoichiometric and exhibit long range order. The two ordered V_8C_7 (cubic) and V_6C_5 (hexagonal) structures have been identified (fig. 3).

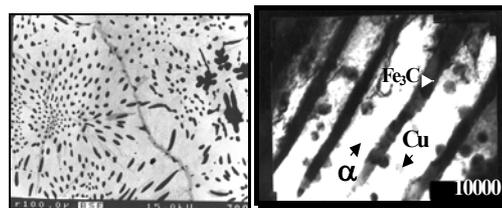


Fig.1 : SEM micrograph of Cu1 **Fig.2-** pearlitic matrix with particles of copper

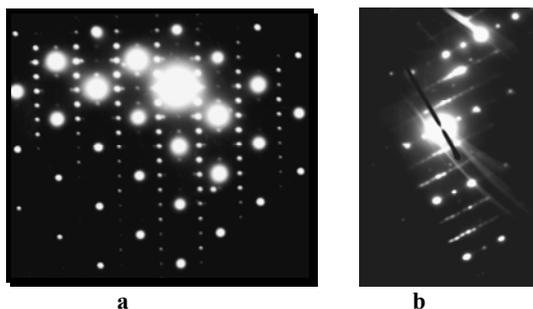


Fig. 3: a - $[0\bar{1}1]_c$ diffraction pattern of V_8C_7 superlattice. b - $[11\bar{2}]_c$ diffraction patterns of V_6C_3 superlattice.

MS26 P04

On formation of curved kikuchi lines Robert Karakhanyan, Karine Karakhanyan. Department of Physics, Yerevan State University, Yerevan, E-mail: rkarakhanyan@yandex.ru

Keywords: electron, diffraction, crystal.

In our previous report [1] about the curved Kikuchi lines in the transmission diffraction patterns from silicon was concluded that the curvature of the Kikuchi lines is conditioned by the presence of the limited defects in the investigated specimens. In the present report this curvature is explained within the framework of the Kikuchi patterns formation elementary mechanism [2] with due regard for the Kikuchi electron double diffraction. In this case the diffracted electron beams contribute to the formation of the Kikuchi patterns, for example, to the appearance of the forbidden and unindexed Kikuchi lines [3], as well as to the enhancement and to the contrast reversal of Kikuchi lines [4,5]. It is founded that as distinct from the specimens giving the straight lines, when the primary electron beam forms the Kikuchi lines at full length, in the case of the curved Kikuchi lines the primary electron beam does not form the Kikuchi lines at full length, and forms only the segments positioned in the vicinity of the perpendicular drawn from the zero spot to the given pair of the Kikuchi lines. Similarly, the diffracted beams also form only the segments disposed in the vicinity of the perpendiculars drawn from the corresponding spots to the same Kikuchi lines. It is obtained that as the Kikuchi lines are rigidly attached to the spots [2], any displacement of the spots from their normal position leads to the corresponding displacement of lines segment conditioned by these spot reflections. The different displacements of the diffracted spots situated along the Kikuchi lines are revealed. The displacements are increased in the segments, which are more distant from the zero spot. The corresponding changing of the location of the Kikuchi line segments is obtained. The curved Kikuchi lines are formed in consequence of the join of the separate segments displaced from each other. The direction of the line curvature depends on the direction of the spots displacement. The continuity of the Kikuchi lines is due to the continuous distribution of the inelastic scattered electrons forming the Kikuchi lines.

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MS26 P05

In situ TEM study of the domain evolution in $Pb(Zr,Ti)O_3$ under electric field Jens Kling^a, Ljubomira A. Schmitt^a, Ralf Theissmann^b, Hans Kungl^c, Michael J. Hoffmann^c, Hartmut Fuess^a, ^aInstitute for Materials Science, Darmstadt University of Technology, Germany. ^bInstitute of Nanotechnology, Research Centre Karlsruhe, Germany. ^cInstitute of Ceramics in Mechanical Engineering, University of Karlsruhe, Germany. E-mail: j_kling@st.tu-darmstadt.de

Keywords: transmission electron microscopy, ferroelectric oxides, domain structure

The microstructure and especially the domain configuration have an important influence on the properties of ferroelectrics. Lead zirconate titanate ($Pb(Zr,Ti)O_3$) is one of the most prominent ferroelectrics due to its excellent properties near the morphotropic phase boundary. Under cyclic load, however, the material exhibits considerable fatigue which seems to be closely related to the domain configuration.

We have performed *in situ* transmission electron microscopic investigations under electric field in order to elucidate this correlation further. Electrode geometry for a modified TEM heating holder was developed, to apply voltage and thus an electric field to the specimen.

The microscopic reaction was examined on specimen of the morphotropic phase with a composition $Pb(Zr_{1-x}Ti_x)O_3$ between $x=47.5$ and $x=44$.

In the examined voltage range only a small change of the domain configuration was observed, but the contrast in the microdomains responded to the applied field. This indicates a change in the internal domain structure. In samples with composition $Pb(Zr_{0.54}Ti_{0.46})O_3$ these modifications are related to nanodomains, which are known for these compositions [1]. A change in nanodomain morphology was recognisable. Observations on other compositions strongly support this conclusion.

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