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Pure $\text{Ni}_{0.5}\text{TiOPO}_4$ was prepared via a traditional solid-state reaction, and then $\text{Ni}_{0.5}\text{TiOPO}_4/\text{C}$ composites with core-shell structure were synthesized by hydrothermally treating $\text{Ni}_{0.5}\text{TiOPO}_4$ in glucose solution. X-ray diffraction patterns confirmed that $\text{Ni}_{0.5}\text{TiOPO}_4/\text{C}$ crystallized in the monoclinic $\text{P}2_1/\text{c}$ space group. The morphology and the microstructure were characterized by scanning electron microscopy and transmission electron microscopy. The small particles with irregular shapes were coated with uniform carbon film of ~ 3 nm in thickness. Raman spectroscopy also confirmed the presence of carbon in $\text{Ni}_{0.5}\text{TiOPO}_4/\text{C}$ composites. The electrochemical performances of $\text{Ni}_{0.5}\text{TiOPO}_4/\text{C}$ and $\text{Ni}_{0.5}\text{TiOPO}_4$ were compared through galvanostatic charge/discharge tests, cyclic voltammetry and electrochemical impedance spectroscopy. $\text{Ni}_{0.5}\text{TiOPO}_4/\text{C}$ composites exhibited improved electrochemical performances due to the existence of carbon shell. During the first discharge, the NTP/C electrode delivered a capacity of 530mAh/g Fig 1. This high capacity corresponds the intercalation of more than 3 mol lithium ions per $\text{Ni}_{0.5}\text{TiOPO}_4$; however, not all lithium atoms could be extracted during the subsequent charge [1]. The redox couples of $\text{Ti}^{4+}/\text{Ti}^{3+}$, $\text{Ti}^{3+}/\text{Ti}^{2+}$ and Ni^{2+}/Ni in NTP/C can involve the insertion of only 3 mol lithium atoms. Therefore, the excess capacity during the first discharge should attribute to the formation of SEI passivation layer

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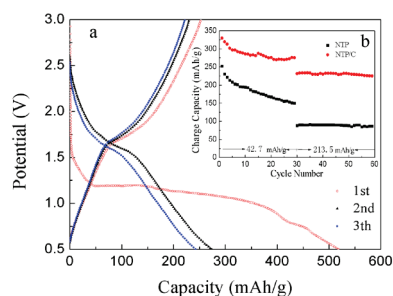


Fig. 1. The initial three galvanostatic charge/discharge curves of NTP (a) and the cycle performances of NTP and NTP/C measured at C/10 (42.7 mAh/g) and C/2 (213.5mAh/g) in the potential range of 0.5 to 3.0 V (b).

Keywords: $\text{Ni}_{0.5}\text{TiOPO}_4/\text{C}$, lithium-ion batteries, core-shell, anode materials

FA2-MS16-P25

X-ray diffraction study of σ -phase formation in super duplex stainless steel. Jorge Garin^a, Rodolfo Mannheim^a, Manuel Camus^b. ^aMetallurgical Engineering, Universidad de Santiago de Chile, Chile. ^bMechanical Engineering, Universidad de Antofagasta, Chile.

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Super duplex stainless steels (SDSS) are two-phase alloys based on the iron-chromium system, with minor contents of molybdenum, nitrogen, tungsten and copper. Their remarkable mechanical and chemical properties make them suitable for

widespread industrial applications. The microstructure of these alloys is composed of approximately equal proportions of BCC ferrite and FCC austenite phases. However, exposure to elevated temperature processes causes embrittlement and loss of mechanical properties due to precipitation of intermediate phases, principally sigma-phase, in the microstructure. This phase is a complex intermetallic compound of Fe and Cr, based upon an ideal stoichiometric composition AX_2 , Pearson's code tP30 and space group $\text{P}4_2/\text{mmn}$, [1]. The formation of sigma-phase in cast ASTM A890 steel was investigated by means of quantitative X-ray diffraction. Three different temperatures (1023 K, 1073 K and 1123 K) and various annealing times (1 to 96 hours) were utilized in the experimental procedure. Owing to the usually complex powder diffraction pattern of the sigma compound, Rietveld refinements were performed based upon typical measurement and global parameters. The results obtained of the present study have assessed the application of the Rietveld method to quantify the formation of sigma-phase in SDSS subjected to annealing at relatively high temperatures. The refinement yielded the lowest R-values and much better represented the relative amount of phases in the samples. From the metallurgical standpoint, the results of XRD followed by Rietveld analysis indicated that the larger the annealing time of the alloy at a given temperature, the larger will be the volume fraction of the precipitated particles of sigma. Furthermore, sigma particles nucleates and grow reaching saturation levels depending on the specific type of the alloy.

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Keywords: σ -phase, X-ray diffraction, duplex steels

FA2-MS16-P26

Microstructure of coke deposited by a high temperature process. Sven Gerhardt^a, Reiner Staudt^b, Klaus Bente^a, Jörg Hofmann^b. ^aInstitut für Mineralogie, Kristallographie und Materialwissenschaft, University of Leipzig. ^bInstitut für Nichtklassische Chemie, University of Leipzig.

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It is well known that metal dusting is an undesirable corrosion phenomenon that can be observed in a wide range of chemically and petrochemically working industries. Due to strongly carburizing atmospheres, it leads to a decomposition of materials into metal particles and carbon. The aim of this work is to clarify open questions by identifying and modeling fundamental correlations between vapour-solid reactions and their consequences for microstructure, surface morphology and catalytic behaviour of the metal. The high temperature grown coke coating and metal pipe were inspected by 3D X-ray tomography (volume properties), XRD (existing phases), XPS (chemical bonding), HRTEM (nanostructures), SEM (surface morphology), electron microprobe analysis and TEM-EDX (chemical composition).

Keywords: transmission electron microscopy, X-Ray tomography, coke

FA2-MS16-P27

Mullite-type $(\text{Bi}_{1-x}\text{Sr}_x)_2\text{Al}_4\text{O}_{9-x/2}$: HT-XRPD, TEM and XPS investigations. Thorsten M. Gesing^a, Marco Schowalter^b, Claudia Weidenthaler^c, Andreas

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Bi₂M₄O₉ compounds are known to have potential as electrolytes in solid oxide fuel cells (SOFC) [1]. The conducting properties could be related to an increased number of oxygen vacancies due to the requirement of charge neutrality with the substitution of Bi³⁺ by Sr²⁺ [2]. The samples were synthesized using the glycerin method as described in [3] and finely washed with nitric acid to remove impurity phases. Two different heating procedures were used for samples having an initial Sr content of $x = 0.2$. Heating steps of 353 K (1/2 h), 473 K (1/2 h) and 1023 K (2h) were used for one sample (#173) and the strontium free reference (#174), whereas the faster heated sample (#172) was treated at 473 K (1/4 h) and 1023 K (2h). The unit-cell volumes calculated from X-ray powder diffraction data of all samples calculated against silicon as internal standard show only a very small variation around $357.3(5) \cdot 10^{-6} \text{ pm}^3$. Whereas small amounts of Bi₂O₃ and SrAl₄O₉ were found in the as synthesized samples no indication for these impurities were detected in the washed samples. To prove the incorporation of Sr into the mullite-type structure small crystals of a sample with an initial Sr content of $x = 0.2$ (#172) were selected in a HRTEM. Suitable crystallites were analyzed by EDX in a scanning TEM mode using a spot size of approximately 300 pm. Several particles were evaluated showing a variation of x between 0.05 (often) and 0.24 (rare). A line scan on a crystal having a high Sr content ($x = 0.244$) shows homogeneous strontium distribution. If strontium is incorporated into the Bi₂Al₄O₉ structure two possibilities for charge balance have to be discussed. On one hand oxygen deficiency with vacant oxygen positions are possible. On the other hand for incorporating 2 Sr²⁺ ions 1 Bi³⁺ could be oxidized to Bi⁵⁺. To exclude the latter possibility XPS investigations were carried out on two samples with $x = 0.2$ (#172, #173) and Bi₂Al₄O₉ (#174) and Bi₂O₃ as references. No indication was found that Bi⁵⁺ is formed during the strontium incorporation. Nevertheless, for the fast synthesized sample (#172) the Bi4f photoelectron peaks are much more asymmetric than in the slowly synthesized compound (#173). X-ray powder heating experiments between 298 K and 1272 K for Bi₂Al₄O₉ show a (nearly) linear thermal expansion in all three orthorhombic lattice directions without any changes during the heating and cooling runs. For the strontium doped samples an irreversible deviation for the thermal expansion was observed at ~1085 K (#172) and ~1135 K (#173) for the as synthesized and at ~1185 K for both washed samples. Adding 15 wt-% Bi₂O₃ to the latter sample (#173) the expansion deviation was observed at ~1035 K. In all cases the refined (Rietveld method) strontium content on the bismuth position was reduced from 15(3)% to zero at these temperatures.

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Keywords: mullite-related phases, high temperature, strontium doping

FA2-MS16-P28

Polytypism in Na[C₆H₄Cl(SO₃)]·H₂O analysed by Order-Disorder (OD) Theory. Jürgen Glinnemann,

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Ordered and disordered crystal structures of the title compound are built up by two chemically rather distinct kinds of alternating layers: (1) hydrophilic NaSO₃(H₂O), and (2) hydrophobic C₆H₄Cl layers [1].

The hydrophilic layers are ordered and act like templates on the hydrophobic layers. Due to this steric condition, the C₆H₄ rings have to be mutually perpendicular aligned in their respective layers. These have an orthogonal metric with layer-group symmetry *C1m1*, layer orientation (001). The ability for different stacking sequences of these layers gives rise to a family of structures, ordered and disordered ones. Their discussion will follow the lines of order-disorder (OD) theory [2, 3], and starts with the OD groupoid family symbol:

$$C \ 1 \ m \ (1) \\ \left\{ \begin{array}{l} 1 \ 2_s \ 1 \\ 2_r \ 1 \ n_{r,s} \end{array} \right\}$$

The OD approach results in three maximum degree of order (MDO) polytypes and in the disordered family structure, all structures consisting of four layers (1) and (2) in their respective unit cells. Two of the MDO structures have space-group type *P12₁/c1* ($Z = 8$) (layer orientation (010)) with the same lattice parameters. The third MDO structure has space-group type *C1c1* ($Z = 16$), with an orthogonal metric (layer orientation (001)). The disordered family structure ('average structure') is orthorhombic, space-group type *Pnma* ($Z = 4$) (layers parallel (100)).

One of the MDO '*P2₁/c* polytypes' could be identified by X-ray single-crystal analysis, yet with severe disorder, as exemplified by streaks of diffuse scattering in direction perpendicular to the structural layers [1]. Moreover is this structure twinned, the twin laws reflecting the two mirror planes of the point group of the orthorhombic family structure perpendicular to the layers.

In addition to the three MDO polytypes and the disordered family structure, all with a periodicity of four layers, less-ordered structures with larger periodicities or disordered ones without any stacking periodicity may occur. Altogether these polytypes establish the OD family of structures of the title compound.

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Keywords: polytypes, order-disorder (OD) theory, sodium *p*-chlorobenzenesulfonate monohydrate

FA2-MS16-P29

RT and high T crystal structure of ferroelastic

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