

is known to be precipitated from glass phase, we got glass ceramics with high Q_f . In this paper, phase relations in the glass ceramics are examined by powder X-ray diffraction patterns.

Powders obtained from glass melted at 1550 °C are crystallized for 10 h at 1200, 1300 and 1400 °C, and for 50 h at 1200 °C. The powder diffraction data are collected with the multiple detector system on beam line BL4B2 at the Photon Factory in Tsukuba (Toraya *et al.*, 1996). As corundum phase are precipitated in all samples, it was used for internal standard. These samples are analyzed by Rietveld method using RIETAN-FP by Fujio Izumi. Initial atomic parameters for indialite, cordierite and corundum are sited from ICDD-PDF4+ cards.

Sample #1: 1200 °C, for 10 h was analyzed by two models: (1) Indialite + Corundum, and (2) Cordierite + Corundum. As 100 - diffraction peak profile of indialite shows slight asymmetric, it was considered that the symmetry of indialite becomes lower. But model (2) has a 311-diffraction peak which was not observed in the diffraction pattern. So, this crystal structure is similar to indialite, and cordierite percentage ρ is 3.5 %. Sample #2: 1300 °C for 10 h was analyzed by four models: (1) Indialite + Corundum, (2) Cordierite + Corundum, (3) Indialite + Cordierite + Corundum, and (4) Cordierite I + Cordierite II + Corundum. In the case of model (3), the fittings of some reflections were not sufficient. So, two cordierite I and II with different lattice parameters are applied. The cordierite percentage ρ is 32.5 %. Sample #3: 1400 °C for 10 h was analyzed by three models: (1) Cordierite + Corundum, (2) Indialite + Cordierite + Corundum, and (3) Cordierite I + Cordierite II + Corundum. In the case of model (3), the cordierite percentage ρ is 83.3 %. Sample #4: 1200 °C for 50 h was analyzed by three models: (1) Indialite + Corundum, (2) Cordierite + Corundum, (3) Indialite + Cordierite + Corundum. In the case of model (3), the cordierite percentage is 21.2 %.

Here, cordierite percentage $\rho = (a/\sqrt{3}b-1)/(17.0448/9.7127\sqrt{3}-1)$, a and b : lattice parameter of cordierite obtained, $a=17.0448$ Å, $b = 9.77605$ Å, and $c = 9.34498$ Å are lattice parameters of pure cordierite [ICDD-PDF#01-089-1487].

The cordierite percentage increases according to temperature and crystallizing time. And the quality factor Q_f is depending on the indialite (with high symmetry) percentage $1-\rho$. High symmetry affects to Q factor more than ordering.

Keywords: millimeterwave microwave dielectrics, indialite and cordierite, rietveld refinement

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Phase formation in deformed iron-based alloys under saturation by N and C

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The diffusion saturation of iron-based alloys by nitrogen and carbon is widely used in industry for increase of strength, hardness, wear and corrosion resistance of metal products. Inexhaustible and unrealized potentialities of such treatment are opened when applying it under strain and stress condition [1]. The topical question in this direction is clarification of mechanisms of N and C diffusion and phase formation in strained iron and iron-based alloys.

The structure, phase composition and properties of surface diffusion layers formed in the preliminary deformed α -Fe and Fe-Cr, Fe-Ti, Fe-Cr-Ti, Fe-Ni alloys after diffusion saturation by N and C were studied. Preliminary plastic deformation (PPD) was realized by rolling with deformation degrees: $\varepsilon = 3, 5, 8, 10, 15, 20, 25, 30, 35, 40, 50, 60,$

70 %. The following gaseous saturation with nitrogen and carbon was performed from ammonia and propane-butane at $T = 623\div 853$ K.

According to the SEM, TEM, XRPD investigations the diffusion layer is a combination of surface layers of different nitride (ξ -, ε -, γ' -) phases, nanostructured ($\gamma' + \varepsilon$) - eutectoid layer and a zone of internal saturation ($\alpha_{N,C}$ -phase with *bcc* structure). According TEM the eutectoid consists of γ' -phase (*fcc* structure with lattice parameter $a=0.378\pm 0.004$ nm) and ε -phase (*hcp* structure). The lattice constant of the α -solid solution of N in Fe is $a=0.286\pm 0.004$ nm.

PPD considerably effects on the phase formation, structure, microhardness and thickness of nitrided layers. The thickness of ε -phase layer depends on deformation degree and changes in the range of 25-43 μm . The nitrided layers formed in Fe-Ni alloys some differ from those in α -Fe. These differences consist in the presence of additional layer of ξ -phase on the surface. The diffusion layers in Fe-Cr-Ti alloys consist of a surface layer of ε -phase ($\text{Fe}_{2.3}\text{N}$ type) with hexagonal lattice or θ -phase with orthorhombic lattice isostructural to cementite (Fe_3C type) crystal lattice and a zone of solid solution of nitrogen and carbon in α -phase.

The distribution of carbon and nitrogen atoms in Fe after deformation and nitriding were studied using Mössbauer spectroscopy. An analysis of the hyperfine parameters has shown that the NGR spectra consist of several components relating to iron atoms in α -Fe and iron nitrides (ε -, γ' -phases).

The microhardness test of nitrided layers has discovered the narrow intervals of deformations of 3-8 % and 20-30 % in which the considerable rise (in about 2 times) of microhardness of the surface diffusion layer after nitriding of α -Fe exist. The high microhardness of the diffusion layers results from the formation of the ε - and γ' -nitrides. Iron doping with Ni leads to changing of the ε -, γ' -phases composition.

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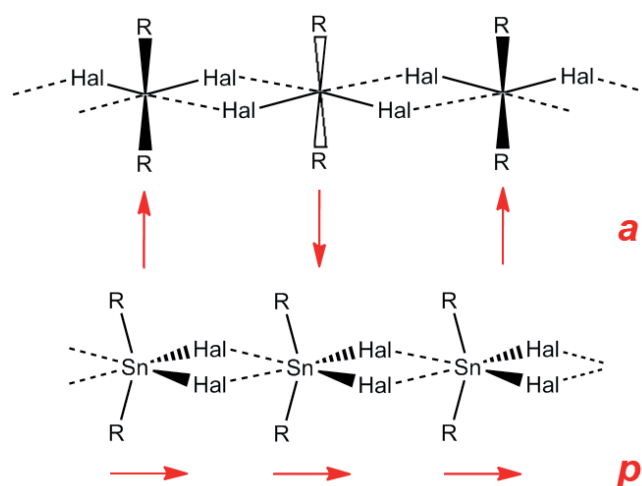
Keywords: deformation, diffusion, nitride

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Structure types of intermolecular tin halide interactions in diorgaotin dihalides

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Diorganotin dihalides, R_2SnHal_2 with Hal = Cl, Br, I, belong to the basic chemicals in organotin(IV) chemistry because they are versatile precursors for the preparation of other diorganotin compounds like complexes, hydrolysis products, hydrides and so on. Since the first structural characterization of Me_2SnCl_2 in 1970 by Davies et al. [1] some dozens structures of dihalides were determined but no general classification scheme was evolved. In solid state, many of these dihalides exists as monomers without any intermolecular tin-halide interactions because of intramolecular Lewis-base Lewis-acid adduct formation or because of steric hindrance but especially the common ones with hydrocarbon rests are dominated by intermolecular tin halide interaction with strong effects on intramolecular bond angles and lengths. For this kind of interaction which is often termed *secondary bonding* [2] or *supramolecular architecture* [3] two main types of arrangements were found: 1) side by side with the dipole moments more or less antiparallel (a) to each other or 2) one after another with two equal or different tin halide interactions and a more or less parallel (p) ordering of the dipole moments.

In the course of a systematical study on the crystal structures of diorganotin dihalides we obtained – in addition to the already known structure types - a lot of new ones which gave us the possibility to develop a general classification scheme for the intermolecular tin-halide interaction. To discriminate the different structure types, our scheme takes into account the dipole-dipole interaction represented by the angle bisector between the halide atoms, the crystallographic symmetry elements between the interacting molecules and a tin-halide respectively tin-tin aggregation plane. The poster will show the limitations and options of our scheme covering oligomeric ($2a$, $4a$) arrangements, the different chainlike ones built up by monomers with parallel ($P_0 - P_2$) or antiparallel ($A_0 - A_2$) orientations of the dipole moments, the chainlike ones built up by dimers [$C2(2a_1)$] as well as the band [$B(pp)$] and sheet [$S(2a_1)$] like structure types.

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Crystal structures of few new β -octamolybdates

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Octamolybdates are important and interesting group of compounds. Among the molybdates, they are the most numerous group of compounds. In addition, there are several types of octamolybdates. Why are they important to study, when their applications are not especially numerous yet?. The reason is a desire to understand the rules governing the formation of certain polymetallate anions. These rules are quite complex, the type of anion depends on the reaction time, concentration and type of reactants. An additional element is the richness of anionic forms and their potential applications in crystal engineering. For a deeper understanding of principia of polymetallate construction, structural studies of a group of newly obtained compounds, have been undertaken. We investigated octamolybdates of aniline derivatives with the general formula $NH_2-Ph-R_{(1 \text{ or } 2)}$ and mm2 symmetry.

Synthesis: aqueous solution of molybdic acid and amine, acidified with HCl or CH_3COOH was boiled under reflux for 1-24hrs.

Among the most surprising findings is the fact that; short-term synthesis using 4-methylaniline results in octamolybdate, while the result of a long (24hr) synthesis is pentamolybdate. Similar results were observed in the case of 4-iodoaniline. Interestingly, using 3,5-dimethylaniline exclusively trimolybdate is produced, regardless of the reaction time. In the case of 4-fluoroaniline and 4-chloroaniline, 24-hour reactions conducted under the same conditions, have resulted in a complex polycrystalline material, which has not been fully explored yet.

Among anilinium octamolybdates only β -isomers were obtained.

The results indicate that, the creation of a specific polymolybdate type depends on many factors, and structural 'synthons' present in the 'reacting system', may help to describe the resulting structure. However, on their basis, it is much harder to predict resulting anionic species.

All compounds were synthesised for the first time in our laboratory. Structural studies were performed mainly by single crystal methods (Bruker-Nonius), phase composition and structural studies for compound **5** by powder diffraction methods. All compounds were also characterised by IR spectroscopy, SEM, XRD vs. temp and DSC investigations. Some crystallographic data are listed below:

1. $Mo_8O_{26} \cdot 4\{NH_2-C_6H_4-CH_3\}$ – tetrakis(p-toluidinium) β -octamolybdate, Sg. P 2₁/c (14), 8.388(2), 26.669(4), 9.346(3) Å, 103.65(3)°, V=2031.8 Å³, Z=2
2. $Mo_8O_{26} \cdot 4\{NH_2-C_6H_4-F\}$ – tetrakis(p-fluoroanilinium) β -octamolybdate, Sg. P-1(2) 10.567(4), 14.605(3), 15.724(5) Å, 117.16(2), 96.67(4), 97.80(4)°, V=2095.8(8) Å³, Z=2
3. $Mo_8O_{26} \cdot 4\{NH_2-C_6H_4-Cl\}$ – tetrakis(p-chloroanilinium) β -octamolybdate, Sg. P-1(2) 10.586(3), 14.705(5), 16.407(4) Å, 116.26(2), 93.43(2), 98.38(3)°, V=2243 Å³, Z=2
4. $Mo_8O_{26} \cdot 4\{NH_2-C_6H_4-(CH_3)_2\}$ – tetrakis(2,6-dimethylanilinium) β -octamolybdate, Sg. P-1(2), 10.508(5), 11.578(5), 11.686(5) Å, 118.59(5), 93.74(5), 101.146°, V=1204 Å³, Z=1
5. $Mo_3O_{10} \cdot 2\{NH_2-C_6H_4-(CH_3)_2\}$ – bis(3,5-dimethylanilinium) trimolybdate, Sg. P-1(2) 8.936(3), 16.729(4), 7.580(2) Å, 92.20(2), 93.138(3), 98.18(3)°, V=1118.4(4) Å³, Z=2

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Crystal structure of $C_4H_8ONH_2-PbBr_3$ by neutron and X-ray diffraction experiments

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$C_4H_8ONH_2-PbBr_3$ has a lead-based inorganic-organic perovskites structure. One-dimensional chains of face shearing $PbBr_6$ octahedra are isolated by $C_4H_8ONH_2^+$ cations to be quantum wire, so this material can be regarded as a naturally self-organized one-dimensional system. The crystal structure of the material is orthorhombic with space group of $P2_12_12_1$. $PbBr_6$ octahedra are strongly distorted because of the existence