MS31.P34

 estão coexistentes na matriz cristalina. Nas outras ligantes, apenas um isômero é observado.

Keywords: Yb, YAG transparent ceramic materials, transparent ceramics, nanopowder

MS31.P35

The sol-gel combustion method was used to synthesize Yb: GGG laser ceramic nanopowder. The phase structure, thermal stability, particle size and morphology of Yb: GGG powder were studied by means of XRD, TG-DTA, IR and SEM methods. It was found that the as-synthesized powder belongs to the GGG phase. The crystalline size grew with the increase of the heat treatment temperature. The size of powder calcined at 1000°C was about 70-150 nm, which is favorable for good sinterability of Yb: YAG ceramics.

Keywords: Yb: YAG, laser materials, transparent ceramics, nanopowder

MS31.P36

Structure and properties of Mo(II) complexes with dinitrogen bidentate ligands

Maria José Calhorda, Maria Vasconcelos Dias, Marta Saraiva, Margarida Meireles, Daniel Bandarra, Miguel Lopes, Vítor Félix, Faculdade de Ciências da Universidade de Lisboa, 1749-016 Lisboa (Portugal). 

Reaction of [M(μ-η2-C5H4)(CO)3X(NCMe)2] (M = Mo, W; X = halide) complexes with bidentate nitrogen ligands leads to substitution of the two nitriles, and two different isomers may be formed. The two carbonyls and the centroid of the allyl group always adopt a facial arrangement in the pseudo octahedral environment around Mo(II), with the halide occupying a position trans (equatorial isomer) or cis (axial isomer) to the allyl. When C5H4NCH=CH(NCMe)2 (pyca) is used, the two main isomers have very close energies (DFT calculations) and they coexist in the crystal. With the other ligands, only one isomer is observed in the solid, though often several are detected in solution, especially when ligands are small, owing to the fluxional behavior of the complexes in solution (NMR).

Some of the complexes display catalytic activity, both in homogeneous and heterogeneous conditions, in olefin oxidation in the presence of t-butylhydroperoxide.[1] and others can act as antitumor agents. [2] The antitumor properties have been tested in vitro against human cancer cell lines such as cervical carcinoma (HeLa) and breast carcinoma (MCF-7), using the MTT metabolic activity test (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide). 1,10-phenanthroline (phen) seems to be the most effective ligand, with a IC50 of 23.7 µM (Mo, Br). The substitution of Br by trflate usually enhances the cytotoxic activity. In [M(μ-η2-C5H4)(CO)3(CF3SO3)(phen)2], for instance, it drops to 2.9 µM. The binding constant of this complex with ct DNA, determined by absorption titration, is 2.08 (±0.98) × 107 M⁻¹, a value comparable to the one determined for ethidium bromide. This result suggests that the complexes may interact with DNA changing its conformation. The molecular structures may give information about the possibility of complex intercalation in DNA. Crystal structures exhibit different features (inter and intra molecular hydrogen bond, π-π stacking) depending on the functional groups available.

Crystal structure of bacterial citrate lyase

Farhad Forouhar, Scott Lew, Jayarama Seetharaman, Rong Xiao, Thomas B. Acton, Gaetano T. Montelione, John F. Hunt, Norteast Structural Genomics Consortium, (USA). Department of Biological Sciences, Columbia University, New York, NY 10027, (USA). Center for Advanced Biotechnology & Medicine, Department of Molecular Biology & Biochemistry, Rutgers University, Piscataway, NJ 08854, (USA). Department of Biochemistry, Robert Wood Johnson Medical School, Piscataway, NJ 08854, (USA). E-mail: farhadf@columbia.edu

Citrulline lyase activity exists in eukaryotes, bacteria, and archaea. This underscores its central importance in energy metabolism and biosynthesis. There are two types of citrate lyases, ATP-dependent and non-ATP-dependent. The later bacterial citrate lyase converts citrate to acetate and oxaloacetate by a two-step reaction for which a complex of αβγ-lyase is required. We present the crystal structure of the bacterial citrate lyase complex comprising six copies of αβγ-lyase at 4 Å resolution. The structure reveals how the 18-subunit complex is assembled and provides insights into citrate catalysis.

Keywords: structure, citrate, lyase

MS32.P01

Microstructure of synthetic Mg-Co corderite studied by X-ray diffraction methods

José Maria Amigo, Noemi Montoya, Francisco Javier Serrano, Marek Andrzej Kojdecki, Maria Mercedes Reventós, José Luis
In the present study we have performed an experimental and high strength and resistance to fatigue to produce bearings with an acceptable lifetime. The room-temperature microstructure of these steels consists of martensite, metastable austenite (10-25 vol.%) and a small amount of carbides (< 5 vol.%). Small dimensional changes in the bearing during operation influences its function and thereby that of the machine in which it operates. One key process that critically affects the bearing dimensions during operation is the decomposition of the metastable austenite phase at elevated temperatures. The austenite face-centred cubic (fcc) structure transforms into the bainite body-centred cubic structure, together with the appearance of very fine nm-sized precipitates.

The combined use of the high-energy X-rays available at the ESRF and our dedicated micro-tensile stage with heating capabilities has allowed us to monitor in-situ the austenite decomposition in the bulk microstructure as a function of time for different combinations of temperature and applied tensile stress. The measurements were performed by continuously recording the diffraction patterns on a two-dimensional detector placed behind the sample as a function of time at selected temperatures in the range of 476-504 K for an applied tensile stress of: \( \sigma = 0 \) and \( \sigma = 295 \) MPa. This combination of temperature and stress mimics the operating conditions of the bearings during its lifetime. The available optics at the ID15A beam line of the ESRF permits to reduce the beam dimensions down to 5×25 µm². This has allowed us to study both the average behaviour of the metastable austenite phase and the transformation kinetics of individual austenite grains. The observed transformation behavior has been correlated to the change in sample dimensions that are recorded simultaneously during the in-situ diffraction experiment. The Rietveld analysis of the powder diffraction data reveals that the time to completely transform the metastable austenite reduces from 6 to 3 hours when increasing the temperature from 476 to 489 K. The presence of the applied tensile stress of 295 MPa reduces the activation energy from 138 to 104 kJ/mol. We have also observed a continuous decrease in the austenite lattice parameter while keeping the sample at a constant temperature. This effect is ascribed to the depletion in carbon of the remaining austenite grains resulting from the formation of iron carbides. The results of our in-situ diffraction study constitute a key input to control the austenite stability in high-carbon steels during the industrial processing of the material, in order to keep the changes in the bearing dimensions within the required limits during its lifetime.

**Keywords:** steel, creep, diffraction

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**MS32.P03**

A multidisciplinary study of the resolution of hydrotropic acid by ephedrine

Miguel Aridid Candeli, Sarah L Price, Alan G Jones, Department of Chemical Engineering, University College London, Torrington Place, London WC1E 7JE, Department of Chemistry, University College London, 20 Gordon Street, London WC1H 0AJ, (United Kingdom). E-mail: m.candel@ucl.ac.uk

Following the introduction of regulatory guidelines for the production of stereoisomeric drugs in 1992, the number of drugs marketed as single enantiomers has increased considerably [1]. Classical resolution is often used to obtain enantiopure compounds by the pharmaceutical industry. However, despite its importance, the choice of chiral acid/base to use as a resolving agent is still mostly made on a trial and error basis. An understanding of the relationship between the crystal structure of the diastereomeric salt pairs, their relative energies and crystallization behaviour, would lead to improvements in the separation process.

In the present study we have performed an experimental and