The nano-size iron oxide, γ-Fe_2O_3, lithiated chemically was subjected to neutron scattering studies for crystal structure analysis by Rietveld method and for local structure analysis by total diffraction technique. The lithium intercalation of the nano-size γ-Fe_2O_3 proceeded by a biphasic reaction through the defect spinel and the disordered rock-salt phases. Pair distribution functions calculated from the total scattering data indicated a correlation of Li-O with a distance of 2.36 Å, which is much longer than the bond distance in ionic crystals. Lithiation mechanism of the nano-size materials will be discussed based on the bulk and local structure changes clarified by these neutron scattering techniques.

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Keywords: nanocrystals, iron oxides, neutron scattering

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Free oxygen ions in nanoporous material $12CaO\cdot 7Al_2O_3$ and cage deformation at high temperature

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There has been great interest for a decade in the nanoporous material $12CaO\cdot 7Al_2O_3$ (C12A7) because of variety of fascinating characteristics the material exhibits, including high ionic conductivity of oxygen ions at high temperature. The characteristics are mostly due to its unique structure. The frame work structure of C12A7 in a unit cell can be represented as $[Ca_{24}Al_{28}O_{64}]^{4+}$ which consists of twelve cages with inner free space of ~ 4 Å in diameter. Two remaining oxygen ions, so called free oxygen ions, are believed to be captured in the inner space of two cages out of the twelve cages. The free oxygen ions are considered to play a important role in the oxide ion conduction. In order to obtain precise structural information, including the position of the free oxygen ions, neutron powder diffraction studies were carried out over a wide temperature range, 50 K - 700 K. The structure analyses clearly indicated that the free oxygen ion is located inside the cage and the position is displaced from the S_4 axis running through the center of the cage. It was also confirmed that the presence of the free oxygen ion in the cage induces a deformation of the cage. Calcium ions at the top and the bottom of the cage are shifted toward the center of the cage when the free oxygen ion is present in the cage. The framework structure was found to be further deformed as the temperature is elevated. The deformation involves; lengthening of aluminum-oxygen bond lengths; shortening of a distance between an oxygen ion in the cage wall and the free oxygen ion. These variations may enhance the oxygen ion migration at high temperature. A complete description of the deformation at high temperature and a possible mechanism of the oxygen ion migration will be presented.

Keywords: crystal structure-physical property relationships, ionic conductors, neutron diffraction

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Residual stress investigation of dissimilar overlap-friction stir welds made from Al and steel

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One of the main research topics at the GKSS Research Centre is the investigation and further development of friction stir welding techniques. In this context dissimilar overlap joints of sheets made from aluminum and steel were produced by friction stir welding at GKSS. A set of four different specimens was produced with different welding and tool speeds. Aluminum alloy AA5754-H22 and dual phase steel alloy DP600 were used. The aluminum and steel sheets were 1.5 mm thick and the stirring zone was 12 mm wide. The residual stress distribution in these specimens was investigated at the high energy materials science synchrotron beamline HARWI II operated by GKSS at the HASYLAB / DESY, Germany. A beam with a photon energy of 100 keV and a size of 2 x 0.2 mm^2, with the larger beam dimension being parallel to the weld, was used in transmission geometry. A Mar345 detector system was employed to monitor complete Debye-Scherrer rings. The diffraction peak shifts of the Al {311} and Fe {211} lattice planes were used to determine the residual stress in the respective material. Peak positions were determined relatively to the Cu {220} peak of a Cu-powder directly attached to each specimen. This allowed the correction for peak shifts induced by the distortion of the specimens and the resulting change of the sample-to-detector distance. The results for the weld zone show that the stresses in weld direction are close to the yield strength for the steel and about 70% of the yield strength for Al. Furthermore, high welding and tool rotation speeds result in steeper stress gradients in steel and almost symmetric stress profiles in both materials, whereas low welding and tool rotation speeds result in a broader stress profile in steel and asymmetry of the profiles in both materials.

Keywords: friction stir welding, residual stress, high energy synchrotron radiation

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Residual stresses associated with laser bending of mild steel plates

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Bending of metal plates with high-energy laser beams presents a flexible materials forming technique where bending results from the establishment of a steep temperature gradient through the material thickness. This inevitably leads to non-uniform thermal expansion/contraction and subsequently residual stresses. Non destructive residual strain mapping with diffraction techniques through the 8mm thickness of a series WA 300 grade structural steel plate samples, focused on the region straddling the centerline of the heating bead location, shows the presence of large residual stress fields. Directly below the laser track the longitudinal strains are tensile and dominant, normal strains compressive and transverse strains slightly tensile.