

the structure of one of the intergrowths using single crystal X-ray diffraction data. For the temperature range studied the *CS* phases are within a few degrees of their melting points, and this is reflected in a departure from complete structural order. The results show both long-range disorder in the intergrowth sequences and also an absence of correlation between the metal atom ordering in one *CS* plane and the next. This disorder is manifested by diffuse scattering in the diffraction patterns. The (020), *CS* structures transform reversibly to the well-known (121)_r–(132)_r family of ordered rutile *CS* structures below 1450°C.

Magnetic Susceptibility of Single Crystal Fe_{1-x}S. J. L. HORWOOD, M. G. TOWNSEND, AND A. H. WEBSTER, Mineral Sciences Laboratories, Canada Centre for Mineral and Energy Technology, Ottawa, Canada K1A 0G1. Stoichiometric FeS is characterized by three phase transitions, one structural at T_α and the others magnetic at T_s and T_N . We have obtained magnetic susceptibility data in the temperature range 80° to 600°K on single crystals of several compositions of Fe_{1-x}S for $0 \leq x \leq 0.07$. The magnetic susceptibilities of very nearly stoichiometric FeS show the two transitions well separated [$T_\alpha = 420^\circ\text{K}$, $T_s = 445^\circ\text{K}$]. In the region of T_s , the spins do not rotate gradually with temperature but flip locally due to a small spread in composition. By comparing the data with that of NiS we infer that FeS is a localized spin antiferromagnet in contrast to NiS, which is generally considered to be an itinerant antiferromagnet. $T_s(x)$ decreases linearly with increasing metal deficiency. This dependence can be partly accounted for by a decrease in the spin-orbit coupling energy arising from increasing covalency; however, we conclude that a concomitant increase in magnetocrystalline anisotropy energy also occurs in this range.

Mossbauer Effect in Single Crystal Fe_{1-x}S. J. R. GOSSELIN, M. G. TOWNSEND, R. J. TREMBLAY, AND A. H. WEBSTER, Mineral Sciences Laboratory, Canada Centre for Mineral and Energy Technology, Ottawa, Canada K1A 0G1. Mossbauer spectra have been recorded for single crystals of very nearly stoichiometric FeS and Fe_{0.93}S at temperatures between 80° and 600°K. The spectra of nearly stoichiometric FeS indicate that the low-temperature 2C structure with spins $\parallel c$ preserves the spin orientation $\parallel c$ during the structural transformation to 1C at $T_\alpha (= 420^\circ\text{K})$. However, because of a miscibility gap, a few percent of the high-temperature 1C structure with spins $\perp c$ is present even at 300°K and the concentration of this component increases with temperature. At $T_s (= 445^\circ\text{K})$ the spins in the 1C structure that are $\parallel c$ rotate $\perp c$. In Fe_{0.93}S, the hyperfine splittings indicate that the rotation of the spins from $\parallel c$ to $\perp c$ at 170°K is a first-order process.

Zur Kristallstruktur von FeI₃O₉. M. JANSEN, Institut für Anorganische und Analytische Chemie der Justus Liebig-Universität, 63 Giessen, Sudanlage, Germany. Single crystals of FeI₃O₉ were obtained by oxidizing FeI₂ at 520°C and 500 atm oxygen pressure. The crystal structure has been determined and refined by full matrix least-squares procedures using diffractometer data to a final *R*-value of 0.067; the space group is $P6_3-C_6^6$, $a = 9.225 \text{ \AA}$, $c = 5.224 \text{ \AA}$, $c/a = 0.57$. The structure consists of FeO₆-octahedra connected by iodine, each of which is coordinated by three oxygen [$d(\text{I}-\text{O})$; 1.89 and 1.90 Å for bridging O; 1.79 Å for terminal O]. The Cr and β-Sc iodates have the same structure.

Phase Equilibria in the System Fe–Fe₂O₃–Lu₂O₃ at 1200°C. T. SEKINE AND T. KATSURA, Department of Chemistry, Tokyo Institute of Technology, Tokyo 152, Japan. Phase equilibria in the title system have been established at 1200°C. The following phases were stable: lutetium sesquioxide, hematite, magnetite, wustite, metallic iron, lutetium–iron–perovskite, lutetium–iron–garnet, LuFe₂O₄, and Lu₂Fe₃O₇. The standard free energy of formation from metallic iron, Lu₂O₃, and oxygen at 1200°C of LuFe₂O₄, Lu₂Fe₃O₇, LuFeO₃, and Lu₃Fe₅O₁₂ has been determined to be -100.3 ± 0.2 , -158.2 ± 0.2 , -57.5 ± 0.3 , and -279.1 ± 0.9 kcal/mole, respectively.

Preparation, Phase Equilibria, and Crystal Chemistry of Lanthanum, Praseodymium, and Neodymium Hydroxide Chlorides. E. T. LANCE AND J. M. HASCHKE, Department of Chemistry, University of Michigan, Ann Arbor, Michigan 48104. The preparation of hydroxide chlorides of lanthanum, praseodymium, and neodymium has been achieved by hydrothermal methods at 550°C and 1530 atm. Three phases [Ln(OH)₃, Ln(OH)_{2.55}Cl_{0.45}, and Ln(OH)₂Cl] have been characterized by analytical and X-ray methods. The observed compositions are closely defined by the $x = 0, 0.5$, and 1 members of the hom-

ologous anion substitution series, $\text{Ln}(\text{OH})_{3-x}$. The previously unreported $\text{Ln}(\text{OH})_{2.55}\text{Cl}_{0.45}$ phases are clearly substoichiometric in chloride and are described by the $\text{Ln}_7(\text{OH})_{18}\text{Cl}_3$ composition. X-ray diffraction data for the phases at $x = 0.45$ show a pronounced UCl_3 -type substructure and complex superstructure reflections which have been indexed on a hexagonal cell. For the lanthanum phase, $a = 17.662(6)$ and $c = 3.914(1)$ Å. Efforts to obtain single crystals have been unsuccessful. Thermal decomposition processes of the hydroxide chlorides have been investigated, and the characterization of $\text{LaO}(\text{OH})_{0.55}\text{Cl}_{0.45}$, a monoclinic YOOH-type intermediate phase, is reported. Structural features and phase equilibria of the hydroxide chlorides are discussed and analogies with the hydroxide nitrate systems are drawn.

Positron Annihilation with Valence Electrons in Th_3As_4 and U_3As_4 . B. ROZENFELD, E. DEBOWSKA, Institute of Experimental Physics, University of Wrocław AND Z. HENKIE, Institute for Low Temperature and Structure Research, Polish Academy of Sciences, Wrocław, Poland. The angular distribution of annihilation photons (ADAP) for polycrystalline samples of the nonmagnetic semiconductor Th_3As_4 and the magnetically-ordered-at-lower-temperatures, semimetallic U_3As_4 are compared. The "tail" component (high angle part) in the ADAP curve for U_3As_4 , markedly higher than for Th_3As_4 , is supposed to be the result of annihilation with $5f$ electrons being localized at the uranium ionic core. The number of valence electrons per molecule calculated in the free-electron model approximation from the small angle part of the ADAP curve is equal to 31.7 ∓ 1.4 for Th_3As_4 and 32.7 ∓ 1.4 for U_3As_4 .