The Production of Sr2 VO4 for Neutron Scattering Experiment

Abstract: The purpose of this experiment is to successfully create an appreciable amount of Strontium Vanadate, Sr2 VO4, which will be significant enough for use in a neutron scattering experiment at ISIS. We are attempting to gain more knowledge about the orbital ordering transition. Magnetic susceptibility, x-ray diffraction, and heat capacity measurements were completed to investigate the spin ordering at ~97 K. Neutron scattering experiments will be completed at the ISIS research center near Oxford, using the HET spectrometer.

Strontium Vanadate, or Sr2 VO4, is an interesting compound in the family of layered perovskites. A key to a successful reaction as gas will be the other precipitate. The half-pipe of sample is then placed in a tube furnace under Hydrogen flow at 1323 K for 30 hours. At this time, the sample must be ground again and re-fired. After a third time, the sample has been fired for a total of 60 hours and the reaction is finished. The Sr4 V2 O9, a white, chalky powder now, is then distributed along a Alumina half pipe, keeping in mind that surface area is key to a successful reaction as gas will be the other precipitate. The half-pipe of sample is then placed in a tube furnace under Hydrogen flow at 1323 K for 30 hours. At this time, the sample must be ground and an X-ray is taken. Then the sample is re-fired. After a total of 90 hours, the Sr4 V2 O9 has turned from chalky white to jet black, and the transition to Sr2 VO4 is complete. There are many factors which play a part in this reaction, and if one of them is out of sync, the sample can be flawed. One of the most important of these factors is temperature. Because the temperature must be so high, only a small amount, about 0.3 grams, can be fired in the tube furnace at once, so that it can be contained in small space surrounding the heating implement of the furnace. This is the only area of the furnace where the temperature will remain hot enough to induce reaction with the H2 gas. Needless to say, this procedure had to be conducted several times in order to achieve the desired amount of sample.

The crystal structure of Sr2 VO4 and the 3d orbital levels for its V4+ ion.

Experimental Procedure

The Production of Sr2 VO4 is a two-step reaction, which takes precision and time. The chemical formulae are listed here:

(1) SrCO3 + V2 O5 + 2 SrO + 4 CO2
(2) Sr4 V2 O9 + H2O → 2 Sr2 VO4 + H2

The first step of the reaction, in which Sr4 V2 O9 is produced, is prepared by grinding the Strontium Carbonate and Vanadium Oxide with a mortar and pestle until mixed thoroughly. Then, the mixture is fired at 1073 K for 20 hours. At this point the powder is mixed again and re-fired. After a third time, the sample has been fired for a total of 60 hours and the reaction is finished. The Sr4 V2 O9, a white, chalky powder now, is then distributed along an Alumina half pipe, keeping in mind that surface area is key to a successful reaction as gas will be the other precipitate. The half-pipe of sample is then placed in a tube furnace under Hydrogen flow at 1323 K for 30 hours. At this time, the sample must be ground and an X-ray is taken. Then the sample is re-fired. After a total of 90 hours, the Sr4 V2 O9 has turned from chalky white to jet black, and the transition to Sr2 VO4 is complete. There are many factors which play a part in this reaction, and if one of them is out of sync, the sample can be flawed. One of the most important of these factors is temperature. Because the temperature must be so high, only a small amount, about 0.3 grams, can be fired in the tube furnace at once, so that it can be contained in small space surrounding the heating implement of the furnace. This is the only area of the furnace where the temperature will remain hot enough to induce reaction with the H2 gas. Needless to say, this procedure had to be conducted several times in order to achieve the desired amount of sample.

Diffraction occurs as wave interacts with a regular structure whose repeat distance is about the same as the wavelength. The phenomenon is common in the natural world, and occurs across a broad range of scales. For example, light can be diffracted by a grating having scribed lines spaced on the order of a few thousand angstroms, about the wavelength of light. It happens that x-rays have wavelengths on the order of a few angstroms, the same as typical interatomic distances in crystaline solids. Using x-ray diffraction, the structure of atomic compounds can be studied and categorized. For all of the experiments conducted here were recorded by a HUBER imaging plate Guinier camera 670 using Cu Kα radiation (1.5406Å) with a Ge monochromator. Data were collected in steps of 0.05° with temperatures down to 9 K obtained with a closed cycle He fridge.

These are X-ray diffraction patterns of Sr4 V2 O9 and Sr2 VO4 (left to right).