

The structure of $A_x\text{Nb}_{15}\text{W}_{13}\text{O}_{80}$, where $A = \text{Na}, \text{K}, \text{Ag}$, has been determined by a combination of electron diffraction, HREM imaging and single crystal X-ray diffraction. The structure is built up of pentagonal columns, which are linked to each other either directly or via MO_6 octahedra in such a way that large S-shaped tunnels are formed. In the tunnels the A atoms seem to be statistically distributed. (Marinder and Sundberg, *Acta Cryst.* Submitted.) The image contrast of different A atoms located in tunnels will be discussed and compared with calculated images. Computerized image processing can also be used to locate the heavy atom positions more accurately.

Some recent examples of compounds with structures related to that of tetragonal tungsten bronze will also be described.

14.4-9 ATOMIC CONFIGURATIONS IN DEFECTS, INTERFACES AND GRAIN BOUNDARIES OF SiC AND Si_3N_4 STUDIED BY HIGH RESOLUTION ELECTRON MICROSCOPY.⁴ By K. Hiraga and M. Hirabayashi, The Research Institute for Iron, Steel and Other Metals, Tohoku University, Sendai, Japan

High resolution electron microscopy is a powerful technique to observe microstructures of defects in crystals on the atomic scale. This technique was applied for studying the atomic arrangements in planar defects, interfaces and grain boundaries in ceramic materials as Si_3N_4 and SiC . The specimens were prepared by either chemical vapour deposition or sintering method. The high-resolution images with the end-on orientation made possible to determine directly the atomic arrangements in planar defects and grain boundaries, and in interfaces between the matrix and inclusions or substratum. As a result, we succeeded in observing two-dimensional high-resolution images of tilt grain boundaries with the common [110] rotation axis in the CVD SiC . The atomic fitting, coincidence-relationship and symmetry at boundaries and interfaces were analyzed directly from the images of the adjoining grains.

14.4-8 TIME RESOLVED ANALYSIS OF HIGH RESOLUTION ELECTRON MICROSCOPE IMAGES.* By A. Holladay and L. Eyring, Department of Chemistry, Arizona State University Tempe, Arizona 85287 U.S.A.

A comparator system has been developed which allows the direct comparison of images calculated from a structural model and experimental images obtained by digitizing electron microscope negatives with a microdensitometer. This system provides several quantitative measures of the agreement between experimental and calculated images including a fractional mean average deviation which is closely related to the R factor used in X-ray crystallography. One example of the usefulness of the comparator has been the analysis of several phases of praseodymium oxide. These materials undergo electron beam-induced reduction within the electron microscope. A number of intermediate phases have been examined but a method for analyzing such reactions in situ to deduce mechanisms of reduction and identify intermediate states as they are formed would be highly desirable.

In order to accomplish this goal a JEOL 200CX electron microscope has been modified to permit videotape recording of live sessions when desired. An image processing system constructed to allow the digitization and image enhancement of individual frames from the videotape provides the ability to analyze time resolved high resolution images. This new facility will be used to monitor the in situ reduction of PrO_2 in the electron microscope. After the session is recorded the videotape will be used as the source of experimental images of the intermediate phases evolved in the electron microscope for comparison with images calculated from structural models. A description of the hardware and software comprising the comparator as well as its application to the praseodymium oxide system will be presented.

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