14.4–5 HIGH RESOLUTION SURFACE IMAGING. By L.D. Marks, Dept. of Physics, Arizona State University, Tempe, AZ 85287, U.S.A.

With the advent of high resolution electron microscopes with good signal-to-noise characteristics below 2Å, it has recently proved possible to directly resolve surface structure on metals (Marks, L.D. and Smith, D.J., Nature 303 316 (1983)). When coupled with detailed image simulations (Marks, L.D., Surface Science in press), local information including direct measurement of surface relaxations (Marks, L.D., Physical Review Letters 51 1000 (1983) and Figure 1) and inhomogeneous surface strains (Marks, L.D., Heine, V. and D.J. Smith, submitted to Physical Review Letters) has proved possible. The required conditions for obtaining this information are very localised electron-optical imaging (Marks, L.D., Ultramicroscopy in press) and a column approximation for the dynamical diffraction. The latter depends upon a good zone axis orientation, and can be explained (Marks, L.D., submitted to Acta Cryst) by a swift electron analysis based upon spherical wave X-ray diffraction theory (e.g. Pinsker, Dynamical Scattering of X-rays in crystals (1978) and Azaroff et al, X-ray diffraction (1974)).

In this paper some of the recent results of high resolution imaging of gold surfaces will be described, concentrating upon the importance of local strains and surface dislocations, and the necessary conditions for the diffraction to obey a column approximation based upon an electron wavepacket scattering model briefly described.

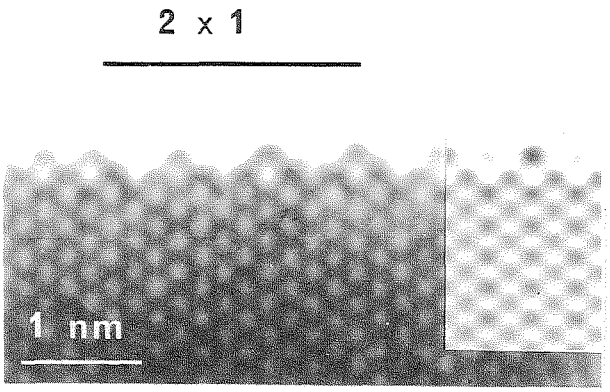

Figure 1 Experimental image of an Au(110) surface showing local 2x1 reconstruction with a numerical image simulation to the right (see Marks, L.D., Phy Rev Lett 51, 1000 (1983).

14.4–6 HIGH RESOLUTION ELECTRON MICROSCOPIC STUDY OF V_6O_{13}. By T. Ohno, Y.Nakamura and S. Nagakura, Department of Metallurgy, Tokyo Institute of Technology, Oh-okayama, Meguro-ku, Tokyo 152, Japan.

Structure images of V_6O_{13} (monoclinic, C2/m, a=11.922, b=3.680, c=10.138 A and β=100.87°) were taken with the aid of Ultra-High-Vacuum High-Resolution 1 MV Electron Microscope with the cut-off resolution 1.4 A. The specimen was prepared by reducing a powder mixture of V_2O_5 and V_2O_3 in hydrogen at 670°C for 3 days, and crushed in acetone to make thin flakes for observation. In the structure image taken along the b axis, indivisual V atom rows as well as tunnels could be resolved clearly, although O atom rows did not give image contrast. The atomic scattering factor depends strongly on the electron state of atoms in the low scattering angle region. By using this fact, the effect of electron state on the structure image was investigated by comparing the observed images and simulated images produced by giving various electron states to the constituent atoms. It was shown that the neutral model $V_6^0O_{13}^0$ explained more satisfactorily the image contrast than the ionized model $V_2^{4+}V_2^{5+}V_2^{4+}O_{13}^{2-}$. This is in accordance with the previous electron diffraction result on V_7O_{13} (Y. Hirotsu, H. Sato and S. Nagakura, Modulated Structures-1979 : AIP Conf. Proc. No. 53, p. 75). A modification of V_6O_{13} was found, and its structure was analysed. This is orthorhombic with a=11.922, b=19.912 and c=3.680 A and belongs to the space group Cmma. Approximate atom positions were determined. This structure can be derived from the structure of V_2O_5 by removing all the atoms on every third (001) oxygen atom plane and then introducing the crystallographic shear $(1/2)[0\bar{1}\bar{1}]$, instead of $(1/6)[\bar{1}03]$ in the case of normal V_6O_{13}.

14.4–7 THE STRUCTURES OF SOME COMPLEX OXIDES CONTAINING NIOBIUM FROM HIGH RESOLUTION ELECTRON MICROGRAPHS. By M. Sundberg, Department of Inorganic Chemistry, University of Stockholm, Stockholm, Sweden.

The performance of modern high resolution electron microscopes enables a point-to-point resolution of 2-2.4 Å. In many materials the separations between heavier atoms in the structure can thus be resolved, and micrographs recorded under certain conditions can be interpreted on the basis of a reliable structure model. However, the interpretation of an image should be checked by matching observed and calculated images. It is also important to consider the chemical soundness of the model. For example, without reasonable assumptions about the coordination of the heavy cations, it is difficult to find a plausible distribution of light anions.

Some examples of complex oxides will be presented which illustrate HREM image interpretation of crystal and defect structures at the atomic level. All these phases have one short unit cell axis which is a favourable line of projection.

The crystal structure of $NaNb_7O_{18}$ has been derived from HREM images and verified by multislice calculations of simulated images. Later, a refinement from X-ray powder data was made. The structure is related to that of $NaNb_{13}O_{33}$. Sodium atoms are located in the tunnels. (Marinder and Sundberg, Acta Cryst. In press.)

The structure of $A_xNb_{15}W_{13}O_{80}$, where A = Na, K, 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.

14.4–8 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 comparitor 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 comparitor 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 comparisor with images calculated from structural models. A description of the hardware and software comprising the comparitor as well as its application to the praseodymium oxide system will be presented.

* Research supported by the U. S. National Science Foundation under Grant DMR-8108306.

14.4–9 ATOMIC CONFIGURATIONS IN DEFECTS, INTERFACES AND GRAIN BOUNDARIES OF SiC AND Si_3N_4 STUDIED BY HIGH RESOLUTION ELECTRON MICROSCOPY. 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.