MicroDiffraction in the Scanning Electron Microscope (SEM)

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ABSTRACT

The identification of crystallographic phases in the scanning electron microscope (SEM) has been limited by the lack of a simple way to obtain electron diffraction data of an unknown while observing the microstructure of the specimen. With the development of Charge Coupled Device (CCD)-based detectors, backscattered electron Kikuchi patterns (BEKP), alternately referred to as electron backscattered diffraction patterns (EBSP), can be easily collected. Previously, BEKP has been limited to crystallographic orientation studies due to the poor pattern quality collected with video rate detector systems. With CCD detectors, a typical BEKP can now be acquired from a micron or sub-micron sized crystal using an exposure time of 1-10 seconds with an accelerating voltage of 10-40 kV and a beam current as low as 0.1 nA.

Crystallographic phase analysis using BEKP is unique in that the properly equipped SEM permits high magnification images, BEKP's, and elemental information to be collected from bulk specimens. BEKP in the SEM has numerous advantages over other electron microscopy crystallographic techniques. The large angular view (~70 degrees) provided by BEKP and the lack of difficult specimen preparation are distinct advantages of the technique. No sample preparation beyond what is commonly used for SEM specimens is required for BEKP.

INTRODUCTION

The identification of unknown micron-sized phases in the SEM has been limited by the lack of a simple way to obtain crystallographic information about the unknown while observing the microstructure of the specimen. A variety of techniques are available that can provide some information about the identity of unknown phases. For example, energy dispersive x-ray spectrometry (EDS) is of some use but obviously cannot distinguish between phases of similar compositions but different crystal structures (an example of this is TiO_2 that has two tetragonal structures with different atomic arrangements and an orthorhombic structure). Other techniques can provide the required information but have significant limitations. Micro area x-ray diffraction techniques are capable of identifying crystalline phases, but cannot be used to identify sub-micron sized areas. Selected area electron diffraction in the transmission electron microscope (TEM) can provide crystallographic information from sub micron-sized regions of the specimen, but TEM requires electron transparent thin specimens to be produced which is time consuming and

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can be very difficult. The first application of BEKP's for phase analysis was to identify symmetry elements and crystal structure in the SEM [1,2,3,4,5]. Phase identification via this approach required a high degree of crystallographic symmetry expertise. In this paper we demonstrate that BEKP in the SEM using a charged couple device (CCD)-based detector can provide crystallographic phase identification of sub-micron sized areas with little or no difficult specimen preparation nor the extensive expertise required previously. The details of the detector and its calibration are described elsewhere [5,6,7,8,9]

Theory

BEKP's are obtained by focusing the incident electron beam on the feature of interest. The electron beam is held stationary and the specimen is tilted, normally about 70 degrees, toward the detector. The highly divergent inelastically scattered electrons are diffracted into flat cones with an apex angle of 90-Θ[10]. Because the wavelength of 20-40 kV electrons is small, the Bragg angle Θ is less than 2 degrees. These flat cones intersect the detector revealing nearly straight Kikuchi lines. Two Kikuchi lines are formed for each diffraction condition because both $+\Theta$ and $-\Theta$ form cones. The resulting Kikuchi pairs are separated by 2Θ. Zone axes are readily observed where two or more Kikuchi line pairs cross. The angular separation of the zone axes relate directly to the angular separation calculated from the crystal structure. The angle formed by the intersection of Kikuchi pairs also relates directly to the angles calculated from the crystal structure [11]. We observed that the apparent intensities of the Kikuchi lines are proportional to the structure factor squared and do not vary significantly as the crystal orientation is changed. this insensitivity to crystal orientation is an important property that makes BEKP useful for phase identification and crystallographic orientation determination [12, 13].

Since the Kihuchi pattern is formed from the intersection of cones on a flat detector parallel to the beam direction, the pattern is distorted in the resulting gnomic projection [14]. In order to correct for the resulting distortion, the Kikuchi pattern center and the specimen to detector distance must be determined accurately. The pattern center is the projection of the electron beam impact point onto the detector scintillator. Finally to obtain high quality imaging, a flat-fielding procedure is used [7].

Experimental

The procedure followed for phase identification in the SEM is illustrated in Figure 1. Fist, a feature of interest is identified in the SEM. The electron beam is then focused on this area. An energy dispersive spectrometer (EDS) is used to determine the elemental constituents from this feature. At the same time or immediately following the collection of the EDS spectrum, the BEKP pattern is collected. The interplanner spacings and crystal plane angles are extracted from the BEKP by a Hough transform algorithm. This

information is used to derive the reduced cell volume. In some cases, this calculation results in a sub cell volume. The subcell is related to the actual reduced cell volume by an integer. The subcell volume along with the elemental information is used to search the ICDD'd powder diffraction data base. The software takes the results of this search and attempts to assign (hkl)'s to the Kikuchi lines that are consistant with the observed (hkl)'s from the pattern. Once a candidate phase is identified, its Kikuchi pattern is simulated and overlaid onto the observed pattern. If more than one candidate phase is found the microscopist can page through all of the possibilities and observe the best choice. It has been our experience that the correct choice is normally obvious. This match mode provides a very powerful verification tool for the analyst.

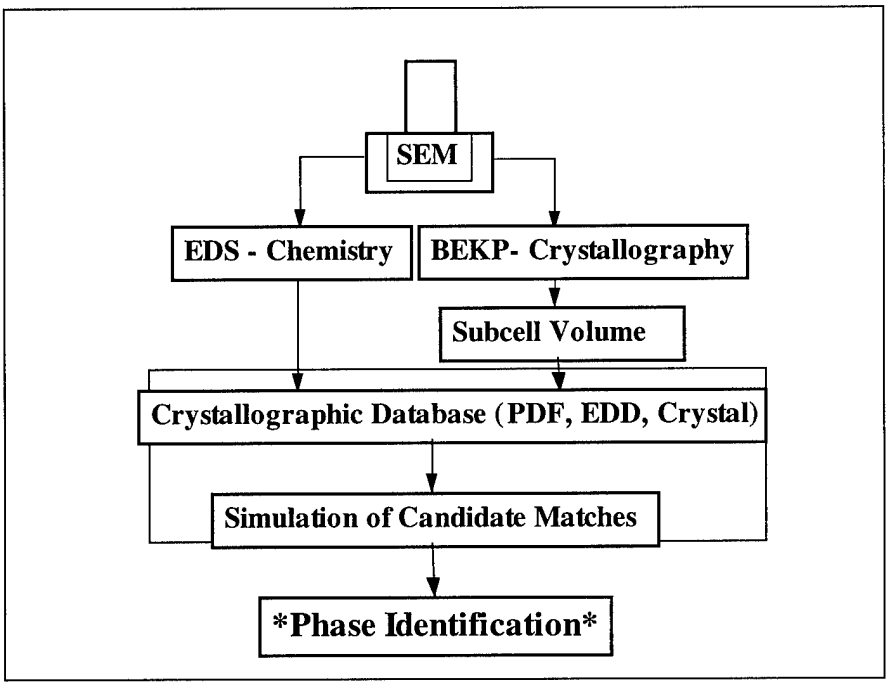

Figure 1
Diagram of phase identification process in the SEM

In order to illustrate the power of this technique, an example of the analysis of solder joint crystallographic phases is used. Figure 2a shows an SEM micrograpgh of the AgSn solder. In particular notice the needle like phases. The width of these needles is ~ 0.1 micron and their length is ~10 microns. Figure 2b is the BEKP pattern obtained from the needle phase after flat-fielding the original image.

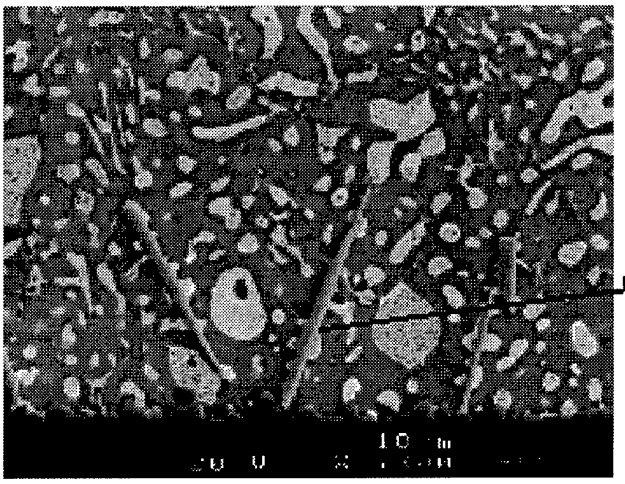

Figure 2a. SEM Micrograph

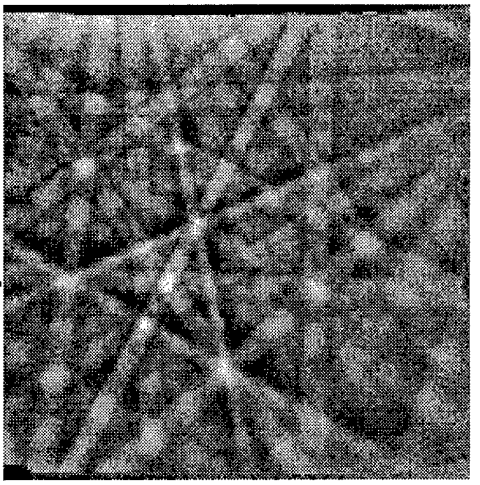

Figure 2b. BEKP from needle like phase

The BEKP pattern obtained from this specimen demonstrates that a suitable pattern for phase Identification can be obtained from ~0.1 micron regions.

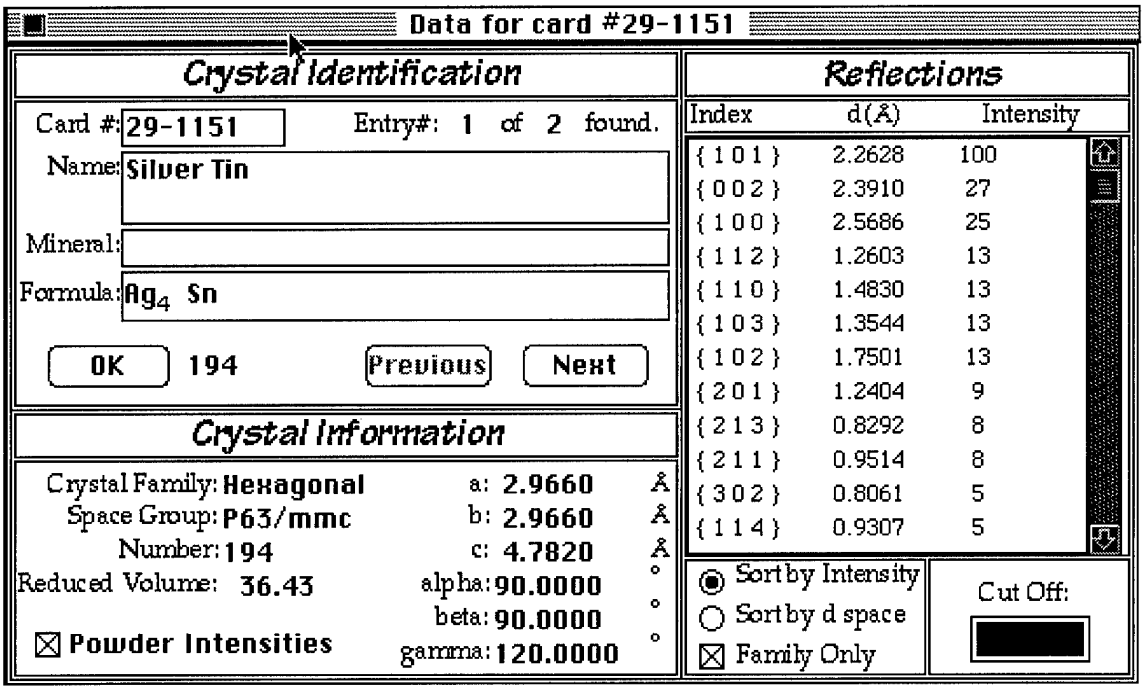

Figure 3. Shows PDF data

Figure 3 shows the results of the ICDD data base search [15]. The phase Ag_4Sn card # 29-1151 is the only phase that fulfilled the elemental and reduced cell volume constraints, and could be indexed. The phase Ag_3Sn does satisfy the chemistry constraints but the experimental BEKP would not index. Figure 4 shows the simulated pattern overlaid directly on to the experimental pattern. The indiceswith the square bracke designate zone axes (directions). In addition, the program can show the results of the indexing procedure

by drawing the Kikuchi lines used to index the pattern directly on the observed BEKP. It can also illustrate the method used by the program to measure the distance between Kikuchi lines and thus calculate the interplanner spacings.

Summary

We have successfully utilized backscatteres electron Kikuchi patterns for crystallographic phase analysis in the SEM utilizing the ICDD powder diffraction data base with over 40,000 inorganic phases. The successful determination of the phase is straight forward and does not require a trained crystallographer in most cases. This technique can now be used where previously microdiffraction and transmission electron microscopy (TEM) were the only choices.
Sandia has licensed the software to NORAN to be used with their Phase ID system.

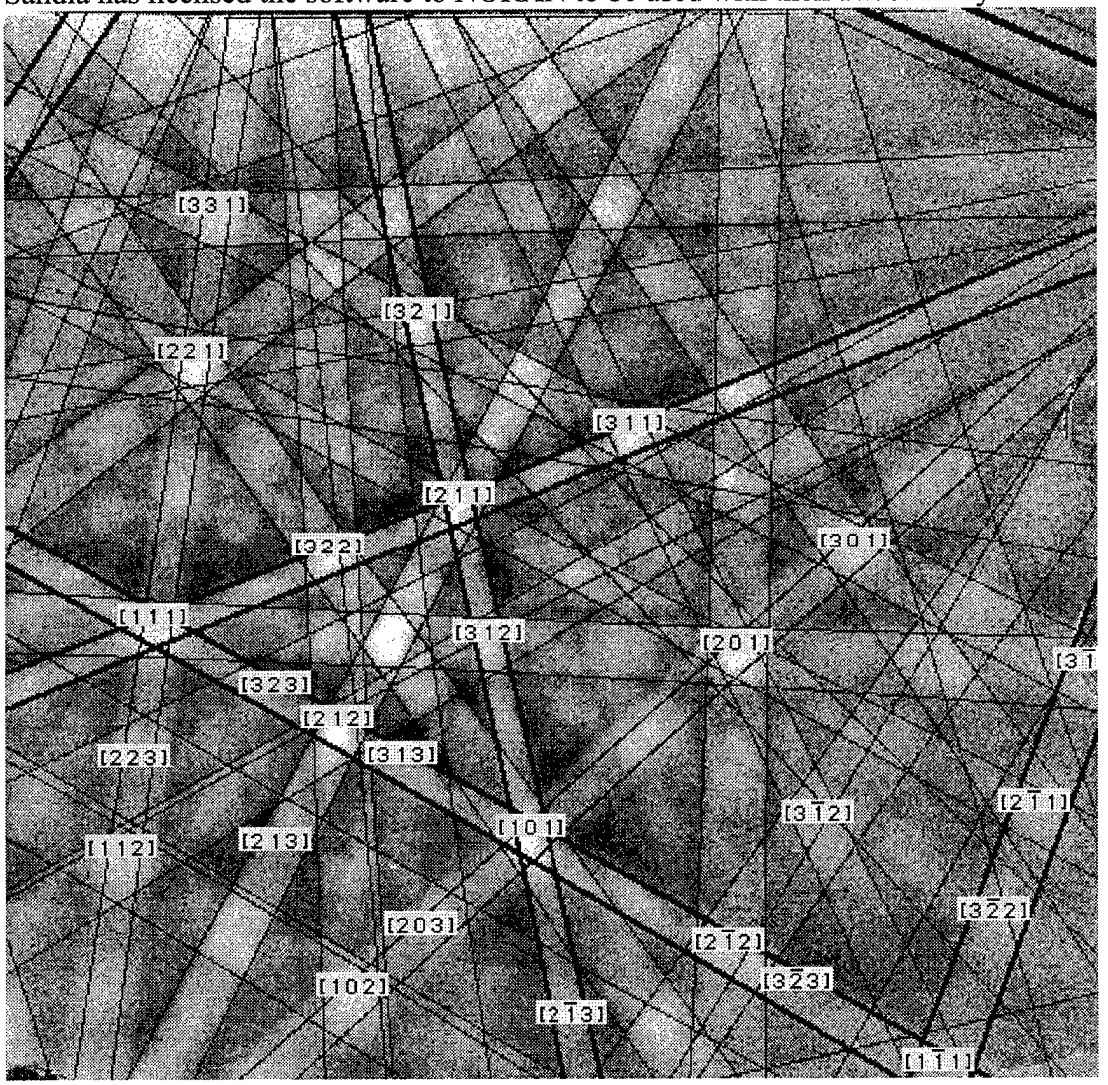

Figure 4. Simulated pattern overlaid directly on experimental pattern

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