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ORIENTATION DETERMINATION BY EBSP IN AN ENVIRONMENTAL SCANNING ELECTRON MICROSCOPE

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Introduction

Remarkable progress has been made in the single orientation measurement of polycrystalline materials using scanning electron microscopy (SEM) (1,2,3). The automation of this technique in the form of Orientation Imaging Microscopy (OIM) provides materials scientists with a reliable technique of texture and micro-texture characterization of materials at high vacuum conditions (4,5). The technique is based on the acquisition of Electron Backscattered Diffraction Patterns (EBSP) in the chamber of an SEM. These diffraction patterns are formed in the same manner as Kikuchi patterns in the TEM, but result from backscattering of electrons out of the top surface of the sample. Hence they can be obtained from bulk samples. In this paper we will discuss some of the unique capabilities provided by EBSP analysis at elevated pressures and temperatures in an Environmental Scanning Electron Microscope (ESEM). It was possible to obtain patterns with good quality for water vapor pressures as high as 6 Torr for single crystals, and 3 Torr for polycrystalline materials (NiAl grains with diameter up to 64 microns were examined). With increasing pressure, polycrystalline diffraction patterns degrade at a much higher rate than single crystal patterns.

Methodology

EBSPs and OIM have been applied to numerous materials systems to obtain better understanding of the microstructure (3,6,7,8,9,10,11,12,13,14). All of this research has been carried out in the ambient-temperature, high-vacuum environment of conventional SEMs. The use of an Environmental Scanning Electron Microscope (ESEM) makes it possible to acquire the lattice orientation information at low-vacuum and other environmental conditions (15). This also provides the opportunity to measure orientations of non-conducting and semi-conducting materials. The spatial resolution of EBSPs is determined by the excitation volume of the electron beam and therefore depends on accelerating voltage, probe current, working distance, and specimen tilt angle (16,17). By indexing successive patterns from hundreds of selected points on the sample surface, sufficient data can be collected to determine both macroscopic texture and local orientations and to provide a detailed survey of nearest neighbor orientation relationships.

This research was carried out using an ElectroScan ESEM (purchased from ElectroScan, Wilmington, MA) operating at 25kV, and OIM software (purchased from TSL, Provo, Utah) was used to index the EBSPs and for OIM (18). The water vapor environment used during imaging permits the orientation

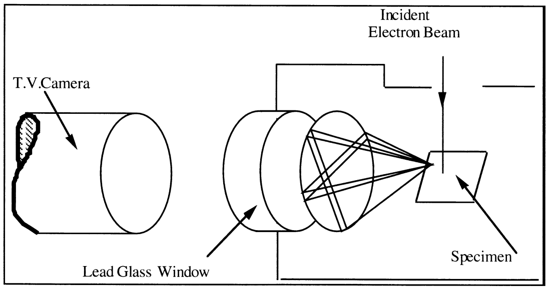

Figure 1. Schematic of EBSP technique.

of non-conducting specimens to be investigated as easily as conducting ones. A specimen stage designed to hold a specimen in a 1 inch diameter mount tilted 70° to the horizontal was used for the collection of patterns at increasing pressure. For the experiments at high temperatures, a heating stage (purchased from ElectroScan) was inclined 70°. To form an EBSP, a stationary electron probe is placed on a single-crystal region of the specimen, and the diffraction pattern is imaged on a phosphor screen placed close to it, as illustrated in Figure 1. For the experiments described below, the distance between the screen and the specimen was ~2.2cm. The phosphor screen is viewed through an optical port using a high gain television camera which is interfaced to a Silicon Graphics Indigo computer. The camera control unit can perform frame averaging up to 128 video images of the pattern. The final image is enhancement is accomplished by dividing the averaged pattern image by a background image obtained by scanning over a large number of grains in a polycrystalline specimen or by integrating over several frames of a defocused pattern from a single crystal specimen. We used averaging/integrating over 64 frames in the collection of each pattern. The EBSPs were recorded in the computer and indexed. (The camera and the control unit were both purchased from TSL, Provo, Utah.)

For each indexed pattern, an image quality (IQ) parameter that represents the sharpness of the EBSPs is determined (6). The IQ is measured from the Hough transform of the gray scale information through the Kikuchi patterns. Similarly a 2_D Fourier transform can provide the same information (9). Under high vacuum, the image quality is a function of atomic number, strain level in the sample, and the quality of the specimen surface (9,12,13). The image quality will be used in the following discussion to compare patterns collected under different environmental conditions.

In OIM, the EBSPs are collected from points on the sample surfaces distributed over a regular grid and automatically indexed. From this data, a map, called an Orientation Image Micrograph, can be constructed displaying changes in crystal orientation or image quality over the specimen surface. In the Orientation Image Micrograph, the orientation of each point in the microstructure is known and hence the location, line length and type of all boundaries. Figure 2 shows an OIM micrograph of NiAl obtained by mapping the information (IQ and the grain boundary lines) obtained from 11500 data points. A misorientation criteria of 5 degrees is used to draw lines distinguishing grain boundaries. The gray scale indicates the image quality with high image quality represented by light gray and low as dark gray. The corresponding pole figures formed from the individual orientations are presented in Figure 3. These two figures present two extremely important applications of this technique in the materials science community: microtexture analysis and grain boundary characterization.

The effect of increasing water vapor pressure on the quality of EBSPs was investigated for polycrystalline NiAl and single crystal silicon (International Wafer Service, Portola Valley, CA), $SrTiO_3$ (Commercial Crystal Laboratories, Naples, FL), and KBr (International Crystal Labs, Garfield, NJ). The NiAl was sectioned from a cast, extruded and annealed specimen, mechanically polished to 0.3 micron, and electropolished in a solution of 5% perchloric acid in methanol. Orientation Image

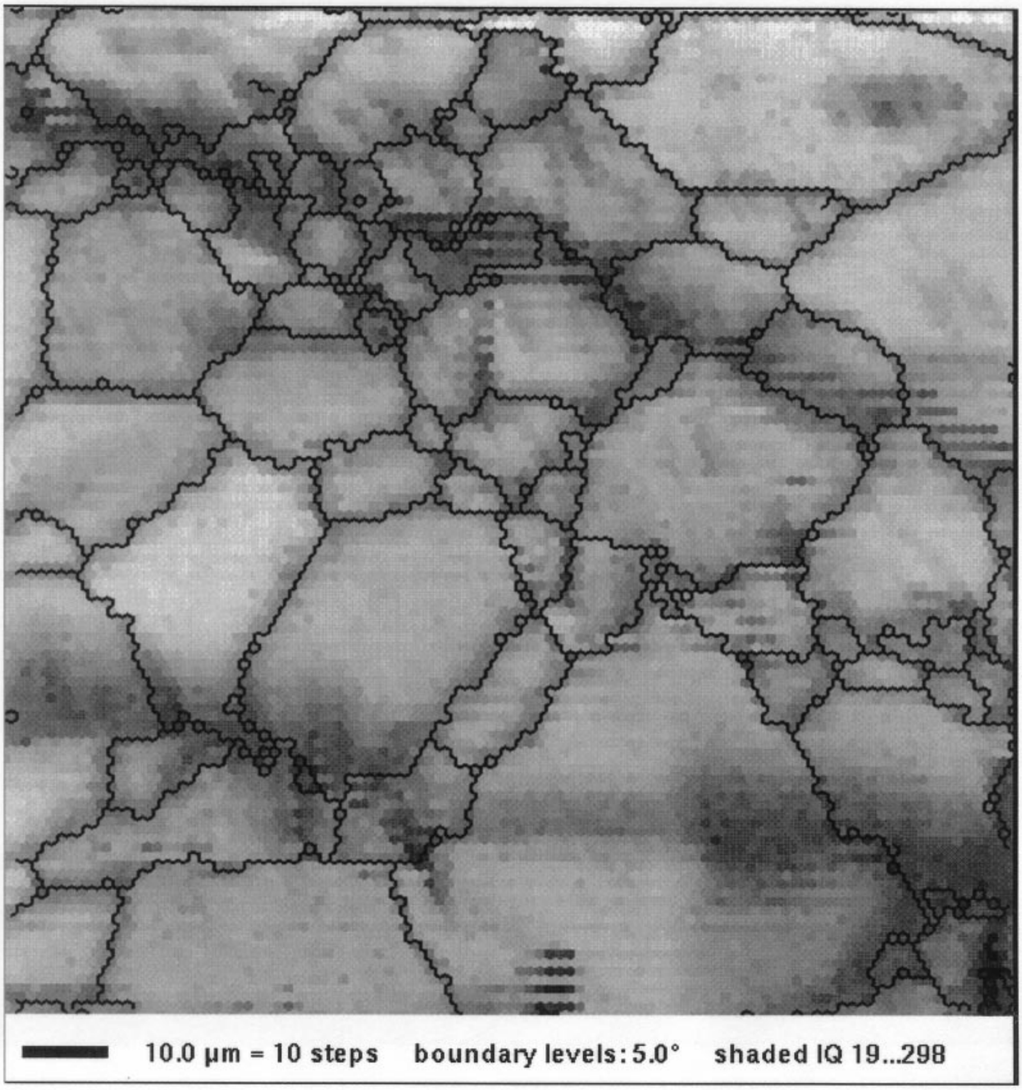

Figure 2. OIM map of NiAl.

Micrographs were collected for the NiAl specimens to determine the size of the grains used for the analysis. The KBr crystal was cleaved immediately before placing it in the ESEM, and the silicon and $SrTiO_3$ were used as received. The influence of heating on single crystal silicon patterns was considered for pressures of 1 Torr and 3 Torr for temperatures up to 650°C.

Results and Discussion

Figures 4 and 5 show the variation of image quality with increasing pressure for single crystal $SrTiO_3$ and a grain in a NiAl specimen (grain diameter = 130 microns). The image quality is plotted against chamber pressure in Figure 6 for polycrystalline NiAl (grain diameter = 29 microns) and single crystal

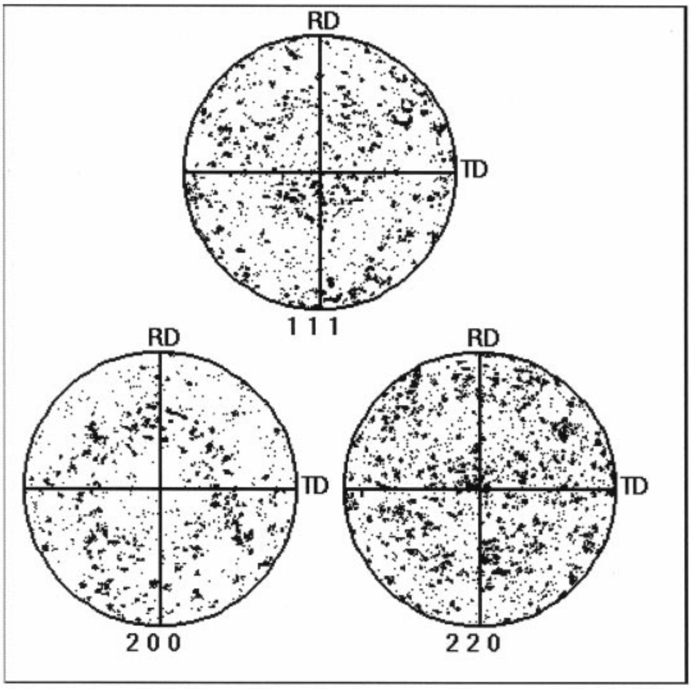

Figure 3. Pole figures for NiAl obtained from discrete orientations measured by OIM.

silicon, KBr, and $SrTiO_3$ specimens. Useful patterns were observed over a range of pressures, however the quality of the patterns decreases as the pressure increases. Pattern quality during heating was examined for pressures of 1 Torr and 3 Torr. Single crystal silicon EBSPs obtained during heating to 650°C at pressure P = 1 Torr are shown in Figure 7. Very little degradation of the pattern was observed.

Operating at elevated water vapor pressures eliminates the problems associated with charging of insulating materials and demonstrates the ability to investigate the orientations of materials in different environments. While the image quality decreases with increasing pressure, patterns with image quality high enough to be indexed by the OIM software were obtained for pressures up to 5.5 Torr for single crystal silicon, 6.0 Torr for $SrTiO_3$, and 4.5 Torr for KBr. Decreasing the distance between the specimen and the phosphor screen will decrease the amount of scattering of the diffracted electrons by the water vapor molecules, improving the ability to obtain useful patterns at elevated pressures.

Although the image quality for polycrystalline NiAl patterns was higher than single crystal patterns at pressure less than 1 Torr, the image quality decreases more quickly, and patterns could only be obtained for pressures up to 3 Torr. This may be due to scattering of electrons by water vapor particles before the probe reaches specimen. Scattered electrons may be diffracted from different grains than the focused probe electrons, leading to a decrease in the pattern quality. The impact of this scattering on EBSP quality is therefore expected to increase as grain size decreases. The ability to observe EBSPs from polycrystalline specimens at increased pressures may be improved by decreasing the path the electron probe travels through the water vapor environment before reaching the specimen.

The image quality may vary for different specimens with the same composition as revealed by comparing the quality of the patterns obtained from silicon during the high pressure experiments with the ones obtained during the heating experiments. These specimens were taken from different wafers

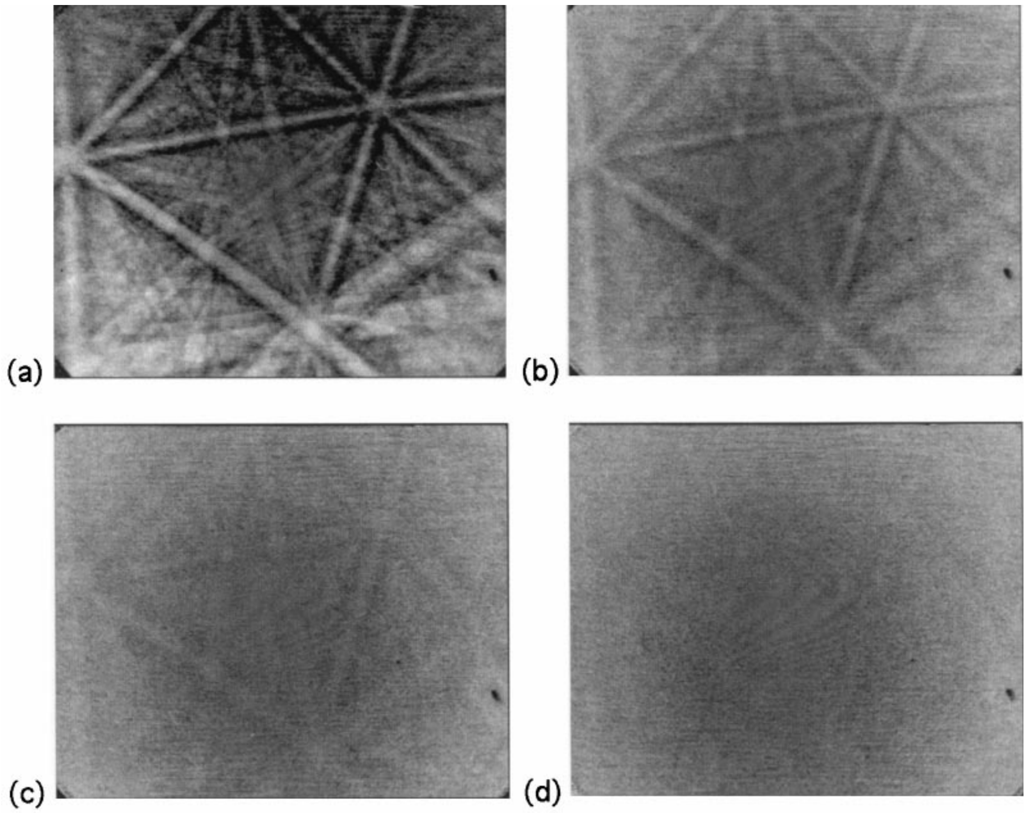

Figure 4. $SrTiO_3$ patterns collected at increasing pressure. (a) P < 1 Torr, IQ = 428.83, (b) P = 2 Torr, IQ = 185.80, (c) P = 4 Torr, IQ = 80.80, (d) P = 6 Torr, IQ = 38.17.

and the variation in image quality is due to other factors that influence pattern contrast such as differences in surface cleanliness or oxide thickness.

Conclusion

This ability to investigate grain orientations at elevated temperatures and water vapor pressures suggests many new possibilities for investigating corrosion and other environmental impacts on advanced materials. Orientation Imaging Microscopy at high temperatures can also facilitate the investigation of such processes as recrystallization, grain growth, and growth of second phase materials and their texture and volume fraction.

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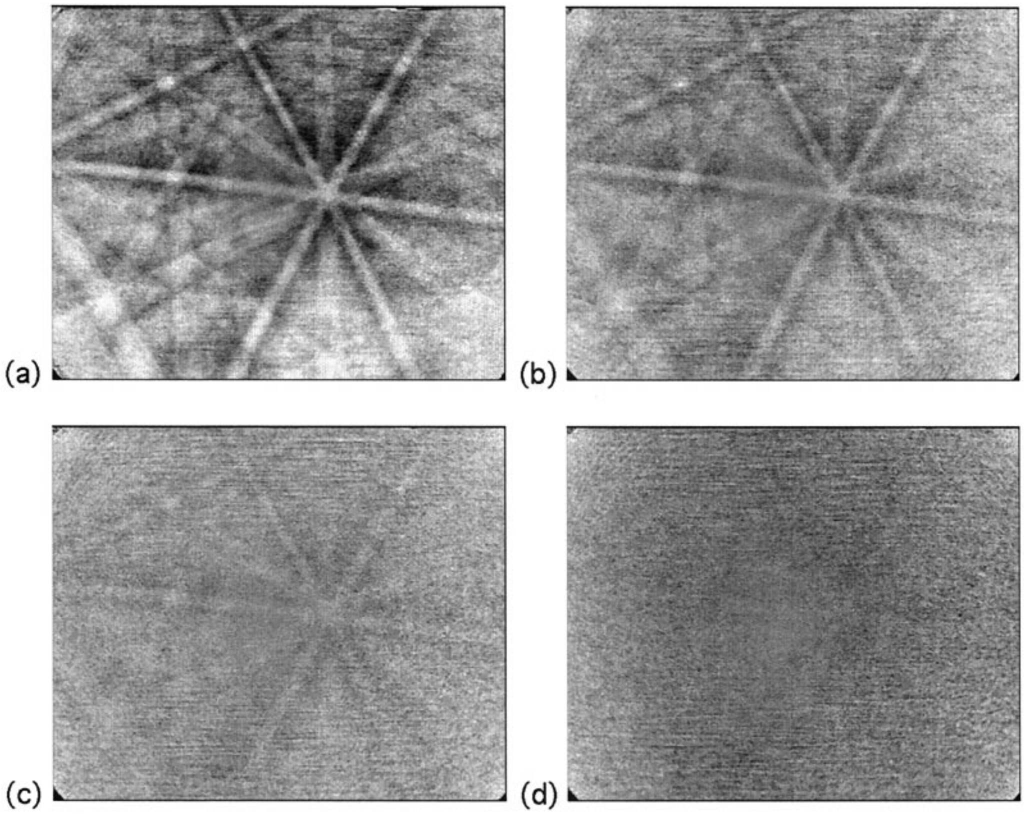

Figure 5. NiAl patterns collected at increasing pressure. (a) P < 1 Torr, IQ = 444.65, (b) P = 1 Torr, IQ = 246.03, (c) P = 2 Torr, IQ = 115.21, (d) P = 3 Torr, IQ = 50.60.

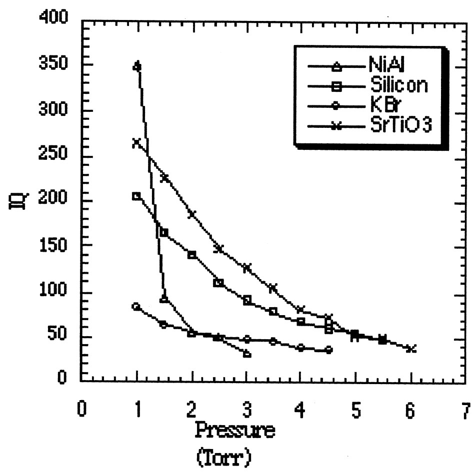

Figure 6. Plot of EBSP image quality vs. chamber pressure

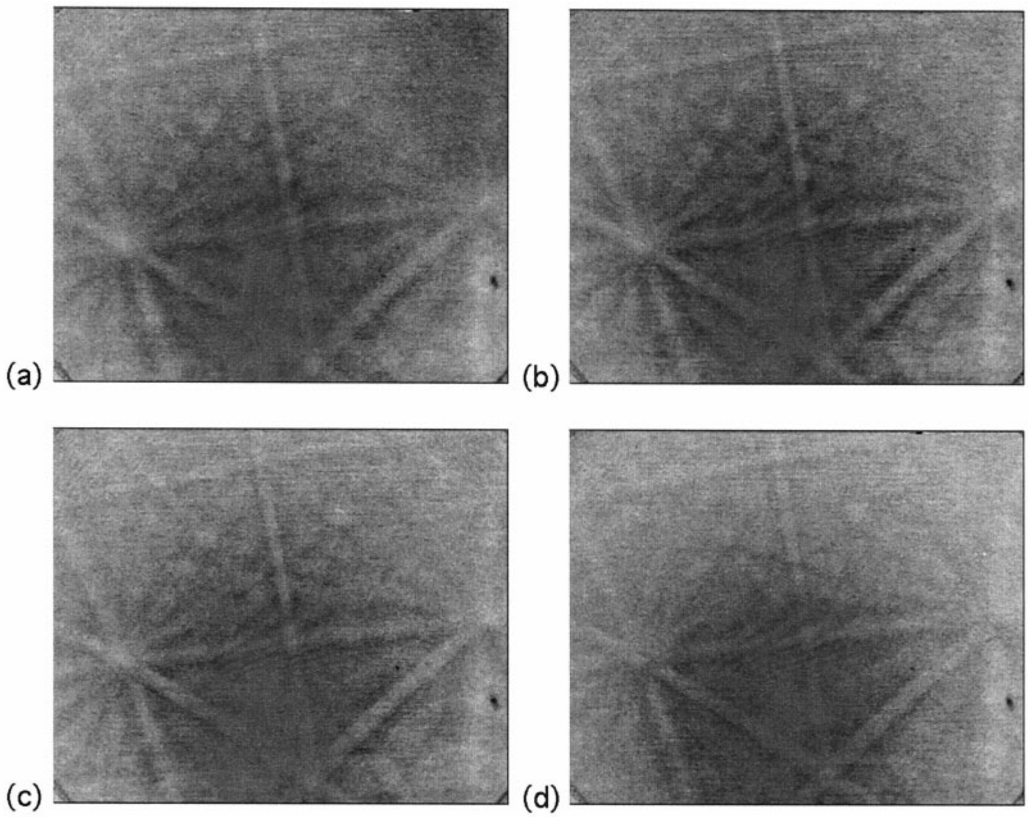

Figure 7. Silicon EBSPs collected during heating at P = 1Torr. (a) T = 22°C, IQ = 114.55, (b)T = 233°C, IQ = 125.30, (c) T = 461°C, IQ = 125.04, (d) T = 650°C, IQ = 93.45.

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