MSEG 624

Practical Electron Microscopy

Lab 3: X-ray Energy Dispersive Spectroscopy

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Abstract

Here, single crystal Au and polycrystalline evaporated Al samples are imaged under a Transmission Electron Microscope (TEM). Resulting spot and ring diffraction patterns are obtained, analyzed, and indexed – with discussion of pertinent theory. Bright and dark field images are obtained of the polycrystalline evaporated Al sample, and their opposing contrast regions are explained with reference to practical TEM operation.

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Introduction

A Transmission Electron Microscope (TEM) is an important and powerful tool in the modern scientific laboratory. As compared to its Scanning Electron Microscope counterpart, TEM utilizes electrons transmitted through a thin sample to provide information characteristic of its internal composition – at high resolution. The electron beam diffracts upon interaction with the internal structure of the sample as the electrons are transmitted through it, and provides diffraction patterns that can be used to characterize those structures. Selected Area Electron Diffraction (SAED) is a technique by which the TEM user selects a controlled specimen region (often controlled by diffraction aperture) over which to examine the diffraction pattern produced. For a single crystal samples, this will produce an array of illuminated dots, as each point discretely satisfy diffraction condition. In a sufficiently polycrystalline sample, a signal of sequenced, concentric rings will be produced as a result of fully varied crystallographic orientation within the sample. Bright field and dark field imaging can be accomplished by utilizing the aperture to select only the incident beam or only diffracted rays, respectively.

Experimental Methodology

In the experiments conducted, electron diffraction from an Au single crystal sample and an evaporated Al polycrystalline sample were investigated and imaged under various operating parameters of the JEM-3010 Transmission Electron Microscope (TEM). Images and their corresponding imaging parameters were recorded under these conditions, and were later used to evaluate and draw conclusions from. The provided procedure entitled *TEM Laboratory Exercise No.1 – SAED, BF, DF* [1] was followed with the aid and direction of laboratory Research Associate Thomas Barkley, changing only the order in which the samples were analyzed.

The Au and Al samples were prepared for imaging beforehand, and placed into their sample holders prior to their imaging sessions (double-tilt and single-tilt, respectively). To image

one of the samples, the sample was placed onto a fine grid and loaded onto the holder, and then entered into the chamber. The vacuum switch was engaged, and the vacuum gages were monitored until it stabilized at the desired vacuum – indicated by a green light by the switch. The JEM-3010 TEM was operated at an accelerating voltage of 300kV for both samples.

Single Crystal Au

To begin analysis of the single crystal Au samples, a low magnification setting was first selected to locate a suitable region of the sample. This region was centered by utilizing the shift knobs, and the magnification was then increased. The eucentric focus was then found and applied as the optimum objective lens current, a DV value of 0 on the screen. The sample stage height was then changed to focus the image until contrast was at a minimum. At this height, the sample disappears on the screen – indicating the focal point. The diffraction aperture was then inserted, and reasonably centered. Diffraction mode was pressed, and a diffraction pattern then appeared on the screen. The camera length was then set to fill the field of view. The sample was then tilted to find a major zone. The aperture was taken out, as beam converges in the middle of the screen. Switch to mag and diffraction modes, tilt the sample until Kikuchi lines converge at a center point and are symmetric – indicating a major zone diffraction pattern. The beam was spread out and the aperture was replaced. A camera length of 30 cm was chosen as it was said to work best with the CCD. The beam stop was inserted to protect the camera, and the fluorescent screen was removed. An image was then captured by the CCD. The diffraction pattern can be indexed by measuring the relative distance between respectively oriented points in the diffraction

pattern; this was done by using a pair of digital calipers to take measurements of a digital image of the pattern.

Polycrystalline Evaporated Al

The LaB_6 crystal was powered down slowly before sample exchanges, as to avoid potential damage to the crystal. A single-tilt sample holder was used for the polycrystalline Al sample, as the crystals are already in different orientations and there is no need to tilt. Again, an acceptable region of the sample was located at low magnification, and the same steps were taken to focus the image and center the beam. The largest diffraction aperture was chosen as to include more crystals in the imaged region. The same camera length was used, and the CCD was viewed to inspect how ensure enough crystals were contained inside the aperture range, and indeed there were. The beam stop was centered, diffraction mode was entered, and the diffraction pattern was centered using projector shift. Diffraction focus was adjusted to make the rings sharper, and an image was then captured with the CCD. The diffraction pattern can be indexed for Miller indices by measuring the relative radii of the rings in the diffraction pattern; this was done by using a pair of digital calipers to take three sampled measurements of each radius, which were then averaged.

Bright Field Imaging

Next, the objective aperture was inserted and centered to block the diffracted electron rings. MAG1 was again selected to return to image mode, with the dark regions of the image

being diffracted electrons which satisfy Bragg's Law. Without the aperture, the same effect would be seen to a much lesser extent, as only the electrons blocked would be those that are diffracted at such an angle that they are blocked by the column itself. The brightness knob was adjusted, the image focused, and the beam stop was placed. An image was then recorded.

Dark Field Imaging

Diffraction mode was then selected and Dark Tilt option was selected to have the beam come down at an angle, off the optic axis. Dark tilt deflector was selected and X and Y deflector knobs were adjusted to tilt the beam to select a portion of diffracted electrons through the aperture. Imaging mode was entered, and brightness was adjusted before recording an image. A slightly longer exposure time was utilized for dark field imaging, as less electrons are present in these diffracted regions as selected – resulting in a weaker relative signal.

Results and Discussion

From the single crystal Au sample investigated in this lab, three of the resulting diffraction spots were measured using the technique described above. The calculated ratio of these relative measurements is calculated to be $R_3/R_1 \approx R_3/R_2 \approx 1.4$ which reinforces the notion that the single crystal Au sample is of FCC structure (Table 1). These three select diffraction dots are indexed to their relative position (Fig. 1). It is important to note that the image used in this analysis is provided, and not the image obtained in this experiment. The original image obtained in this experiment (Appendix A) shows a diffraction pattern composed of multiple discrete dots

at the same diffraction spot location. This is evidence of imperfect single-crystallinity. The center spot is also covered by the beam stop, and overall it was judged to be better to utilize the provided diffraction pattern image.

The diffraction pattern of the polycrystalline evaporated Al sample was measured by the methods described in the relevant methodology above. The ratio of R_2/R_1 was first found and squared to find $h_1^2+k_1^2+l_1^2$ and $h_2^2+k_2^2+l_2^2$; then the of R_{i+1}/R_i ratios were computed and squared for the rest of the rings to find the pertaining $h_{i+1}^2+k_{i+1}^2+l_{i+1}^2$. An index was then found by solving for h, k, and l, values that satisfy the $h_i^2+k_i^2+l_i^2$ computed (Table 3). The indexed ring diffraction pattern can be found below as Fig. 2.

The bright and dark field images of the polycrystalline evaporated Al sample are provided below as Fig. 3. The bright field image can be seen to have a mostly light background with discrete dark regions throughout. This due to the fact that bright field TEM imaging is done by using only the incident (and minimally diffracted) beam for imaging. The aperture is placed such that diffracted rays are covered and excluded as imaging sources. The result is presented as dark regions where the electrons have been diffracted, as there is no (or less) signal being included from those regions. Opposingly, dark field imaging is conducted by placing the aperture such that the incident (and minimally diffracted) beam is blocked, and only regions of diffracted rays are used to image the sample. This produced signal exclusively from those diffracted regions, those which were previously excluded in the case of bright field imaging. The result is an image composed of bright speckled regions of diffraction surrounded by dark non-diffracting areas.

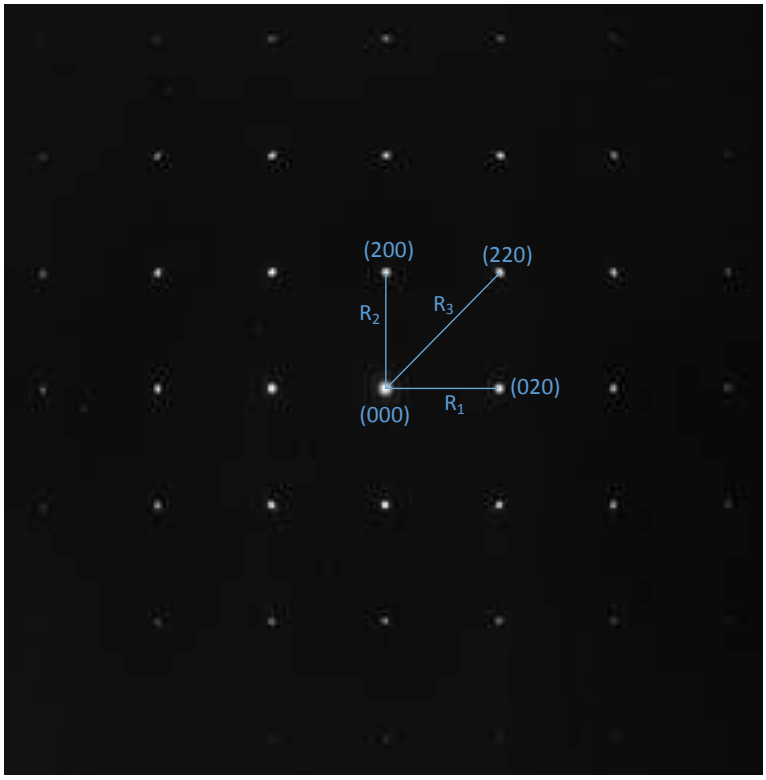

Fig. 1: Diffraction pattern of single crystal Au sample.

R1	R2	R3	R3/R1	R3/R2
58	57	81	1.396552	1.421053

Table 1: The relative distances of three points from center, and their ratios, are reported.

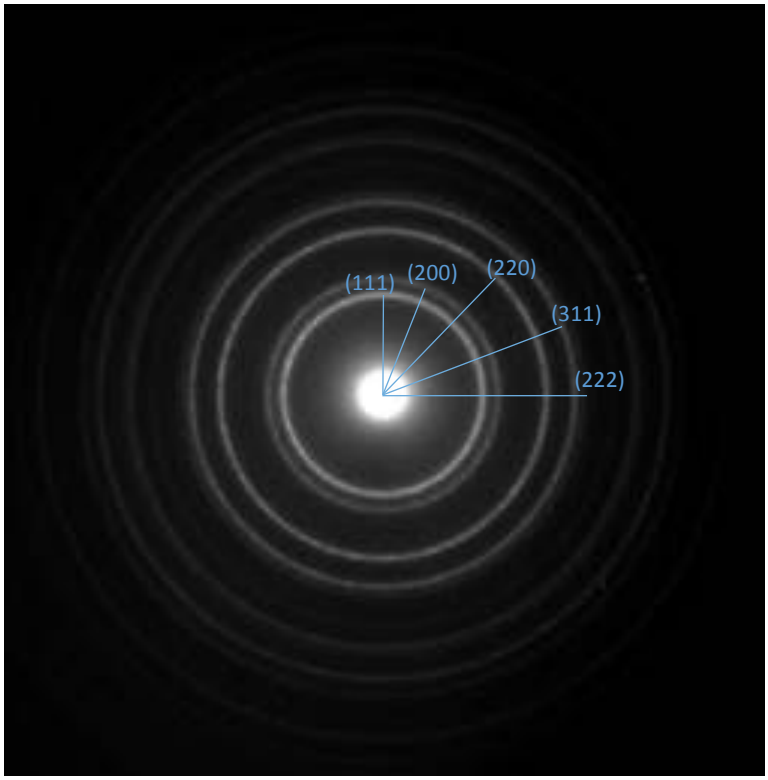

Fig. 2: Diffraction pattern of polycrystalline evaporated Al sample.

Sampling	R1	R2	R3	R4	R5
1	35.98	41.62	58.62	69.29	72.81
2	35.96	41.22	58.83	69.04	72.7
3	36.01	41.5	59.05	69.23	72.43
avg R	**35.98**	**41.45**	**58.83**	**69.19**	**72.65**

Table 2: The radius of each ring was sampled at 3 different locations and averaged.

R	R1	R2	R3	R4	R5
R_i (mm)	35.98	41.45	58.83	69.19	72.65
$(R_{i+1}/R_i)^2$	1.326711	2.014966	1.382923	1.10252	
~	1.33	2.00	1.375	1.09	
$h_i^2+k_i^2+l_i^2$	3	4	8	11	12
indices	**(111)**	**(200)**	**(220)**	**(311)**	**(222)**

Table 3: Diffraction pattern of polycrystalline evaporated Al sample.

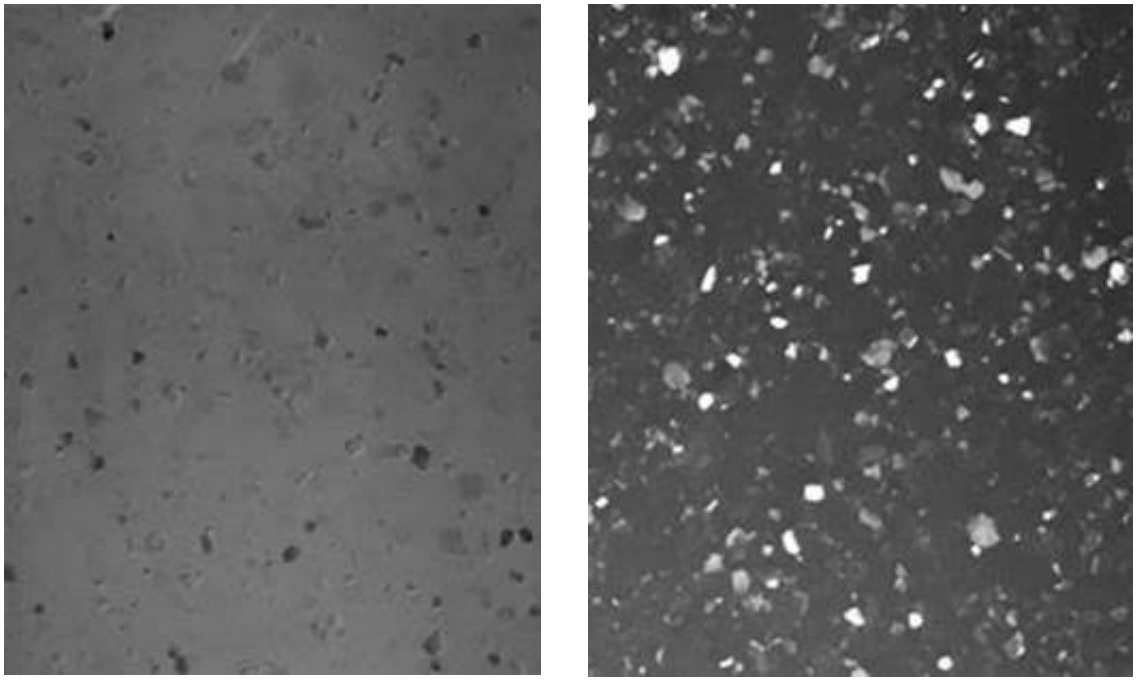

Fig. 3: ***Left:*** Bright field image of polycrystalline evaporated Al sample. ***Right:*** Dark field image of polycrystalline evaporated Al sample.

Summary

Single crystal Au and polycrystalline evaporated Al samples were imaged under a Transmission Electron Microscope (TEM), and their diffraction patterns were obtained, analyzed, and indexed. The indexed diffraction spots for the single crystal Au sample are found to exemplify FCC structure and to correlate to (020), (200), and (220) indices. The diffraction rings obtained in imaging of the polycrystalline evaporated Al sample are indexed to be (111), (200), (220), (311), and (222) – ordered from largest to smallest d-spacing. Bright and dark field images of the polycrystalline evaporated Al sample were obtained and analyzed with reference to their pertaining TEM operating conditions.

Acknowledgements

Thank you to Dr. Ni and Mr. Barkley for teaching me this material and providing lab space in which to conduct these experiments.

References

[1] Ni, Chaoying, "*TEM Laboratory Exercise No.1 – SAED, BF, DF*," MSEG624 Practical Electron Microscopy, University of Delaware, Spring 2019.

Appendix A

Fig. 5: Diffraction pattern of single crystal Au sample taken in lab.

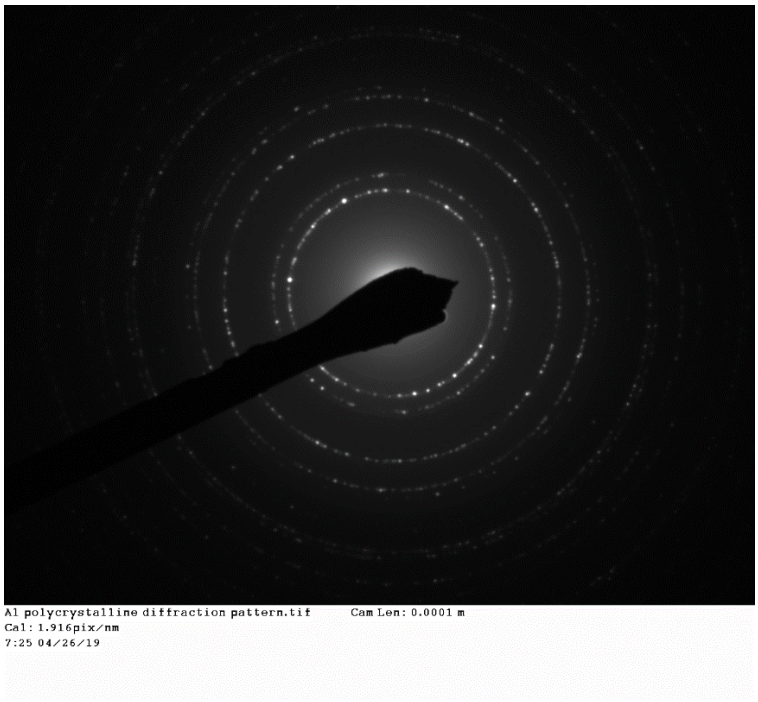

Fig. 6: Diffraction pattern of polycrystalline evaporated Al sample taken in lab.

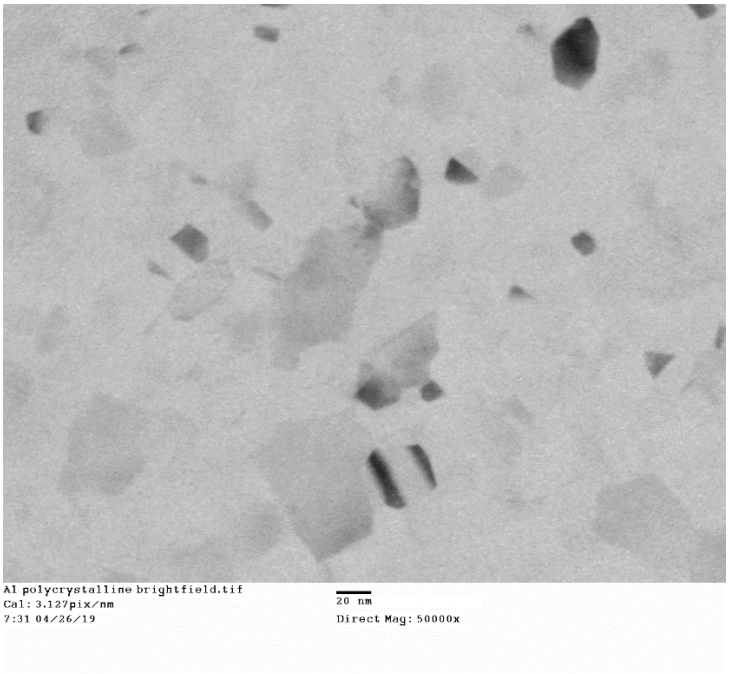

Fig. 7: Bright field image of polycrystalline evaporated Al sample taken in lab.

Fig. 8: Region of diffracted electrons used for dark field imaging in lab.

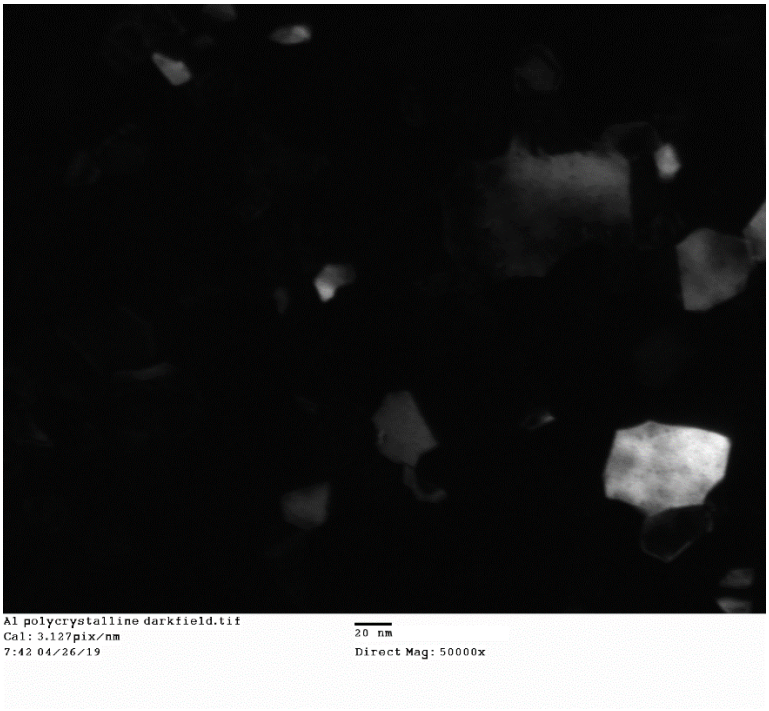

Fig. 9: Dark field image of polycrystalline evaporated Al sample taken in lab.

Supplemental

All files used and supplementary material can be found at:

https://github.com/zswain/MSEG624