A.13.4 Laboratory 2 – Diffraction Analysis of θ' Precipitates

This experiment introduces the important methods of electron diffraction and dark-field imaging to determine the identity and orientation relationship of precipitates in a matrix. For an introductory laboratory, θ' precipitates have proved convenient in size and contrast against the Al matrix. This exercise also provides experience with sample tilt, which may require a prior session of practice. Laboratory 2 couples well with the energy-dispersive x-ray analysis in Laboratory 3, but the two can be performed independently.

Background. The θ' phase is a metastable precipitate that often forms during aging of Al-Cu base alloys. It has a tetragonal crystal structure with space group symmetry $I4/mmm$ and $a = 0.404\,\text{nm}$ and $c = 0.58\,\text{nm}$. A perspective drawing of the unit cell of the θ' phase is shown in Fig. A.13. The unit cell contains four atoms of Al and two atoms of Cu. The θ' precipitates form as thin plates on the 100 planes in the Al matrix with the orientation relationship $(001)_{\theta'} \parallel (001)_{\text{Al}}$ and $[100]_{\theta'} \parallel [100]_{\text{Al}}$.

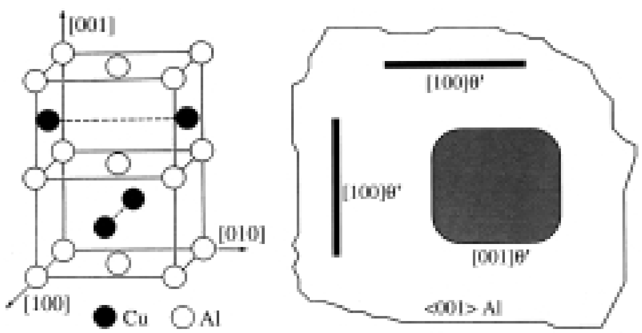

Fig. A.13. Left: Labeled crystal structure of θ' precipitate. Right: Orientations of three variants of θ' plates in the fcc Al matrix.

The θ' phase forms as thin plates on all three $\{001\}_{\text{Al}}$ matrix planes. When a thin foil is viewed along a $\langle 001\rangle_{\text{Al}}$ orientation, one variant of θ' phase is face-on, while the other two variants are edge-on and perpendicular to each another (see Fig. A.13). The Al matrix and each variant of θ' phase each produce a different diffraction pattern. When all three variants are present within the selected area aperture, all of these diffraction patterns are superimposed. If a small selected area aperture is used, however, it may be possible to obtain diffraction patterns from only one or two variants of precipitate. Figure A.14 shows diffraction patterns for the Al matrix in a $\langle 001\rangle$ orientation, and two variants of the θ' phase, one face-on along $[001]_{\theta'}$ and the other edge-on along $[100]_{\theta'}$. (The diffraction pattern for the third variant of θ' can be obtained by rotating the $[100]_{\theta'}$ pattern on the lower right by 90°.) All three of these patterns can then be superimposed to obtain the composite diffraction pattern in Fig. A.15. An experimental $\langle 001\rangle_{\mathrm{Al}}$ SAD pattern containing all three precipitate variants (and also double-diffraction spots) is also shown in Fig. A.15.

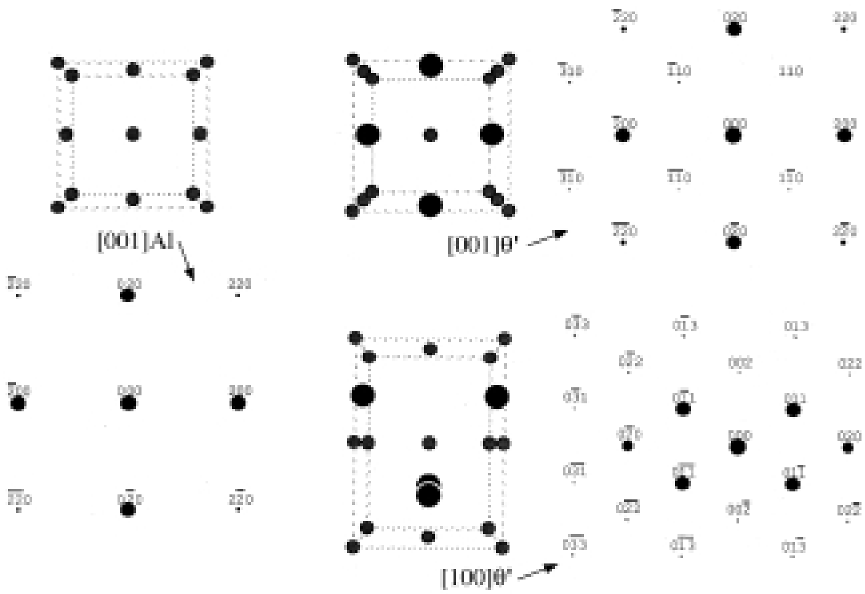

Fig. A.14. Indexed $\langle 001\rangle$ diffraction patterns from fcc Al matrix (left), and two variants of θ' precipitates within the Al matrix (right).

The different variants of precipitate can be identified by bringing each of the precipitate diffractions labeled 1, 2 and 3 in the composite pattern onto the optic axis within a small objective aperture, and making a dark-field (DF) image.

Specimen. Electropolished thin foils of Al–4.0 wt% Cu alloy. A sheet of polycrystalline alloy about 150 μm thick was solution treated for 1 h at 550°C, quenched into water and aged for 12 h at 300°C to produce well-developed θ' precipitate plates. Disks 3 mm in diameter were punched from the sheet and electropolished in a twin-jet Fischione apparatus using a 25 %HNO_3–methanol solution at about –40°C and 15 V.

(Alternative samples: carbon extraction replicas from a medium carbon steel, or pieces of aluminum beverage cans.)

Procedures

(**a**) Before going to the microscope, photocopy and enlarge the low index fcc diffraction patterns in the Appendix of this book. On a second set of diffraction patterns you should prepare a set of Kikuchi line patterns. To do

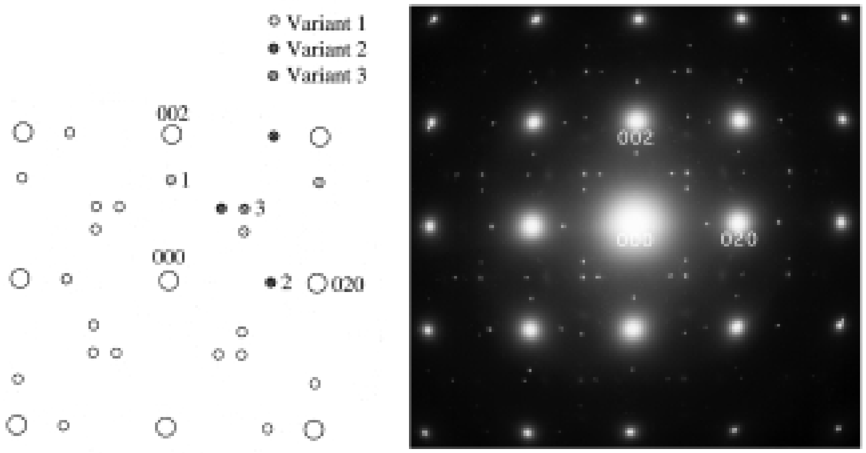

Fig. A.15. Composite diffraction pattern from all three variants of θ' precipitate in Al matrix in [100] zone axis. Left: schematic, Right: experimental SAD.

so, draw straight lines through the low index spots. The line through the spot $\boldsymbol{g}$ should be oriented perpendicularly to the direction $\widehat{\boldsymbol{g}}$ (the direction of the spot from the origin). You may want to plot other low-index diffraction patterns for the θ' phase using a computer program, if available. Please read some of the four references below. They contain information about the crystal structure, morphology, interfacial structure, and growth kinetics of the θ' phase.

(**b**) Obtain SAD patterns of the matrix and precipitates by tilting the specimen to low-index orientations such as $\langle 001\rangle_{\mathrm{Al}}$, $\langle 011\rangle_{\mathrm{Al}}$ or $\langle 112\rangle_{\mathrm{Al}}$. Use Kikuchi line patterns and indexed diffraction patterns to help you. The $\langle 001\rangle_{\mathrm{Al}}$ zone axis is the easiest to interpret, so you should try to obtain this orientation. Orient the specimen so that the pattern is exactly on the zone axis. Spread the illumination and take long exposures when photographing diffraction patterns so the faint precipitate spots will be sharp and visible. You might try several different exposures until you get a feel for the best exposure (typically about 1/4 of the automatic exposure reading). Don't forget to focus the diffraction pattern!

(**c**) To identify the precipitates in the intermediate aperture that contributed to the SAD pattern, photograph the corresponding BF images using the double-exposure technique. You may want to experiment with different size apertures, using a large aperture to obtain a pattern from all three θ' variants, using a smaller aperture to obtain diffraction patterns from only one or two variants.

(**d**) Photograph DF images of each of the θ' variants on the three $\{100\}_{\mathrm{Al}}$ planes. Do this by tilting the incident beam into the position of the precipitate diffraction spot, so the $-\boldsymbol{g}$ diffraction appears on the optic axis.