Question 1: Ni_3Al super alloy phase analysis

Single crystal super alloy turbine blades are based on the composition Ni_3Al. The Ni_3Al forms a γ FCC matrix which has a random distribution of the Ni and Al atoms on the lattice points (each lattice point 75% chance to be Ni, 25% chance to be Al), and coherent γ' precipitates in which the Ni and Al atoms order to form a simple cubic $L1_2$ structure.

a) For scattering from planes (*h k l*), the structure factor of a unit cell is given by the expression:

$$F_{hkl} = \sum_i f_i e^{2\pi i(hx_i + ky_i + lz_i)}$$

where f_i is the atomic scattering factor for each atom at position in the unit cell x_i, y_i, z_i.

For the γ' phase (atom positions below), calculate the structure factor rules for planes (*h k l*) in terms of atomic scattering factors f_{Ni} and f_{Al}.

#	Atom	Wyckoff	x	y	z
0	Al	a	0.000	0.000	0.000
1	Ni	c	0.000	0.500	0.500
2	Ni	c	0.500	0.500	0.000
3	Ni	c	0.500	0.000	0.500

Answer:

In the γ' phase Ni atoms are in the face-centered sites. Therefore we obtain the following expression for the structure factor:

$$F_{hkl} = f_{Al} + f_{Ni}\left(e^{\pi i(h+k)} + e^{\pi i(h+l)} + e^{\pi i(l+k)}\right)$$

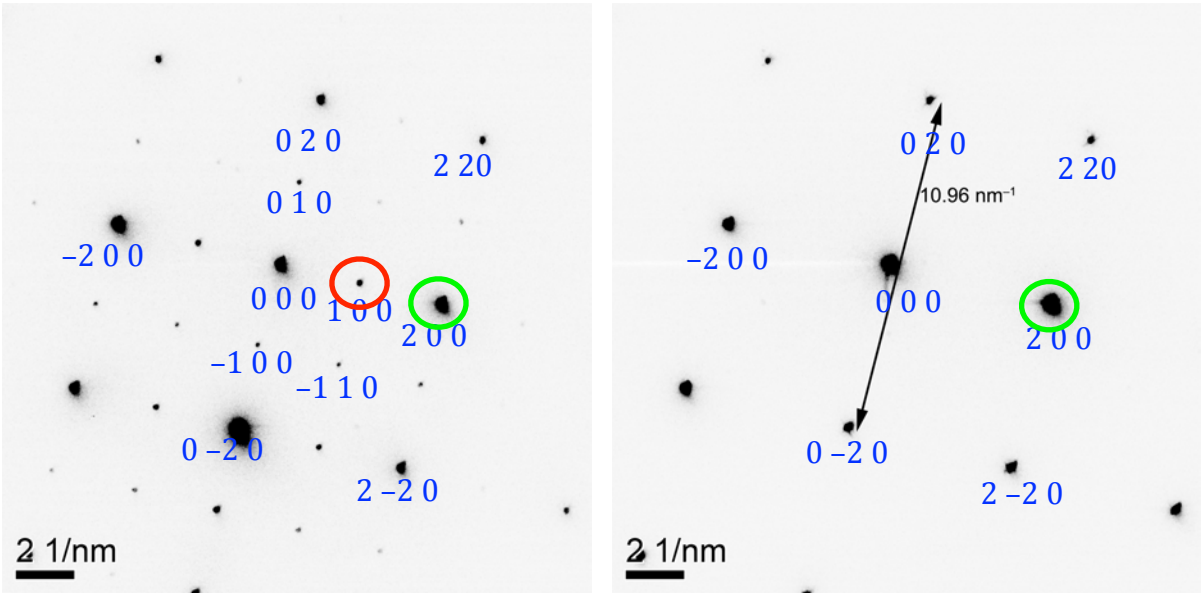
Now we find:

- *if h, k, l are even or if all are odd:* $F_{hkl} = f_{Al} + 3f_{Ni}$
- *if h, k, l are mixed even and odd:* $F_{hkl} = f_{Al} - f_{Ni}$

Reflections for h, k, l are mixed even and odd are not absent but given that: $I_{hkl} \propto |F_{hkl}|^2$

they will be much less intense than those for h, k, l all even or odd.

b) The two nanobeam electron diffraction patterns below were taken from a superalloy sample prepared for TEM.



Which pattern comes from which phase?

The right-hand pattern shows few reflections than the left hand pattern, consistent with there being forbidden reflections. Therefore the right-hand pattern is from the FCC γ phase and the left-hand pattern is from the simple cubic ordered γ' phase which does not have systematic absences.

What is the zone axis for the patterns?

The patterns both display [4]-fold rotational symmetry. For these cubic structures this is consistent with a <0 0 1> zone axis.

Index the reflections on the patterns.

Starting with the FCC phase on the right, we can take a zone axis of [0 0 1]. Any reflections (h k l) must be consistent with hU + kV + lW =0. The smallest reciprocal plane spacing consistent with this is from a (2 0 0) type of reflection. Once this is chosen the other reflections are straightforward to index by angles and vectorial addition. The ordered γ' phase also shows reflections with (h k l) mixed even and odd (e.g. (1 0 0)).

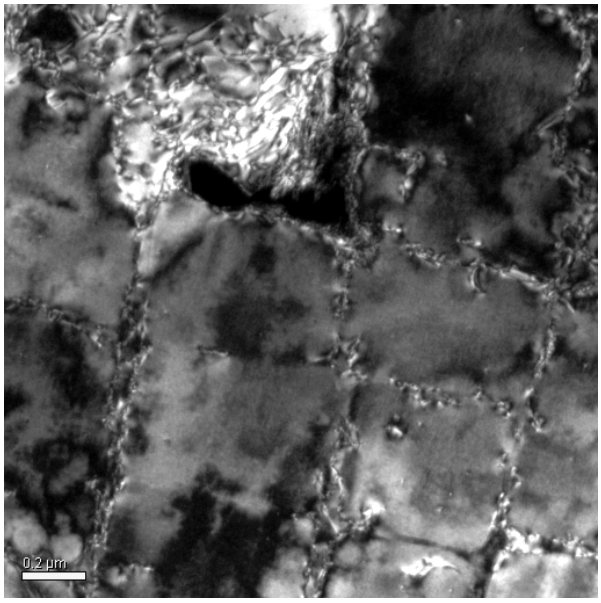
From the reciprocal distance indicated calculate the lattice parameter *a*.

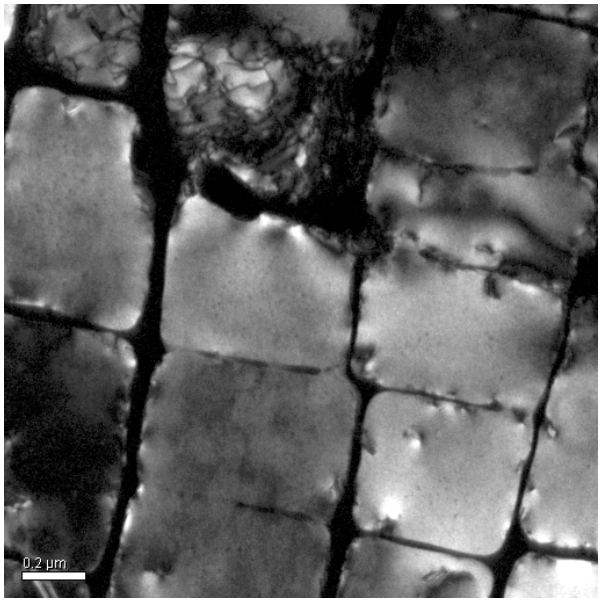
From the measurement we find: $d_{0\,2\,0}^{-1} = 10.96/2 = 5.48\ nm^{-1}$.

Plane (0 2 0) cuts the y axis at half the lattice parameter a, therefore: $d_{0\,2\,0} = a/2$

=> a = 2/5.48 = 0.365 nm

c) The DF images below were taken using different reflections from the Ni_3Al sample.





Which DF image shows the distribution of γ' precipitates?

The image on the right shows dark areas without intensity; this is a phase distribution and therefore shows the distribution of γ' precipitates. The image on the left does not show this phase distribution but instead a lot of contrast from defects.

On the diffraction patterns in **b)** indicate reflections that could have been used to take each of the DF images.

By taking a DF image with the objective aperture selecting a reflection which is only present for the γ' phase, only this phase will have intensity in the image. This could for instance be the (1 0 0) indicated by the red circle in b). The left hand image show both phases and so was made selecting a reflection that both phases give such as the (2 0 0) reflection marked with a green circle.

From looking at the DF images, why was nano-beam electron diffraction used to take the diffraction patterns and not selected area diffraction?

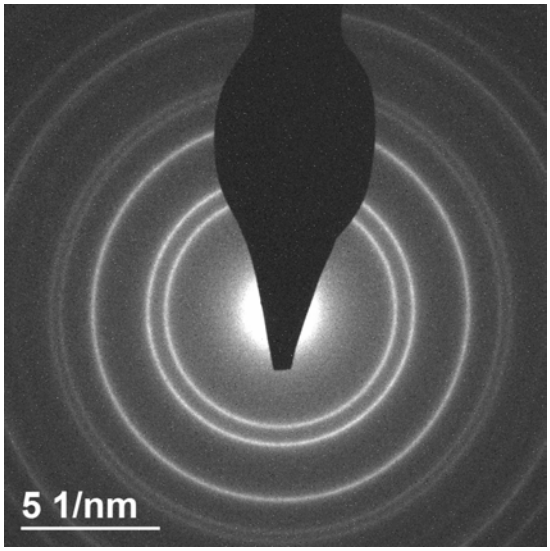
The regions of matrix phase are at most only ~50 nm wide. However at the smallest a selected area aperture selects a ~200 nm diameter region of sample. Therefore using selected area diffraction we cannot make a diffraction pattern only from the matrix phase. By using a nano-beam set-up it is possible to take a parallel beam (spot) diffraction pattern from a much smaller region of sample, so a diffraction pattern could be obtained from the matrix phase alone.

What other features can you see in the DF images (particularly the one on the left)?

The left image shows a lot of contrast from defects such as dislocations, particularly in the matrix phase.

Question 2: SADP analysis nanocrystalline pattern

Below is a selected area diffraction pattern which was taken from a sample of a large number of nanocrystals.



This pattern comes from a material that is either FCC or BCC. How can we use the pattern to identify which?

FCC and BCC have different systematic absences, and so different diffracting planes.
Ratios of the plane spaces therefore have fixed combinations that can be compared to the experimental ratios on the pattern.

The diameters of the first 5 rings are measured as:
8.27 nm^{-1}
9.62 nm^{-1}
13.64 nm^{-1}
15.95 nm^{-1}
16.6 nm^{-1}

From these identify if it is FCC or BCC and calculate the unit cell parameter *a*. Being cubic you can use the formula:

$$d_{h\,k\,l} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

The planes with largest spacings in FCC and BCC (and hence smallest reciprocal spacings giving rings with smallest diameters) are:

FCC: (1 1 1), (2 0 0), (2 2 0), (3 1 1), (2 2 2)
BCC: (1 1 0), (2 0 0), (2 1 1), (2 2 0), (3 1 0)

Diffraction ring diameter $\propto d^{-1}$

Ratios of 3rd/1st ring and 3rd/2nd ring should be:

	FCC	BCC	experimental
Ratio 3rd/1st	$\sqrt{\frac{8}{3}} = 1.63$	$\sqrt{3} = 1.73$	1.65
Ratio 3rd/2nd	$\sqrt{2} = 1.41$	$\sqrt{\frac{3}{2}} = 1.22$	1.42

Therefore experimental values match FCC ratios to within the 1–2% measurement accuracy for the pattern.

Taking the (2 0 0) ring for convenience, diameter measures as two time reciprocal lattice spacing for plane so: $d_{200}^{-1} = 9.62/2 = 4.81$ nm–1

$a = 2d_{200} = 0.416$ nm

This compares very favourably with actual lattice parameter $a = 0.4177$ nm