THE ROOM PRESSURE SPINELLOID PHASES OF THE $NiGa_2O_4$ - Ni_2SiO_4 SYSTEM

THE ROOM PRESSURE SPINELLOID PHASES OF THE $NiGa_2O_4$ - Ni_2SiO_4 SYSTEM

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ABSTRACT

The ternary oxide system NiO - Ga_2O_3 - SiO_2 has been studied in the temperature range 1400-1550°C at room pressure. Three phases, corresponding to the spinelloid phases I, II, and V, have been identified on the Ni_2GaO_4 (spinel) - Ni_2SiO_4 (olivine) join. These room-pressure phases are isostructural with the high-pressure spinelloid phases of the nickel aluminosilicate system. Single crystals of all three phases have been grown from a silica-rich melt and their crystal structures have been determined by X-ray diffraction. The structure refinements have revealed a strong ordering of the Ga and Si atoms on the tetrahedral sites of all three phases, as well as a clear correlation between spinelloid structure-type and composition. This correlation accounts for the increase in Ni_2SiO_4 content across the series spinel - phase V - phase I - phase II.

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It is said that more than one source is required to make a thesis. It also takes more than one person to create one. I would like first and foremost to thank my supervisor Dr. Jacques Barbier for his assistance with this work.

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CHAPTER 1

INTRODUCTION

The four oxides SiO_2, MgO, Fe_2O_3, and Al_2O_3 are thought to be the most abundant oxides in the earth's mantle and olivine, $(Mg, Fe)_2SiO_4$, is believed to be the most common mineral in the upper mantle. It is known that at high pressures and temperatures olivine undergoes a phase transformation to the denser spinel structure . The phase relationships of olivine and spinel, and the transformation between the two phases, have been extensively studied (Akimoto et al. 1965, Akimoto and Ida 1966, Akimoto and Fujisawa 1966, 1968, Akimoto and Syono 1970, Dachille and Roy 1960, Kawai et al. 1966, Ringwood 1956, 1962, 1963, Ringwood and Major, 1966) because of their importance to understanding the geophysical properties of the earth's interior.

In 1970 Ringwood and Major (1970) reported the discovery of a new phase, observed during their study of the Mg_2SiO_4 - Fe_2SiO_4 system. This orthorhombic phase, the β-phase, occurred as an intermediate in the olivine $\leftrightarrow$ spinel transition of synthetic olivines between $(Mg_{0\cdot8}\ Fe_{0\cdot2})_2SiO_4$ and pure Mg_2SiO_4. A naturally occurring sample of this phase was later found in a meteorite and was given the name Ringwoodite (Binns et al. 1969). Work by Moore and Smith (1970) has shown that the β-phase crystallized with a modified spinel structure. The same phase was later observed in the Co_2SiO_4 and Mn_2GeO_4 systems (Akimoto and Sato 1968). In 1974 Ma undertook a new type of study (Ma 1974) involving an aluminate (spinel) - orthosilicate (olivine) type join, which used changes in composition to model pressure changes. Ma observed three new orthorhombic phases in the $NiAl_2O_4$ - Ni_2SiO_4system, which he named phase I, phase II, and phase III. Ma's work was

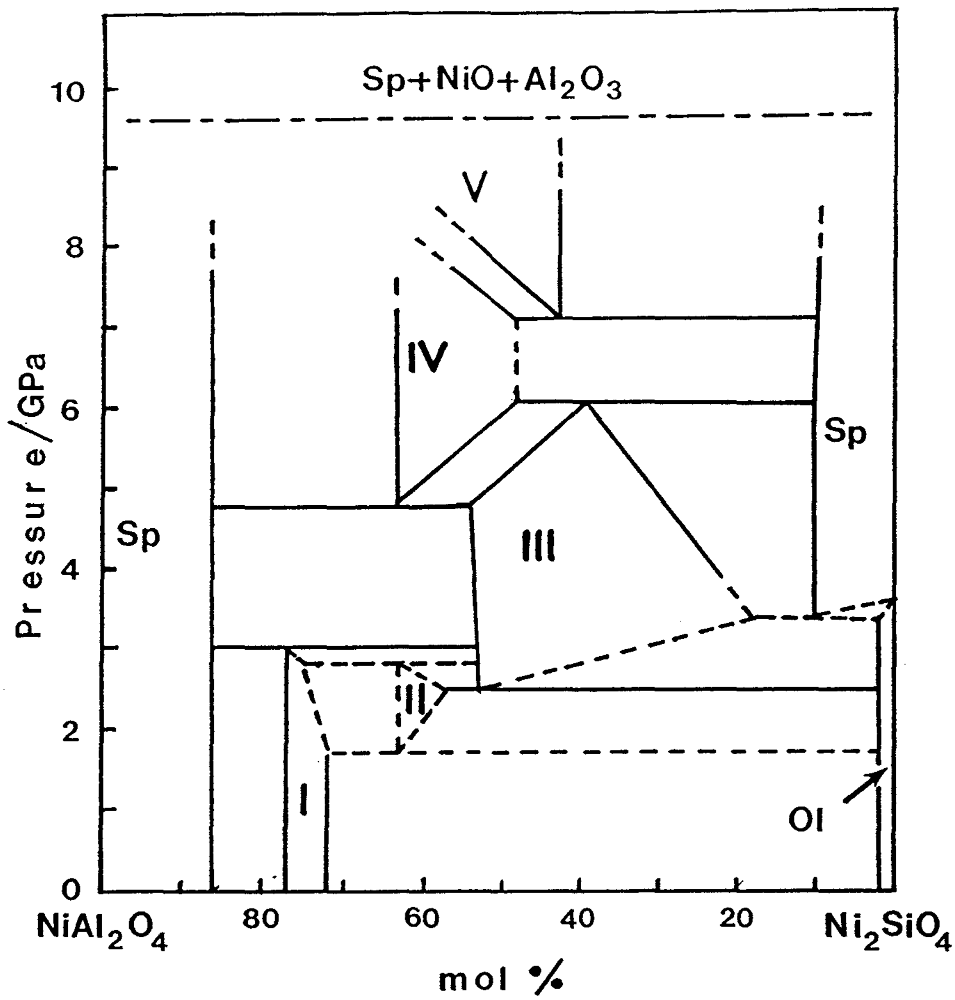

Fig. 1.1 A schematic pressure – composition diagram of the high pressure spinelloid phases in the system $NiAl_2O_4$ – Ni_2SiO_4 (after Akaogi et al. (1982). Notations are: Sp, spinel; Ol, olivine; I, phase I; II, phase II; III, phase III; IV, phase IV; V, phase V.

extended by Akaogi and his co-workers (1982) who studied the system at higher pressures and discovered two more phases, phase IV and phase V (see Fig. 1.1). They collectively termed these five new phases "spinelloids".

The spinelloid phases are considered to be structural derivatives of spinel with compositions lying along the $NiAl_2O_4$ - Ni_2SiO_4 join, ideally given as 3 $NiAl_2O_4$: Ni_2SiO_4 for phase I, 3 $NiAl_2O_4$: 2 Ni_2SiO_4 for phase II, and $NiAl_2O_4$: Ni_2SiO_4 for phases III, IV, and V. Phase I is stable between 0.0 to 2.5 GPa (Ma et al. 1975). Phase II was initially reported stable from 2.0 to 2.5 GPa but later work by Akaogi and Navrotsky (1984) and Barbier (1985) has shown that phase II is apparently also stable at room pressure at lower temperatures. Phase II is isostructural with manganostibite (Ma and Tillmans 1975), Mn_7SbAsO_{12}, a structure related to spinel and first reported by Moore (1970). Phase III is stable between 2.5 and 6.0 GPa and has the same structure as the β-phase of the $(Mg, Fe)_2SiO_4$ system (Ma and Sahl 1975). Phases IV and V are stable from 5.0 to 7.2 GPa and 7.0 to 9.5 GPa respectively and both have been described as hybrid structures of spinel and the β-phase (Horioka et al. 1981a, Horioka et al. 1981b). Above 9.5 GPa the spinelloids decompose into Ni_2SiO_4 (spinel), NiO, and Al_2O_3.

All five phases are orthorhombic with a $\approx$ 5.66 Å, b $\approx$ r x 2.88 Å, which is the typical oxygen - oxygen distance for close packing (r = 4 (phase I and III), r = 6 (phase II), r = 10 (phase IV), and r = 3 (phase V)), and c $\approx$ 8.10 Å. Phases I and V crystallize in the space group *Pmma*, while phases II, III, and IV have *Imma* symmetry. Hyde et al. (1982) have found a simple, unifying description of the spinelloid phases and described their role in a possible mechanism for the olivine $\leftrightarrow$ spinel transformation. In this scheme, the spinel structure is described using a body centered tetragonal unit-cell, which is half the volume of the conventional cubic unit-cell, and is related to it by $a_t = (a_c + b_c)/2$, $b_t = (-a_c + b_c)/2$, $c_t = c_c$. Fig. 1.2 shows a projection on

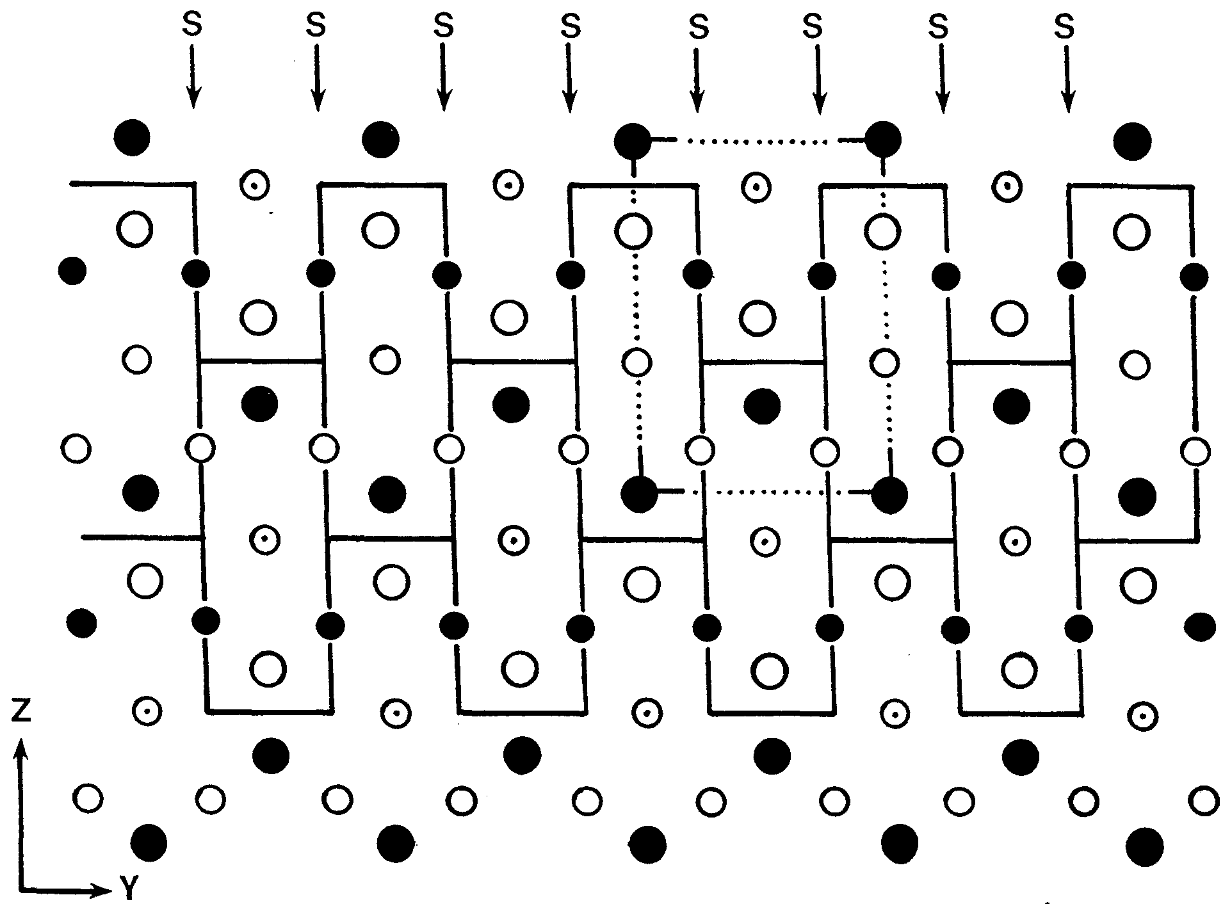

Fig. 1.2 The structure of the spinel phase projected on $(110)_c = (100)_t$ showing only the cations. Large circles are tetrahedrally coordinated cations, small circles are octahedrally coordinated cations, open at $x = \frac{1}{2}$, closed at $x = 0$, and dotted $x = \pm\frac{1}{4}$. The unit cell is shown by the dotted outline. These conventions are also used in Figs 1.3 and 1.4. (Hyde et al. 1982)

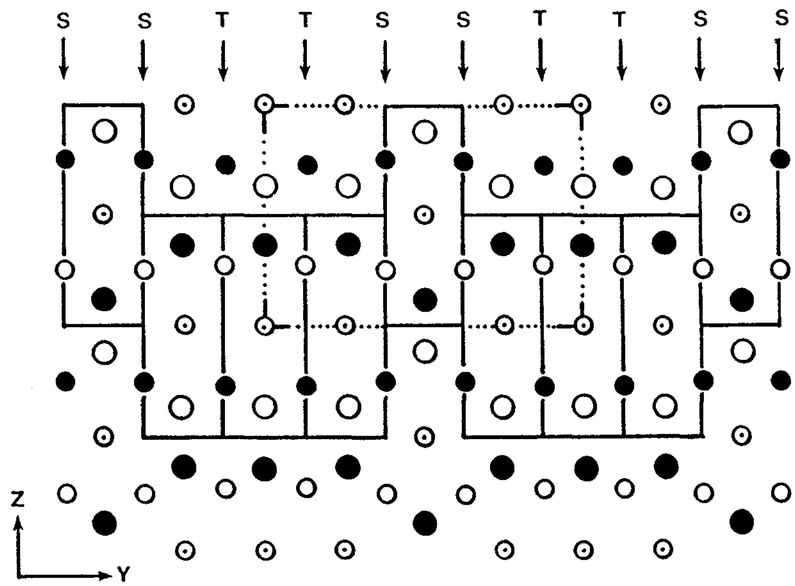

Fig. 1.3a The structure of phase I projected on (100) showing the cations only. (Hyde et al. 1982)

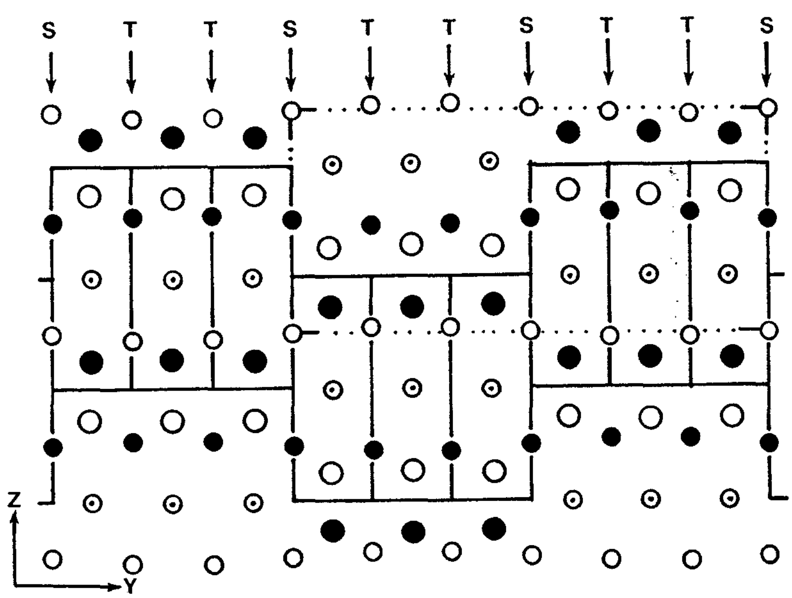

Fig. 1.3b The structure of phase II projected on (100) showing the cations only. (Hyde et al. 1982)

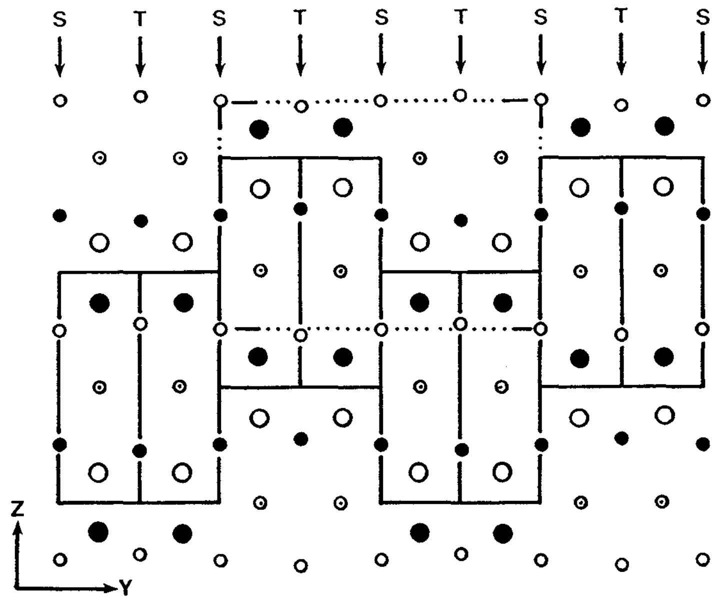

Fig. 1.3c The structure of phase III projected on (100) showing the cations only. (Hyde et al. 1982)

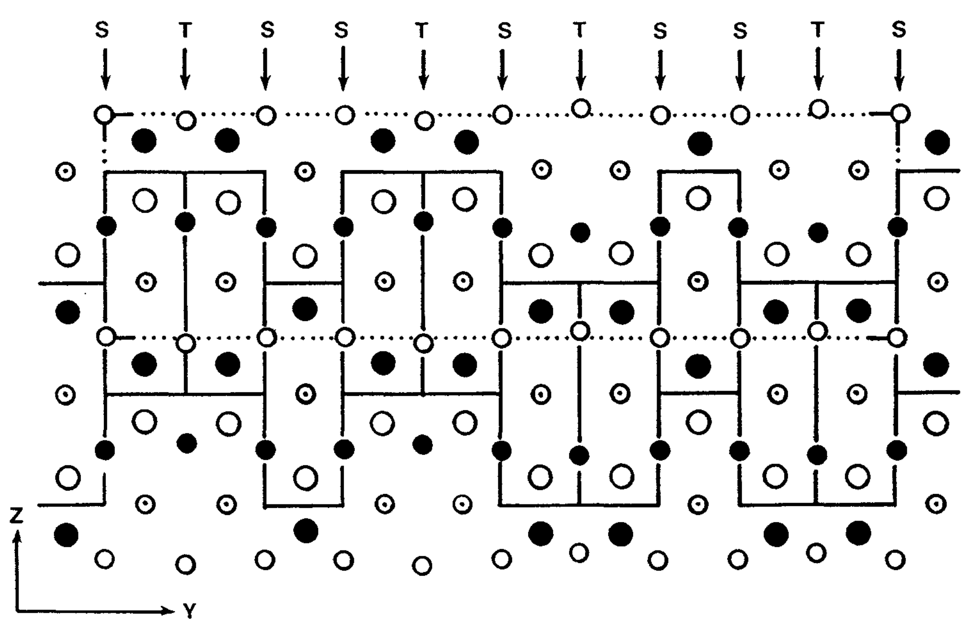

Fig. 1.3d The structure of phase IV projected on (100) showing the cations only. (Hyde et al. 1982)

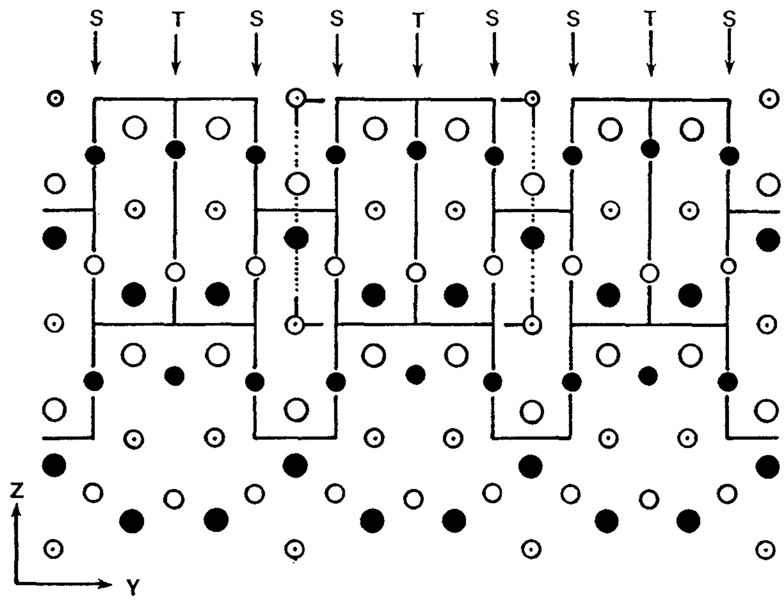

Fig. 1.3e The structure of phase V projected on (100) showing the cations only. (Hyde et al. 1982)

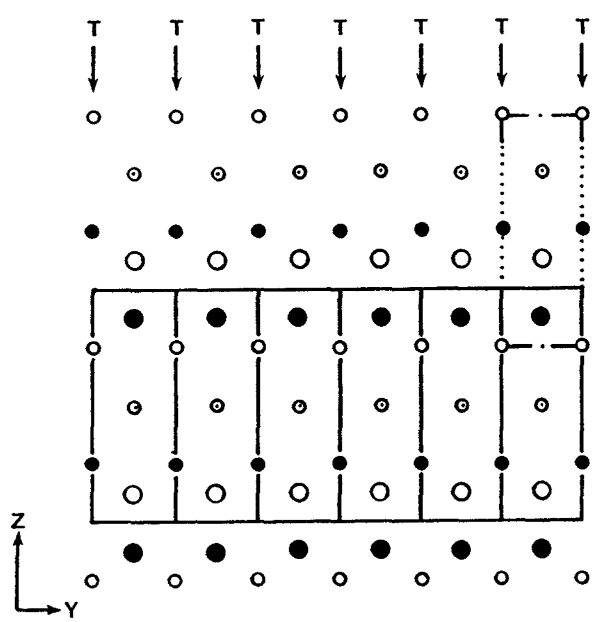

Fig. 1.4 The structure of the ω–phase projected on (100) showing the cations only. (Hyde et al. 1982)

$(100)_t$ of the cation positions in spinel, which can be described as $(010)_t$ slabs of spinel $b_t/2$ thick, separated by anti-phase boundaries (S). The five spinelloid phases are simply derived by replacing some of the S boundaries with reflection/twin planes (T) as shown in Figs. 1.3a-e. Thus the spinelloid phases form a series of structures intermediate between spinel and an unknown ω-phase, obtained by replacement of all the S boundaries with T boundaries as shown in Fig 1.4. Hyde et al. have observed that an $(0\bar{1}2)$ layer of the ω-phase is identical to a single (001) layer, c/2 thick, of olivine. The difference between olivine and the ω-phase lay solely in the way in which the layers are joined, resulting in cubic eutaxy of the anions in the ω-phase, and hexagonal eutaxy in the olivine phase. This structural relationship between the two phase allows a simple mechanism for transformation of one phase to the other. One such mechanism, described by Hyde et al., involves a shear of the cation array followed by a shear of the anion array with some small shuffling of the tetrahedral cations in alternate layers to retain proper coordination. Hence a new diffusionless reaction pathway, α-olivine $\longleftrightarrow$ ω-phase $\longleftrightarrow$ spinelloids $\longleftrightarrow$ γ-spinel is proposed for the olivine $\longleftrightarrow$ spinel transformation.

The relative stabilities of spinelloid related stacking sequences have been analysed by Price (1983), based on the relative magnitudes of the interaction energies between first, second, and third neighbor structural layers, and it was shown that, of all the polytypic modifications considered, only spinel and the five known spinelloid phases possess a minimum value for the interaction energy between layers. In his energy calculations Price considered two factors, i.e. local electrostatic charge imbalances and ion-size mismatch energy, both resulting from changes in composition across the $NiAl_2O_4$ - Ni_2SiO_4 system. The former factor is mainly the effect of cation distribution on the charge balance around the oxygen atoms lying on the T - type interface between two structural units. These mirror-related sheets result in one interfacial oxygen atom being bonded to five octahedral sites and also generate a corner sharing tetrahedral group with another interfacial oxygen atom, bonded to one octahedral and two tetrahedral sites. Such an arrangement is highly unfavorable for Ni_2SiO_4 (with four - coordinated Si^{4+} ions) but becomes more favorable with the introduction of $NiAl_2O_4$ as NiSi units are replaced with Al_2, allowing the oxygen atoms to have a better charge balance. At 80% $NiAl_2O_4$: 20 % Ni_2SiO_4 a zero-average interaction energy is achieved. The effect of structural mismatch results in a strain energy by causing changes in the relative sizes of the cation coordination polyhedra, thus affecting the packing efficiency of the anion lattice. Price showed that for the aluminosilicate system this effect is minimal and hence he concluded that the stability of the spinelloid phase was primarily due to electrostatic contributions to the interaction energy.

More recently, spinelloid phases were discovered in the $MgGa_2O_4$ - Mg_2GeO_4 (Barbier and Hyde 1986, Barbier 1989) and $MgFe_2O_4$ - Mg_2GeO_4 (Barbier 1989) systems. Three spinelloid phases were identified in the $MgGa_2O_4$ - Mg_2GeO_4 system. $Mg_3Ga_2GeO_8$(III), isostructural with the spinelloid phase III in $NiAl_2O_4$ - Ni_2SiO_4, is stable at room pressure up to 1420°C, above which it decomposes reversibly into spinel and olivine. It also transforms into $Mg_3Ga_2GeO_8$(IV), isostructural with the spinelloid phase IV, at around 30 kbar at 1100°C. Further increasing the pressure to 60 kbar causes a new phase transition to a mixture of spinel and $Mg_3Ga_2GeO_8$(V), isostructural with the spinelloid phase V. In the $MgFe_2O_4$ - Mg_2GeO_4 system only one spinelloid phase, $Mg_3Fe_2GeO_8$(III), has so far been observed. Like $Mg_3Ga_2GeO_8$(III), it is stable at room pressure. Several other systems, including $MgAl_2O_4$ - Mg_2SiO_4, $MnGa_2O_4$ - Mn_2GeO_4, $CdGa_2O_4$ - $CdGe_2O_4$, $ZnGa_2O_4$ - Zn_2GeO_4, and $NiGa_2O_4$ - Ni_2GeO_4 were investigated with negative results (Barbier and Hyde 1986). Based on these observations, these authors suggested that the formation of spinelloid phases at room pressure in spinel - olivine systems, requires that the spinel end-member has an inverse structure such as $NiAl_2O_4$ (80% inverse), $MgGa_2O_4$ (85% inverse), and $MgFe_2O_4$ (100% inverse).

The purpose of this study was to extend the work done on room - pressure spinelloids by investigating other systems likely to contain such phases. Four systems, each of which contains an inverse spinel end-member were investigated as a part of this work: $NiGa_2O_4$ - Ni_2SiO_4, $CdGa_2O_4$ - Cd_2GeO_4, $CoGa_2O_4$ - Co_2SiO_4, $MgGa_2O_4$ - Mg_2SiO_4, and $NiFe_2O_4$ - Ni_2SiO_4. The $MgAl_2O_4$ - Mg_2SiO_4 and $MgAl_2O_4$ - Mg_2GeO_4 systems were also investigated, even though $MgAl_2O_4$ is a normal spinel, to determine if high temperatures were sufficient to prepare room-pressure spinelloids. The work reported in this thesis deals mainly with the $NiGa_2O_4$ - Ni_2SiO_4 system.

CHAPTER 2

DIFFRACTION THEORY

This chapter will summarize the basic theories of diffraction for X–rays and electrons by crystalline materials. The information for X–ray diffraction is taken from Ashcroft and Mermin (1976 Chapt. 6) and Stout and Jensen (1989 Chapt. 2), while the material on electron diffraction is drawn from Reimer (1984) and Rymer (1970 Chapts. 3 and 6).

2.1 X–ray Diffraction Theory

In 1912 von Laue proposed that X–rays could be diffracted by crystals, and conducted the experiment which confirmed his prediction. Laue regarded a crystal as a three dimensional array of identical objects in a Bravais lattice, each of which could scatter an incident X–ray beam. Although the beam is scattered in all directions, strong intensities of diffracted radiation will be observed in directions and wavelengths for which the rays scattered from each object are in phase, while no intensity is observed where such rays are out of phase.

To determine the conditions under which the rays are scattered coherently, consider two scattering objects separated by vector **d** (see Fig. 2.1). An incident beam of parallel X–rays, travelling in direction **n** with wavelength λ and wavevector $\mathbf{k} = 2\pi\mathbf{n}/\lambda$ illuminates the objects and is elastically scattered in direction $\mathbf{n}'$ with wavevector $\mathbf{k} = 2\pi\mathbf{n}'/\lambda$. Constructive interference occurs only if the path difference between the two rays is an integral number of wavelengths. The condition for constructive interference is thus

$$d \circ (\mathbf{n} - \mathbf{n}') = m\lambda \quad \text{(Fig. 2.1)} \qquad [2.1]$$

where m is integral. Multiplying [2.1] by $2\pi/\lambda$ gives the conditions on the incident and scattered wavevectors:

$$d \circ (\mathbf{k} - \mathbf{k}') = 2\pi m \qquad [2.2]$$

For an array of scatterers at the sites of a Bravais lattice, displaced from one another by the Bravais lattice vector **R**, the condition for coherent scattering is that

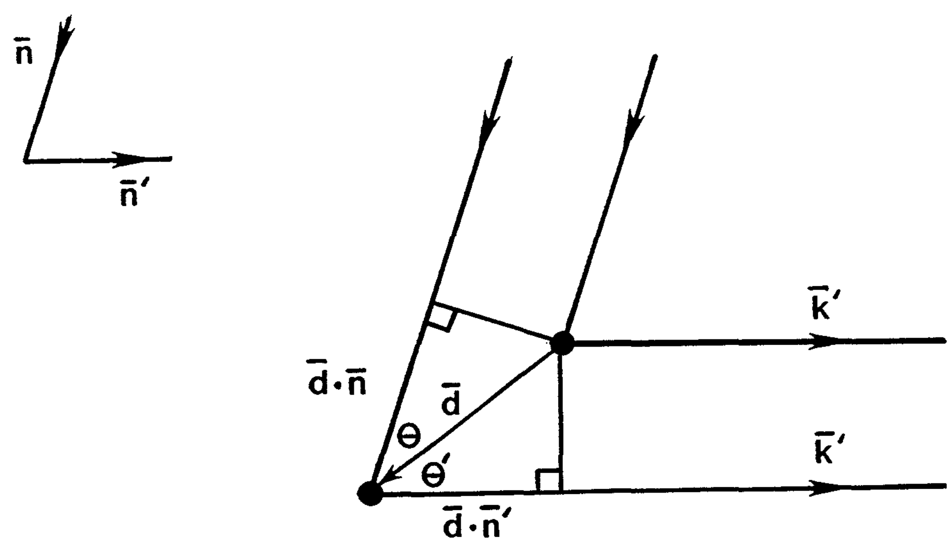

Fig. 2.1 The Laue condition for constructive interference. The path difference between the rays scattered by two atoms is $d \circ (\mathbf{n} - \mathbf{n}')$ where d is the vector separating the two atoms. (Ashcroft and Mermin 1976)

[2.2] holds simultaneously for all values of **d** that are Bravais lattice vectors.

$$\mathrm{R}\circ(\mathbf{k}-\mathbf{k}') = 2\pi\mathrm{m}, \qquad [2.3]$$

for integral m and all Bravais lattice vectors **R**. A reciprocal lattice is defined by the set of all wavevectors **K** that will yield plane waves with the periodicity of a given Bravais lattice, leading to the relation:

$$\exp\{i\,\mathbf{K}\circ\mathbf{R}\} = 1. \qquad [2.4a]$$

Using the relationship $\exp\{\mathrm{n}i2\pi\} = 1$ for integral n, [2.3] can be rewritten in the form:

$$\exp\,\{i(\mathbf{k}-\mathbf{k}')\circ\mathbf{R}\} = 1 \qquad [2.4b]$$

From a comparison of [2.4a] and [2.4b] it is clear that the Laue condition for constructive interference will occur only if the change in wavevector $\mathbf{k}-\mathbf{k}' = \mathbf{K}$, a vector of the reciprocal lattice.

During the same year von Laue described X–ray diffraction based on a three dimensional lattice, W.L. Bragg observed that diffraction by crystalline materials could be treated as a "refection" of the primary beam from planes in the crystal lattice, and could be described by a simple equation.

To derive the Bragg equation, consider a beam of parallel X–rays, R_1 and R_2, being elastically reflected by a pair of parallel planes P_1 and P_2 with interplanar spacing d (Fig. 2.2). The incident X–rays make an angle θ with these planes. The oscillating electric vector of the X–ray photons will cause electrons, assumed to be at O and C, to vibrate with the same frequency and resulting in them radiating in

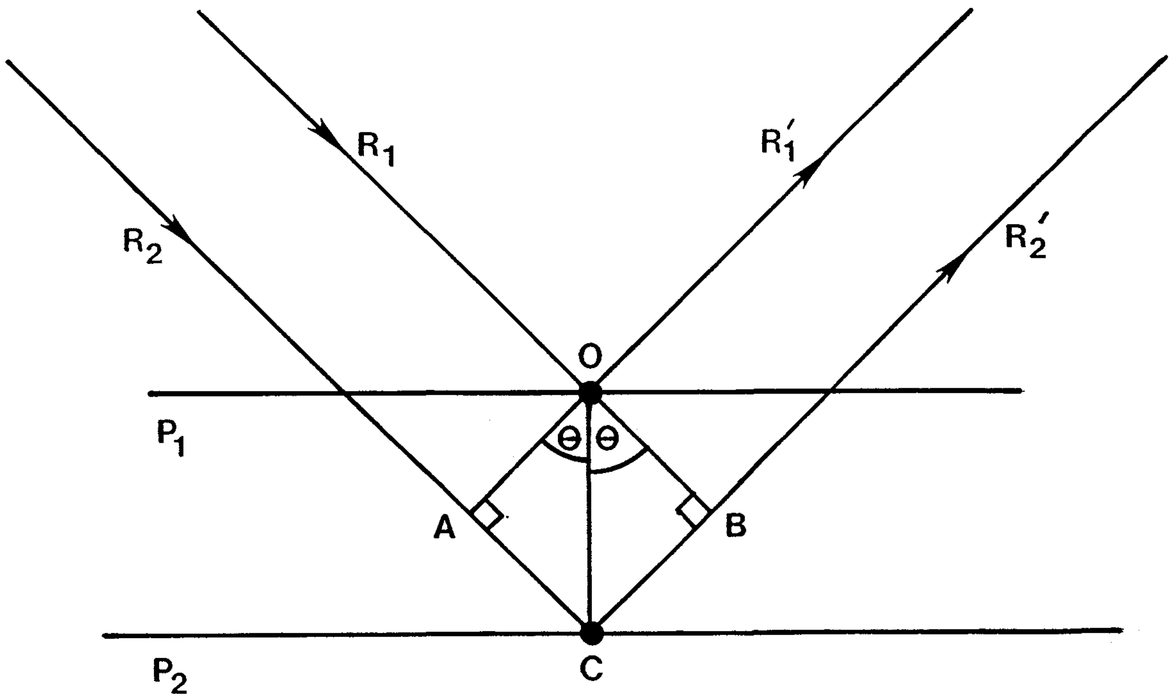

Fig. 2.2 Bragg reflection from a family of lattice planes. The path difference from A to B must equal an integral number of wavelengths. (Stout and Jensen 1989)

all directions. For the particular direction where parallel secondary rays R_1' and R_2' are emitted, at angle θ to the planes, a maximum intensity will be observed if the two rays are in phase. For this to occur, the difference in path length of the rays must be equal to an integral number of wavelengths, λ. Dropping perpendicular lines from O to A and B, it is clear that AC = BC. Thus, for the two rays to be in phase

$$2AC = n\lambda \qquad [2.5]$$

where n is integral. By definition $AC/d \equiv \sin\theta$ and so simple substitution yields

Bragg's Law:

$$2d \sin \theta = n\lambda \qquad [2.6]$$

While the Bragg equation represents the special case of electrons at points O and C, it is a simple exercise of geometry to show that this is also true for the general cases of electrons at any point on, or between, the two planes.

The reciprocal lattice is described by three vectors (**a***, **b***, **c***), which are related to the direct space vectors (**a**, **b**, **c**) of the Bravais lattice, by the relationships:

$$\mathbf{a}^* = \frac{2\pi\ (\mathbf{b} \times \mathbf{c})}{\mathbf{a}\circ(\mathbf{b} \times \mathbf{c})} \quad \mathbf{b}^* = \frac{2\pi\ (\mathbf{c} \times \mathbf{a})}{\mathbf{b}\circ(\mathbf{c} \times \mathbf{a})} \quad \mathbf{c}^* = \frac{2\pi\ (\mathbf{a} \times \mathbf{b})}{\mathbf{c}\circ(\mathbf{a} \times \mathbf{b})} \qquad [2.7]$$

Any point on the reciprocal lattice can be described by a reciprocal lattice vector K = h**a*** + k**b*** + l**c***. If a set of crystal lattice planes are separated by a distance d, then the shortest reciprocal lattice vector normal to the planes will have length 2π/d. The coordinates of this reciprocal lattice vector are known as the Miller indices of the lattice plane. These indices are the reciprocal of the fractional intersection of the unit cell edge by the lattice plane, and so a direct lattice plane described by Miller indices (h k l) has intercept **a**/h, **b**/k, and **c**/l.

Thus a Laue diffraction peak corresponding to a change in wavevector, K = h**a*** + k**b*** + l**c***, a reciprocal lattice vector, corresponds to a Bragg reflection from the family of planes (h k l) perpendicular to K. While the two treatments explain the same phenomenon and the Laue treatment is more rigorous, it is Bragg's Law that is most commonly used in crystallography.

While the unit–cell dimensions and symmetry will determine the location of the reflections in reciprocal space, it is the arrangement of atoms within the unit–cell that will determine the intensity of each reflection. Each atom in the unit–cell has a scattering factor which is a function of the atom type and $\sin\theta / \lambda$, where θ is the Bragg angle. The scattering power of a particular atom for a given scattering direction is known as its scattering factor f_0. For $\sin\theta = 0$, $f_0 = Z$, the atomic number of that atom. As the Bragg angle increases, f_0 will decrease, as X–rays being scattered from different points within the electron cloud will be slightly out of phase, causing some destructive interference, a consequence of the finite size of the electron cloud. Thermal motion of the atom will cause an increase in the size of the electron cloud, causing f_0 to diminish even more rapidly. As a consequence the actual scattering factor is not simply f_0, but rather

$$f = f_0 \exp\{-B \sin^2\theta / \lambda^2\} \qquad [2.8]$$

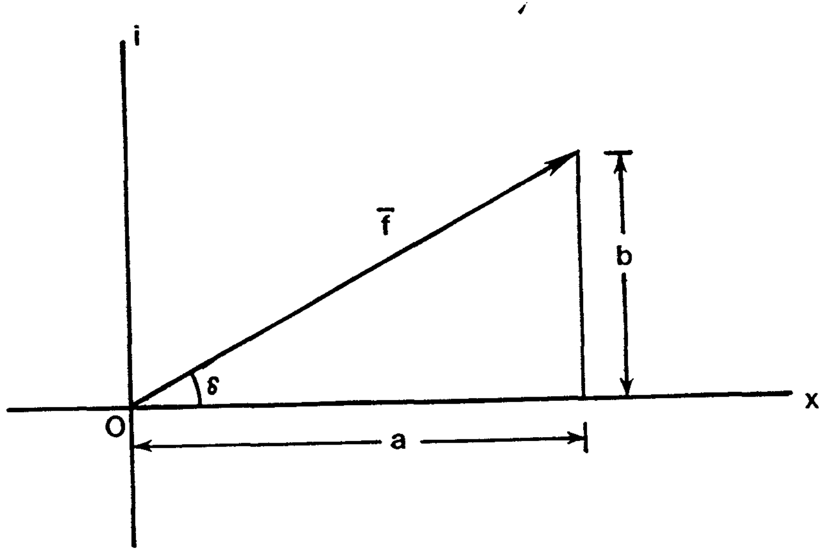

Fig 2.3 The vector **f** in the complex plane with magnitude |f| at phase angle δ. (Stout and Jensen 1989)

The structure factor F of reflection hkl is defined as the sum of the N waves scattered in the direction of the reflection by the N atoms of the unit–cell, each wave having an amplitude proportional to f_j, the scattering factor for atom j, and a phase δ_j measured with respect to the unit–cell origin. The scattering factor and phase angle can be represented as a vector of length f_j in a complex plane at angle δ_j to the positive real axis (Fig. 2.3). In this form the scattering factor can be expressed as:

$$a + ib = f_j\,(\cos\delta_j + i\sin\delta_j). \qquad [2.9]$$

As a result the structure factor can be found by:

$$F_{hkl} = \sum_j f_j\cos\delta_j + i\sum_j f_j\sin\delta_j. \qquad [2.10]$$

Recognizing that the phase difference in radians between the origin and the point (x_j, y_j, z_j) is

$$\delta_j = 2\pi\,(hx_j + ky_j + lz_j), \qquad [2.11]$$

then [2.10] becomes

$$\begin{aligned} F_{hkl} = &\sum_j f_j\cos 2\pi\,(hx_j + ky_j + lz_j) \\ &+ i\sum_j f_j\sin 2\pi\,(hx_j + ky_j + lz_j). \end{aligned} \qquad [2.12]$$

Using the well known relationship

$$\exp\{i\delta\} = \cos\delta + i\sin\delta \qquad [2.13]$$

the structure factor can be reduced to

$$F_{hkl} = \sum_j f_j \exp\{2\pi i\,(hx_j + ky_j + lz_j)\} \qquad [2.14]$$

which is the structure factor in exponential form for the hkl reflection.

While [2.14] shows how the structure factor can be obtained from a particular electron distribution, a crystal structure determination requires the reverse, i.e. to obtain the electron density from the structure factors. Using a three dimensional Fourier series, the electron density can be represented by

$$\rho(x, y, z) = \frac{1}{V} \sum_h \sum_k \sum_l F_{hkl} \exp\{-2\pi i(hx + ky + lz)\}$$

[2.15]

where V is the volume of the unit–cell.

Note that [2.14] represents the structure factors in terms of the electron density of the unit–cell while [2.15] represents the electron density of the unit–cell based on the structure factors, allowing the conclusion that the structure factors are the Fourier transform of the electron density and that the electron density is the Fourier transform of the structure factors.

Thus, by calculation of the structure factors for each observed reflection, it is possible, via a Fourier synthesis, to obtain the electron density distribution of the unit–cell of a crystal. These structure factors are obtained from a model of the atomic positions within the unit–cell, which is refined by minimizing the function:

$$D = \sum_{hkl} w_r\,(|F_o| - |k\,F_c|)^2 \qquad [2.16]$$

where w_r is the weight given to the observed reflection, F_o and F_c are the observed and calculated structure factors, and k is a scaling parameter. The overall agreement between the observed and calculated structure factors is measured by the residual index R, calculated by

$$R = (\Sigma \, ||F_o| - |F_c||) / \Sigma \, |F_o| \qquad [2.17]$$

or by using a weighted residual index wR. Setting $\Delta F = |F_o| - |F_c|$.

$$wR = \sqrt{\left[\frac{\Sigma \; w \cdot |\Delta F|^2}{\Sigma \; w . |F_o|^2}\right]} \qquad [2.18]$$

2.2 Electron Diffraction Theory

According to de Broglie, a particle with momentum p has a wavelength of

$$\lambda = h/p \qquad [2.19]$$

where h is Planck's constant. An electron, accelerated through a potential P will have a wavelength

$$\lambda = h \, / \sqrt{(2 \, m_0 eP)} \qquad [2.20]$$

where m_0 and e are the rest mass and charge of an electron. If the potential is high enough, then the velocity of the electron approaches c and it is necessary to make relativistic corrections to [2.20] resulting in

$$\lambda = \frac{h}{[2m_0ep \; (1 \; + \; ep/2m_0c^2)]^{\frac{1}{2}}}. \qquad [2.21]$$

At 150 eV an electron has a wavelength of about 1Å, small enough to be used to probe crystal structure by diffraction methods, although much higher energies, typically 100 to 120 keV, are used to improve resolution and penetration while decreasing chromatic aberration. As with X–rays, electrons must satisfy the Laue conditions for diffraction to occur and can also be described using Bragg's Law. At 120 keV $\lambda = 0.0317$Å for an electron and, for a typical interplanar spacing $d_{hkl} = 2$Å, the Bragg angle $\theta_B = 0.45°$. Such small angles allow a relaxation of Bragg's law, so that several lattice planes can diffract simultaneously. If the direction of the beam, the zone axis, is [m n o], then all reflections (h k l) which satisfy

$$mh + nk + ol = 0 \qquad [2.22]$$

are permitted. The small wavelength of the electrons also results in a much larger Ewald sphere, which approximates a plane near the origin of the reciprocal lattice allowing an undistorted view of the reciprocal lattice, as well as circles of reflections from the first and higher Laue zones which are cut by the sphere (see Fig. 2.4). Thus, when using electron diffraction, 2–dimensional sections of the reciprocal lattice of a microscopic single crystal can be observed permitting a visual determination of the reciprocal lattice symmetry, something not possible using powder X–ray techniques.

The intensity of the diffracted beams can be reasonably explained by the kinematic theory, which assumes that each atom receives an incident electron wave of the same intensity. The scattering factor for an electron by an atom is

$$f(\sin\theta\,/\,\lambda) = \frac{me^2}{8h^2\pi\varepsilon_0}\frac{\lambda^2\,(Z - F)}{\sin^2\theta} \qquad [2.23]$$

where Z is the atomic number, F the X–ray scattering factor of the atom, and ε_0 is the permittivity of vacuum. Each unit–cell in the crystal is located by the vector

$$\mathbf{r} = m\mathbf{a} + n\mathbf{b} + o\mathbf{c}, \qquad [2.24]$$

a, **b**, **c** being the vectors defining the three unit–cell axis, and m, n, o being integers. Each atomic position j within the unit–cell is also defined by the vector

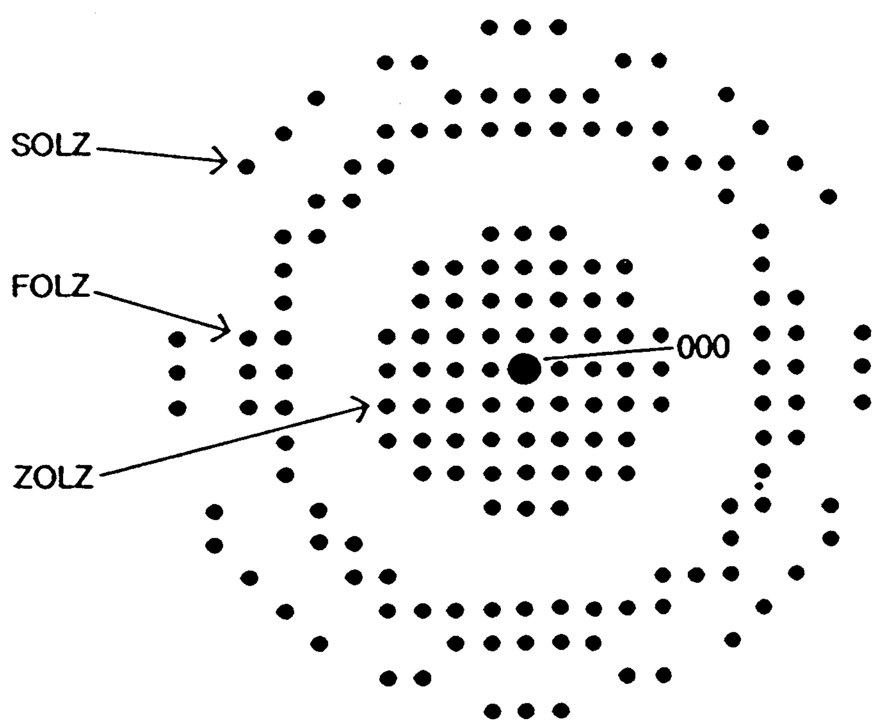

Fig. 2.4 A schematic projection of the diffraction pattern formed by the intersection of the Ewald sphere with the reciprocal lattice at the zero (ZOLZ), first (FOLZ), and second (SOLZ) order Laue zones. (Rymer (1970)

$$\mathbf{r}_j = x_j\mathbf{a} + y_j\mathbf{b} + z_j\mathbf{c} \qquad [2.25]$$

where x_j, y_j, z_j are proper fractions. Thus any atom in the crystal is located by the vector

$$\mathbf{R} = (m + x_j)\mathbf{a} + (n + y_j)\mathbf{b} + (o + z_j)\mathbf{c}. \qquad [2.26]$$

Since the Laue conditions apply, [2.3] holds and the phase difference between the scattered waves is

$$\phi = \frac{-2\pi}{\lambda}\mathbf{R}\circ(\mathbf{k}' - \mathbf{k}). \qquad [2.27]$$

Thus the total wave scattered by all the atoms of the crystal is

$$\psi = \frac{-\exp\{i\mathbf{K}\circ\mathbf{R}\}}{r}\sum_{mno}\sum_{j} f_j \exp\{2\pi i\,(\mathbf{k}' - \mathbf{k})\circ(\mathbf{r} + \mathbf{r}_j)\}$$

[2.28]

which can be rewritten

$$p = \frac{-\exp\{i\mathbf{K}\circ\mathbf{R}\}}{r}\,E\,G$$

where

$$E = \sum_{j} f_j \exp\{2\pi i\,(\mathbf{k}' - \mathbf{k})\circ(\mathbf{r}_j)\}$$

and

$$G = \sum_{mno} \exp\{-2\pi i\,(\mathbf{k}' - \mathbf{k})\circ(\mathbf{r})\}$$

The former term is the structure factor and depends solely on the distribution of the unit–cell contents while the latter term is the crystal shape factor and depends upon the size and shape of the crystal. The intensity of the scattered wave is given by

$$\psi\psi^* = \frac{1}{r^2}\,|E|^2\,|G|^2. \qquad [2.29]$$

There are limitations to the kinematic theory, which arise from the initial premise that the primary beam undergoes a negligible intensity loss as it passes through the crystal, resulting in the theory being valid only for thin crystals in which the diffracted intensity is small.

In spite of the elegance of the theory it is not possible to obtain the structure factors from the intensities of the diffracted beams, the strong interactions between the atoms and the electrons result in multiple scattering of the beams, convoluting the intensity data of several reflections into each observed reflection.

CHAPTER 3

DESCRIPTION OF EXPERIMENTS

3.1 Sample Preparation

The following reagents were used for all syntheses:

NiO	(99.99%, Johnson Matthey Chemical Co.)
Ga_2O_3	(99.99%, Aldrich Chemical Co.)
SiO_2	(99.5%, Johnson Matthey Chemical Co.)
$SiO_2 \cdot n\ H_2O$	(Reagent Grade, Matheson, Coleman, and Bell)
MgO	(99.99%, Aldrich Chemical Co.)
Al_2O_3	(99.99%, Johnson Matthey Chemical Co.)
GeO_2	(99.999%, Johnson Matthey Chemical Co.)
CdO	(99.0%, Allied Chemical Co.)
CoO	(99.5%, Cerac Inc.)
Fe_2O_3	(99.8%, Baker Chemical Co.)

The water content of the silica gel was determined by thermogravimetric analysis. All reagents were dried for 24 hours at suitable temperatures, and stored in a desiccator.

3.1.1 $NiGa_2O_4 - Ni_2SiO_4$

3.1.1.1 Powder Samples

All syntheses were originally performed using NiO, Ga_2O_3, and SiO_2 with 1 to 2 wt% of LiF added as a flux because of the slow nature of SiO_2 reactions (Phillips et al. 1963). It was later determined that the LiF interfered with the formation of spinelloid phases in this system and so its use was discontinued, and silica gel was used in place of SiO_2 because it reacted more rapidly.

Samples of 0.4 to 1.0 gram were prepared by mixing the oxides and, where silica gel was used, preheated for 2 hours at 1100° C to dehydrate the gel. The samples were then pelleted and heated in air at 1200° C to 1550° C for periods of 2 to 18 days, with repeated regrinding until no further changes were observed in the powder X–ray diffraction pattern of the reaction products. Partial melting occurred in samples heated to 1550° C.

The products were characterized by powder X–ray diffraction and were further examined by electron diffraction and high resolution electron microscopy.

3.1.1.2 Single Crystal Samples

All syntheses were performed using NiO, Ga_2O_3, and $SiO_2 \cdot n\ H_2O$ powders. In all experiments excess silica was used as to provide a flux for crystal growth. Samples of 8 to 10 grams with an excess of 10 to 30 wt% SiO_2 were preheated at 900° C for at least 2 hours to dehydrate the silica. The samples were then heated at 200° C/hr to 1540 – 1600° C, soaked for 5 hours, and then slowly cooled at 1° to 2° C/hr to 1550 or 1525° C. The samples were then carefully crushed and the crystals extracted from the flux. It was found that the SiO_2–rich matrix could be softened by soaking the sample in HF, dissolving the SiO_2 without damaging the crystals, thus allowing easier extraction. The crystals were characterized using a precession camera and the best crystals were then studied using a single crystal diffractometer.

3.1.2 Other Samples

3.1.2.1 Powder Samples

A limited number of samples were prepared from five other spinel – olivine systems ($MgAl_2O_4 - Mg_2GeO_4$, $CdGa_2O_4 - CdGe_2O_4$, $CoGa_2O_4 - Co_2SiO_4$, $MgGa_2O_4 - Mg_2SiO_4$, and $NiFe_2O_4 - Ni_2SiO_4$). The 0.4 to 0.5 gram samples were prepared from the oxide powders. The silicate samples were heated in air at temperatures between 1475 – 1600 °C, with the exception of the cobalt samples which could only be heated to 1300° C before melting. Germanate samples were heated in air from 700 – 1100° C, while above 1100° C they were sealed in platinum tubes. The reaction products were characterized by powder X–ray diffraction.

3.1.2.2 $MgFe_2O_4 - Mg_2GeO_4$ Single Crystals

Efforts were made to prepare single crystals of $Mg_3Fe_2GeO_8$(III) which had been previously reported by Barbier (1989). A sample of the composition $MgFe_2O_4 : Mg_2GeO_4$ was prepared from the oxides with a further 20 wt% excess of GeO_2 added. This sample did not melt below 1200° C. New samples were prepared using MgF_2 as a flux instead of excess GeO_2. Pre–reacted samples of $Mg_3Fe_2GeO_8$(III) with 10 to 50 wt% added MgF_2 were heated for 1 to 4 days at 1300° to 1400° C. Some recrystallization did take place but no crystals of sufficient size for single crystal experiments could be obtained.

3.2 Instrumentation

3.2.1 Powder X–ray Diffraction

All powder X–ray diffraction was performed using a Guinier – Hägg focusing camera (λ Cu $K\alpha_1 = 1.54056$ Å). The camera contains a large single crystal quartz monochromator oriented so that one set of strongly diffracting planes is at the Bragg angle to the incident beam. The Bragg angle is set so that only the Cu Kα_1 rays are diffracted, giving a monochromatic beam. The crystal is also bent so that the divergent incident beam is diffracted into a convergent beam focused onto the diffraction circle, which holds a piece of film. The geometry of the camera is shown in Fig. 3.1. The monochromatic radiation passes through the sample at X. Radiation which is not diffracted by the sample will focus on a beam stop at A while diffracted beams will focus on the circle at B, C, etc. where their position is recorded by the film. The beam stop is briefly removed to allow a reference line at $2\theta = 0$ to be recorded. The positions and intensities of the lines were then measured using a KEJ LS–20 computer controlled digital line scanner. The unit–cell dimensions were determined from the powder data using the computer software program LSUDF.

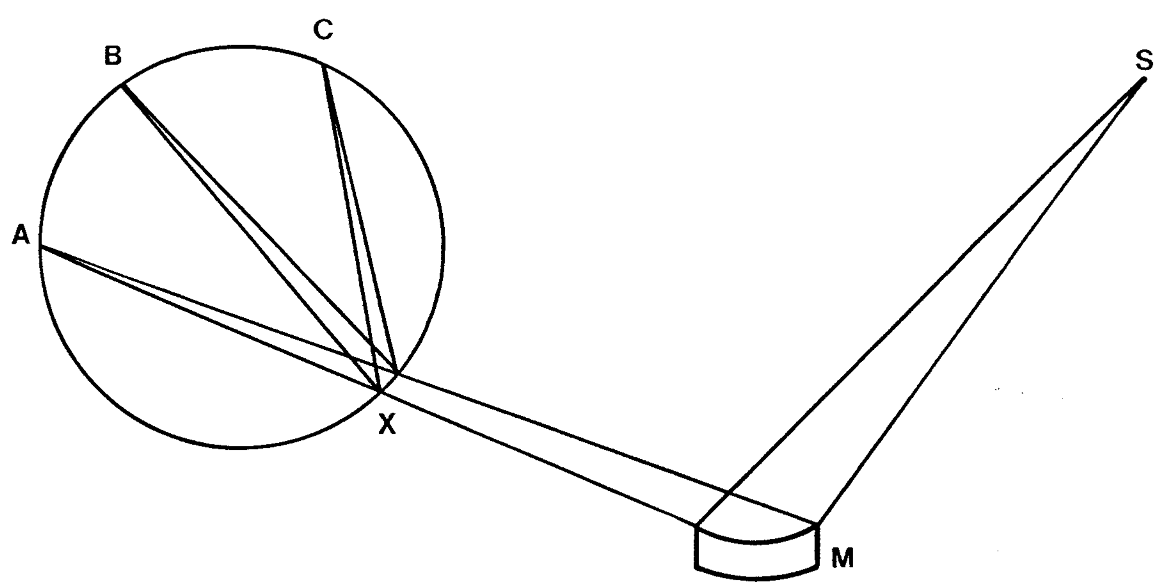

Fig. 3.1 The geometry of the focusing Guinier camera. (West 1989)

3.2.2 Electron Microscopy

A Philips CM–12 transmission electron microscope (TEM) operating at 120 keV was used to examine microscopic single crystals in the powder samples. The TEM was operated in the bright field imaging (BF) and selected area electron diffraction (SAED) modes. A ray diagram in comparing these modes is shown in Fig. 3.2. In the BF mode the intermediate lens

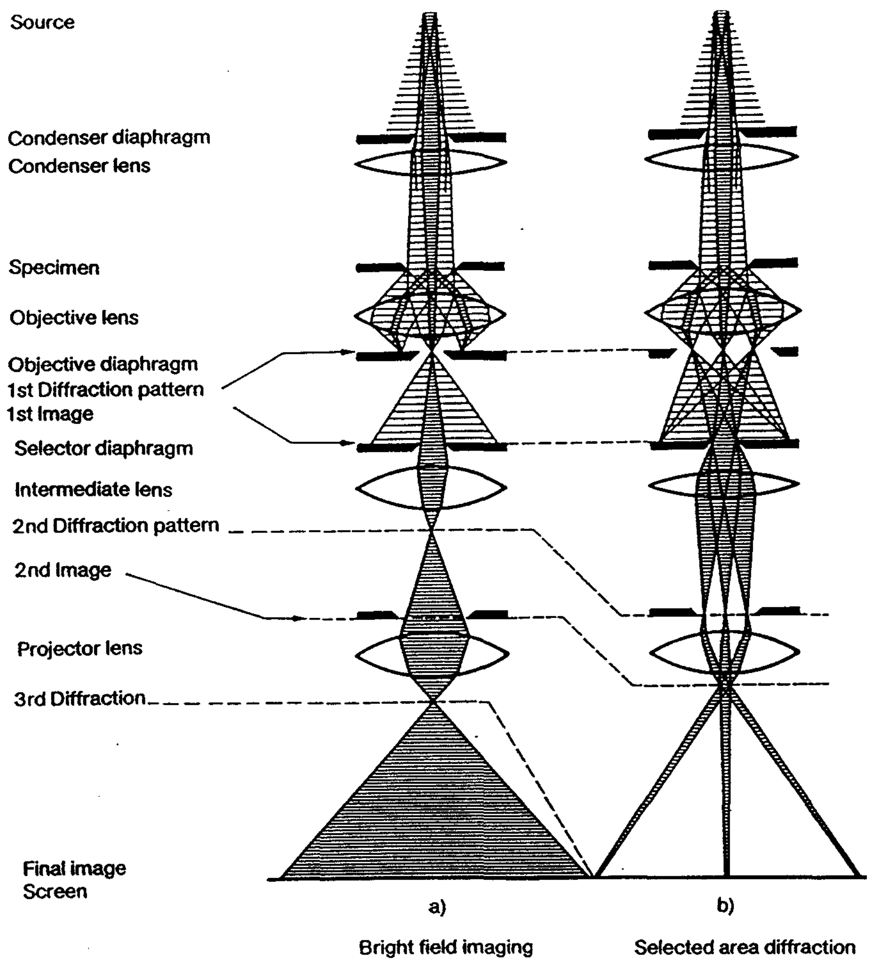

Fig. 3.2 Ray diagram for a transmission electron microscope in **a)** the imaging and **b)** selected area diffraction (SAED) modes. (Reimer 1984)

is focused on the image plane of the objective lens, whereas during an SAED experiment it is focused on the back focal plane of the objective lens. When an SAED aperture is in place, the SAED pattern will directly correspond to the material in the region being imaged in the BF mode.

3.2.3 Single Crystal Precession Photographs

Single crystal precession photographs (λ Mo Kα = 0.71069 Å) were taken to determine the nature and quality of spinelloid single crystals obtained from the flux–growth experiments. Each crystal was mounted on a glass fiber using nail polish as an adhesive. The fiber was mounted on a goniometer and aligned on the camera so that one cell – axis was parallel to the beam. This axis was then offset by an angle μ and precessed about the beam. By keeping a piece of film tangential to the sphere of reflection of the moving crystal, the precession camera allows undistorted images of the reciprocal lattice to be obtained. Photographs of two zero–layers and a first layer containing the b*–axis (i.e. [hk1] or [1kl]) were taken for candidate crystals. It was important to have the b*–axis in the photographs because it is the only characteristic axis in these phases (c.f. –Table 4.1). Crystals of each phase were identified and one crystal of highest quality was selected from each phase for measurement on the single crystal diffractometer.

3.2.4 Single Crystal Diffractometer Data Collection

The single crystal diffractometer data sets were collected on a Syntex $P2_1$ diffractometer (λ Ag Kα = 0.56086 Å) using the Seimens P3/V Data Collection System. The beam was monochromatated by diffracting it off of a highly oriented graphite crystal. Four independent arcs, ϕ, χ, ω, and 2θ (see Fig. 3.3) were used to bring any desired plane into a diffracting position. A detector mounted on the 2θ arc measures the intensity of each reflection, allowing a quantitative determination of the atomic positions within the unit–cell as outlined in Chapter 2. The structure for each crystal was refined by obtaining an initial solution via direct methods. This initial model was then used to locate all of the oxygen and cation positions in the lattice and to determine approximate temperature factors for each site. To prevent strong correlations in the matrix, the temperature factors were fixed before the silicon/gallium occupancies of the tetrahedral sites were allowed to vary. After obtaining an approximate cation distribution for these sites, the occupancies were again fixed and the temperature factors then allowed to vary. This iteration was repeated until no significant changes occurred, at which point the occupancies were fixed. The cation distributions of the octahedral sites were then calculated and input into the model (see Chapter 4.2) and once again a process of alternately fixing and refining the occupancies and temperature factors was undertaken. A final set of refinements, using anisotropic temperature factors, was then performed.

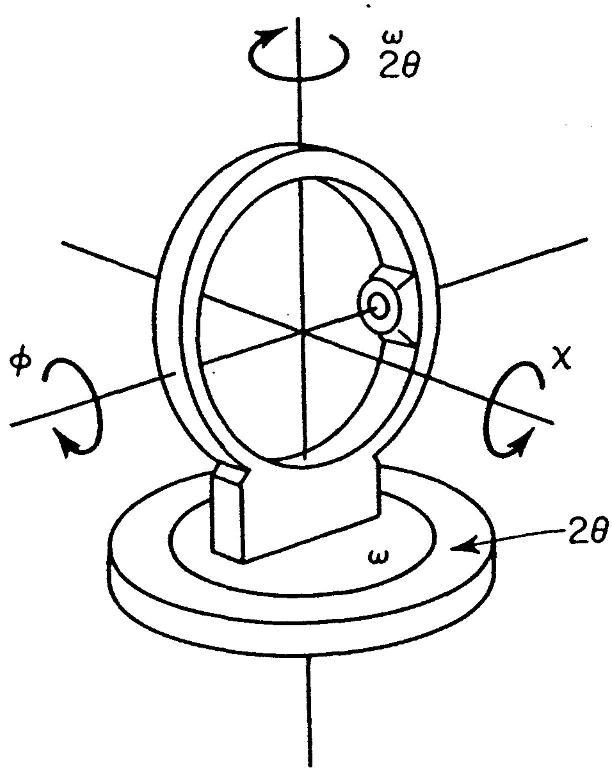

Fig. 3.3 A schematic of a four–circle diffractometer. (Stout and Jensen 1989)

CHAPTER 4

EXPERIMENTAL RESULTS

Of the six systems examined only the $NiGa_2O_4 - Ni_2SiO_4$ system formed spinelloid phases at room pressure. All other systems contained only spinel, olivine, and starting oxides in the reaction products, with the exception of the $CdGa_2O_4 - Cd_2GeO_4$ reaction, which also formed the garnet phase, $Cd_3Ga_2Ge_3O_{12}$. These systems will not be further discussed in this work.

4.1 Powder Sample Results

A survey of the ternary oxide system NiO–Ga_2O_3–SiO_2 was conducted over the temperature range 1200° C to 1550° C at 1 atm pressure. Three previously unknown phases, identified as spinelloid phases I, II, and V, were found, and these were the only ternary phases observed in the system. All of the reaction products contained small amounts of NiO and/or SiO_2. The presence of these phases was probably a result of slow kinetics or of the decomposition of Ni_2SiO_4 olivine at temperatures above 1400° C (Phillips et al. 1963).

The spinelloids were found for compositions lying on the $NiGa_2O_4$–Ni_2SiO_4 pseudo–binary join. Our study focused on the ideal compositions of phase I (3 $NiGa_2O_4$: 1 Ni_2SiO_4 – Ma et al. 1975), phase II (3 $NiGa_2O_4$: 2 Ni_2SiO_4 – Ma 1972), and phase V ($NiGa_2O_4$: Ni_2SiO_4 – Horioka et al. 1981b). At temperatures of 1400 to 1450° C, the gallium–rich 3:1 composition contained mostly spinel whereas, in the initial stages of the reaction, the more silica–rich 7:5 and 1:1

compositions yielded mixtures of spinel and phase II. After prolonged heating (8 days or more), these initial products reacted further, first into a mixture of phases I and V, and eventually phase I alone. A similar phase V → I transformation was observed in samples sintered directly at 1475 to 1525°C, but with no phase II observed in the initial products. Later experiments showed that after 2 hours at 1475°C samples contained large amounts of phase II, but evidence of phase I formation was already apparent from the powder x–ray patterns. At 1550°C, the 3:1 and partially melted 7:5 compositions contained only spinel, while the partially melted 1:1 composition contained only phase V.

It is therefore clear that, in the nickel gallosilicate system, phase V is stable above 1550°C while phase I is stable between 1400 – 1525°C, and that phase II is stable only at lower temperatures. The appearence of phase V as an intermediate phase at 1400 – 1525°C may be a result of the sluggish reaction rates of silicates: with a poorer silicon content than phase I (see section 4.2), phase V would form first as the spinel phase initially present in the sample begins to react with the nickel oxide and silica. Thus the transitional phase V observed below 1550°C would arise from kinetic, rather than thermodynamic, effects.

4.1.1 X–Ray Diffraction

The powder X–ray diffraction patterns of phases I, II, and V were indexed using an automatic indexing program written by Visser (1969). The non–extinction conditions were consistent with the space groups *Pmma* (phases I and V) and *Imma* (phase II) as determined earlier for the spinelloid phases of the $NiAl_2O_4$–Ni_2SiO_4 system. The unit–cell parameters are given in Table 4.1 together with those for the nickel aluminosilicate phases (Ma et al. 1975, Ma and Tillmans 1975, Horioka et al. 1981b). The axial ratios are virtually identical for both series of phases indicating identical small distortions from ideal cubic close packing. As expected from the longer Ga-O bond, Ga substitution for Al results in larger cell parameters with an isotropic volume increase of approximately 6% for all three phases.

The intensities of the powder X-ray diffraction lines were measured using a computer - controlled KEJ LS-20 line scanner. The intensities were also calculated using the program "Lazy Pulverix" (Yvon et al. 1977) and the previously published atomic positions and cation distributions of the nickel aluminosilicate phases (Ma et al. 1975, Ma and Tillmans 1975, Horioka et al. 1981b). These calculated intensities show a reasonably good agreement with the observed intensities (Tables 4.2, 4.3, 4.4) thus supporting the isotypy of the gallium and aluminum phases. This isotypy was later confirmed from the single crystal results (c.f. section 4.2) which were then used in a second set of calculations, also presented in Tables 4.2 - 4.4. This second set of calculated intensities is very similar to the first, thus the discrepancies between the calculated and observed values may be due to a preferred orientation of the powder sample rather than cation distributions.

Table 4.1: Unit–cell data for spinelloid phases I, II, and V in the NiX_2O_4 – Ni_2SiO_4 (X=Al, Ga) systems.

Phase	Cell Parameters (Å) a	b	c
I(X=Al)[1]	5.6664(5)	11.496(2)	8.093(7)
I(X=Ga)[4]	5.7743(5)	11.7152(9)	8.2364(6)
II(X=Al)[2]	5.6603(7)	17.298(2)	8.110(1)
II(X=Ga)[4]	5.765(1)	17.619(3)	8.238(2)
V(X=Al)[3]	5.665(1)	8.590(1)	8.097(3)
V(X=Ga)[4]	5.7914(4)	8.7809(7)	8.2346(6)

[1] Ma et al., 1975
[2] Ma and Tillmans, 1975
[3] Horioka et al. 1982b
[4] powder data for the gallosilicate phases

Table 4.2: Powder X–ray diffraction pattern of phase I nickel gallosilicate (*Pmma*, cell parameters in Table 4.1). Observed intensities were measured using a KEJ LS–20 line scanner. Calculated intensities are based on the aluminosilicate data for I_{cal1} and on single crystal gallosilicate data (cf. Section 4.2) for I_{cal2}.

h k l	d_{cal}	d_{obs}	I_{cal1}	I_{cal2}	I_{obs}
0 0 1	8.236	8.217	65	66	51
1 0 1	4.728	4.726	854	1000	207
1 0 2	3.3528	3.3533	61	102	56
1 1 2	3.2234	3.2235	175	144	118
0 4 0	2.9288	3.9285	132	125	170
1 2 2	2.9099	2.9101	37	41	185
2 0 0	2.8872	2.8872	138	132	145
0 4 1	2.7595	2.7595	27	28	35
0 0 3	2.7455	2.7456	149	140	147
2 0 1	2.7246	2.7249	177	166	182
0 1 3	2.6730	2.6735	233	213	228
1 3 2	2.5438	2.5438	102	80	134
1 4 1	2.4898	2.4894	506	433	858
0 2 3	2.4860		140	161	
1 0 3	2.4795	2.4794	228	200	316
2 2 1	2.4704	2.4706	274	305	239
0 4 2	2.3868	2.3870	140	135	128
2 0 2	2.3640	2.3642	174	166	165
0 3 3	2.2459	2.2457	81	72	84
2 3 1	2.2345	2.2343	192	177	192
1 4 2	2.2058	2.2055	23	43	113
0 0 4	2.0591	2.0574	205	206	750
2 4 0	2.0561		456	459	
0 4 3	2.0030	2.0027	48	43	65
2 4 1	1.9949	1.9950	100	91	128
3 0 1	1.8743	1.8734	56	66	34
0 5 3	1.7822	1.7831	28	24	43
2 5 1	1.7765	1.7768	69	62	104
3 1 2	1.7247	1.7252	27	23	35
1 6 2	1.6873	1.6873	7	7	54
0 4 4	1.6845	1.6848	41	39	79
2 0 4	1.6764	1.6764	64	62	102
3 2 2	1.6712	1.6709	8	8	54
0 1 5	1.6312	1.6306	17	15	47
1 4 4	1.6171	1.6162	19	17	42
0 6 3	1.5912	1.5916	27	31	135
0 2 5	1.5858	1.5861	44	47	231
1 0 5	1.5841	1.5843	58	47	156
3 4 1	1.5787	1.5786	138	119	527
0 3 5	1.5178	1.5177	46	45	77
1 7 2	1.4974	1.4973	9	6	50
0 8 0	1.4644	1.4644	268	262	366
2 4 4	1.4550	1.4549	1000	979	1000
4 0 0	1.4436	1.4437	264	258	343

Table 4.3: Powder X–ray diffraction pattern of phase II nickel gallosilicate (*Imma*, cell parameters in Table 4.1). Observed intensities were measured uing a KEJ LS–20 line scanner. Calculated intensities are based on the aluminosilicate data for I_{cal1} and on single crystal gallosilicate data (cf. Section 4.2) for I_{cal2}.

h k l	d_{cal}	d_{obs}	I_{cal1}	I_{cal2}	I_{obs}
1 1 2	3.292	3.295	203	188	40
0 6 0	2.9364	2.9361	111	107	150
1 3 2	2.9109	2.9104	47	99	74
2 0 0	2.8826	2.8847	107	122	282
0 1 3	2.7133	2.7133	452	404	211
2 1 1	2.6889	2.6893	600	571	155
1 6 1	2.4938	2.4959	1000	1000	1000
1 0 3	2.4791	2.4791	396	419	231
0 6 2	2.3911	2.3901	123	121	101
2 0 2	2.3617	2.3610	163	152	157
0 5 3	2.1660	2.1672	110	95	38
2 5 1	2.1535	2.1534	252	243	43
0 0 4	2.0587 }		210 }	197 }	
		2.0573			681
2 6 0	2.0571 }		479 }	452 }	
0 7 3	1.8555	1.8551	55	54	35
2 7 1	1.8476	1.8484	126	117	58
3 1 2	1.7330	1.7340	30	28	6
2 5 3	1.7316	1.7325	16	14	5
2 0 4	1.6757	1.6758	61	56	74
1 0 5	1.5842	1.5837	121	126	300
3 6 1	1.5782	1.5787	252	269	407
3 5 2	1.5612	1.5609	52	50	8
1 7 4	1.5363	1.5350	27	22	7
0 5 5	1.4925	1.4922	73	68	32
0 12 0	1.4682	1.4678	252	247	231
2 6 4	1.4554	1.4548	920	908	862
1 11 2	1.4452	1.4445	31	31	179
4 0 0	1.4413	1.4419	246	240	199

Table 4.4: Powder X–ray diffraction pattern of phase V nickel gallosilicate (*Pmma*, cell parameters in Table 4.1). Observed intensities were measured using a KEJ LS–20 line scanner. Calculated intensities are based on the aluminosilicate data for I_{cal1} and on single crystal gallosilicate data (cf. Section 4.2) for I_{cal2}.

h k l	d_{cal}	d_{obs}	I_{cal1}	I_{cal2}	I_{obs}
0 1 1	6.000	6.001	111	54	27
1 0 1	4.7326	4.7333	170	90	79
0 3 0	2.9228	2.9233	104	120	116
2 0 0	2.8929	2.8924	103	133	114
0 0 3	2.7424	2.7443	71	51	65
2 0 1	2.7291	2.7297	82	63	45
1 2 2	2.6632	2.6630	201	258	225
0 1 3	2.6174	2.6170	332	309	301
2 1 1	2.6058	2.6054	535	517	284
1 3 1	2.4868	2.4783	1000	1000	1000
1 0 3	2.4781	2.4783	430	438	238
0 3 2	2.3826	2.3830	132	128	114
2 0 2	2.3663	2.3663	154	140	161
0 2 3	2.3250	2.3239	143	150	125
2 2 1	2.3169	2.3164	341	330	196
1 3 2	2.2031	2.2033	29	20	27
0 0 4	2.0568 }	2.0564	245 }	174 }	619
2 3 0	2.0561 }		532 }	377 }	
2 3 1	1.9947	1.9949	42	33	31
1 4 2	1.8347	1.8347	42	52	33
0 4 3	1.7123	1.7116	46	41	63
2 4 1	1.7090	1.7092	104	119	77
0 3 4	1.6821	1.6820	32	46	49
2 0 4	1.6763	1.6763	50	55	55
3 2 2	1.6223	1.6219	48	61	68
0 1 5	1.6172	1.6176	31	36	30
1 0 5	1.5827	1.5823	118	123	191
3 3 1	1.5798 }	1.5794	243 }	248 }	361
3 0 3	1.5776 }		111 }	116 }	
1 5 2	1.5539	1.5544	34	46	23
0 2 5	1.5405	1.5405	74	79	73
0 6 0	1.4614	1.4611	256	237	211
2 3 4	1.4541	1.4541	970	910	831
4 0 0	1.4464	1.4461	253	234	254
3 4 2	1.3658	1.3663	18	22	60

4.1.2 Electron Diffraction

By using electron diffraction on microscopic single crystals, patterns for the [001] and [100] zones of each of the three spinelloid phases were obtained (Figs. 4.1 and 4.2), confirming the unit – cell dimensions determined by powder X–ray diffraction. The [001] orientation shows the systematic absences resulting from the *a*–glide in all three phases, giving similar patterns for phases II and V (due to the extinctions from the *I*–centring of phase II and the fact that $b_{II} \approx 2b_V$ –cf. Table 4.1). However very weak extra reflections of the type {1k0} were observed in the [001] patterns of the flux–grown samples of phases II and V (Fig. 4.2c). The presence of an *a*–glide requires that $h=2n$ for $hk0$ reflections, and so any combination of allowed reflections (i.e. double diffraction) will still result in $h=2n$. Thus the observed extra reflections are primary reflections and indicate a loss of the *a*–glide symmetry. Since these extra reflections were not present in the patterns of sintered samples quenched from high temperature, the associated loss of symmetry may be the result of atomic ordering (probably Ga^{3+}/Si^{4+} on tetrahedral sites) taking place during the slow crystallization process. However, the very weak intensity of these forbidden reflections (undetected on Guinier and precession film patterns) indicates that the structures of the gallosilicate spinelloid phases do not deviate much from the *Pmma* and *Imma* symmetries. This was confirmed by the single crystal analyses.

4.1.3 Electron Microscopy

High resolution electron microscopy was used to obtain lattice images of the spinelloid structures. All of the products showed evidence of decomposition in the electron beam, accompanied by the formation of small crystallites. Fig. 4.3

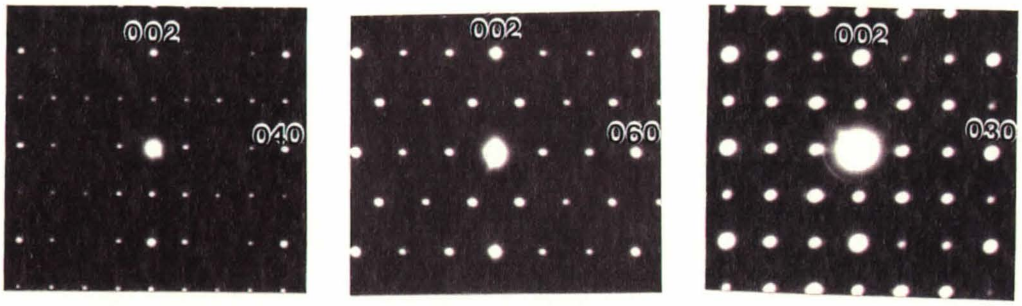

Fig. 4.1 [100] zone axis electron diffraction patterns of nickel gallosilicate: (a) phase I, (b) phase II, (c) phase V. The observed patterns are consistent with the *Pmma* space group for phases I and V and the *Imma* space group for phase II.

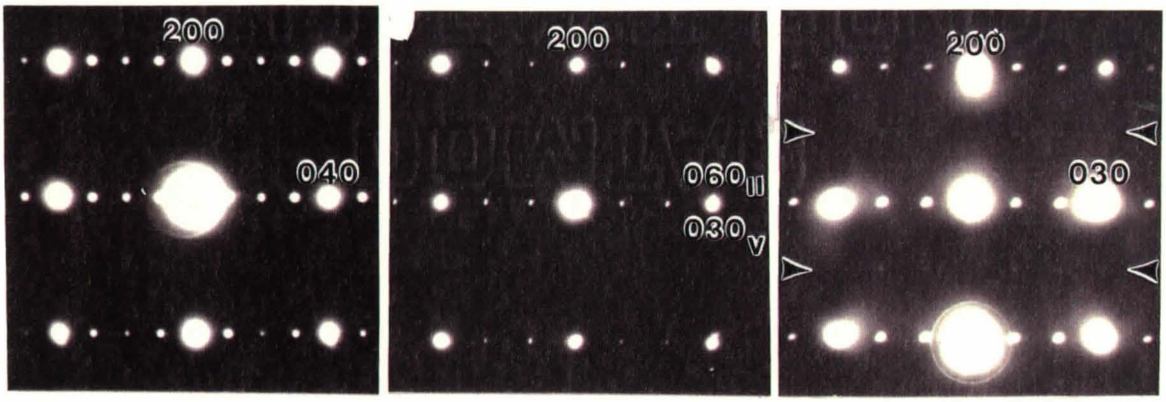

Fig. 4.2 [001] zone axis electron diffraction patterns of nickel gallosilicates: (a) phase I, (b) phase II, (c) phase V. The sample of the flux–grown phase V crystal in (c) shows extra rows of reflections in violation of the *a*–glide.

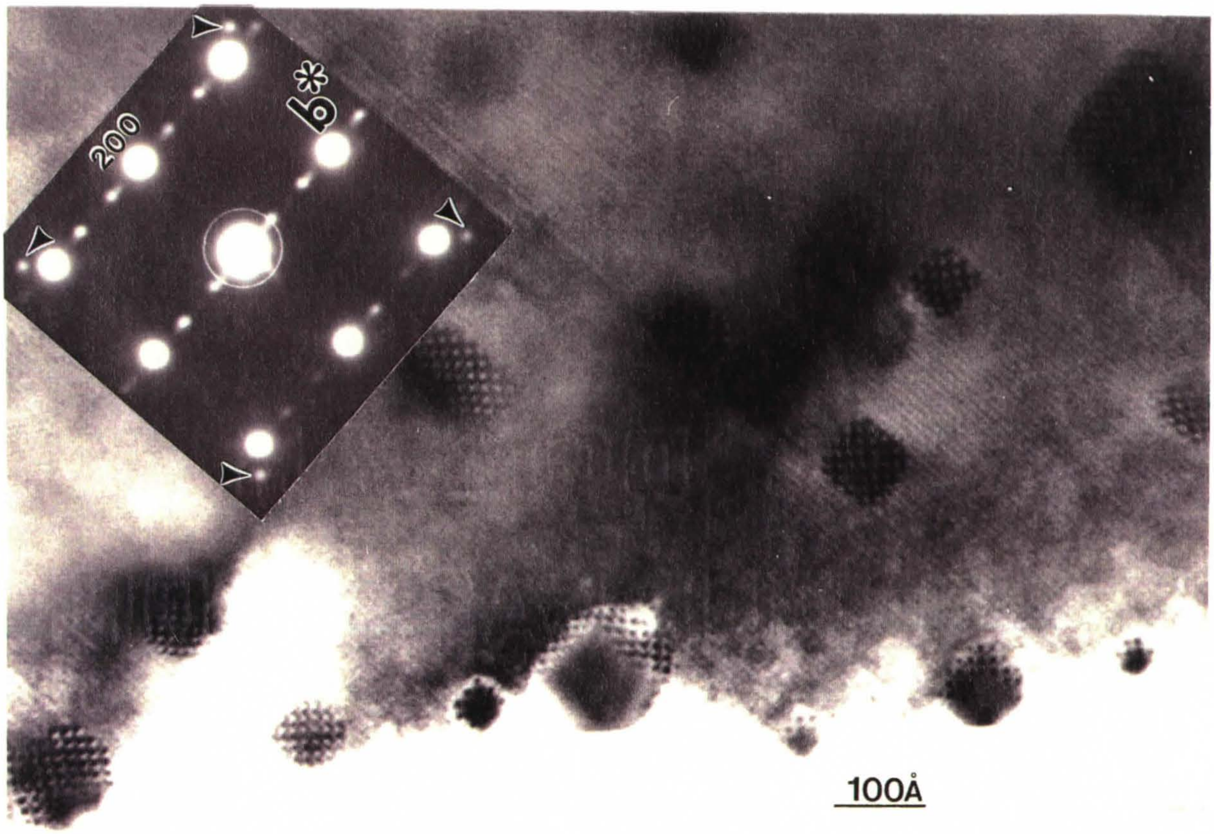

Fig 4.3 High resolution image of a heavily irradiated spinelloid crystal viewed along the [001] zone axis. The Moiré fringes created by the formation of small crystallites on the sample surface are clearly visible. Extra reflections (indicated by arrows) in the diffraction pattern identify the crystallites as Ni metal (d_{200} = 1.77Å). Weaker satellite reflections arising from double diffraction are also visible.

shows a sample with very distinct Moiré fringes arising from the crystallites formed on the sample surface by beam damage. Using the diffraction pattern of a heavily irradiated crystal, these crystallites have been identified as nickel metal.

The [100] direction was the best orientation for imaging work because of the structural relationship between the spinelloid phases. According to Hyde et al. (1982) the spinelloid phases can be described as various sequences of identical building blocks joined by anti–phase boundaries (S), and mirror twin planes (T), parallel to the (010) planes of the spinelloids (see Fig 1.3). These two types of boundaries are observed by viewing the structure along the [001] and [100] directions but, due to the shorter *a*–dimension (≈ 5.8 Å, c.f. Table 4.1), the [100] orientation is preferred for recording lattice images.

Most of the crystals examined were well ordered, although it was possible to observe intergrowth of various phases, especially in samples which had not completely reacted. Intergrowths of spinel and phase I, and of phases I, II, and V are shown in Figs. 4.4 and 4.5 respectively, where the phases intergrow in the *ac*–plane as expected from their structural relationships. Unlike the $NiAl_2O_4$–Ni_2SiO_4 system where intergrowths of large domains of spinel with phase I and to some extent, phase II have been observed (Barbier 1985), the $NiGa_2O_4$–Ni_2SiO_4 system shows only small scale intergrowth with narrow slabs, only a few unit–cells thick, of one phase intergrown in a matrix of another phase. This difference perhaps reflects a lower solubility of Ni_2SiO_4 olivine in the $NiGa_2O_4$ spinel, resulting in a larger composition difference between, for instance, spinel and phase I in the gallosilicate system. Thus intergrowths in this system would involve stronger composition gradients which are unlikely to occur in crystals synthesized under the near equilibrium conditions used here (i.e. prolonged heating or slow cooling from a melt).

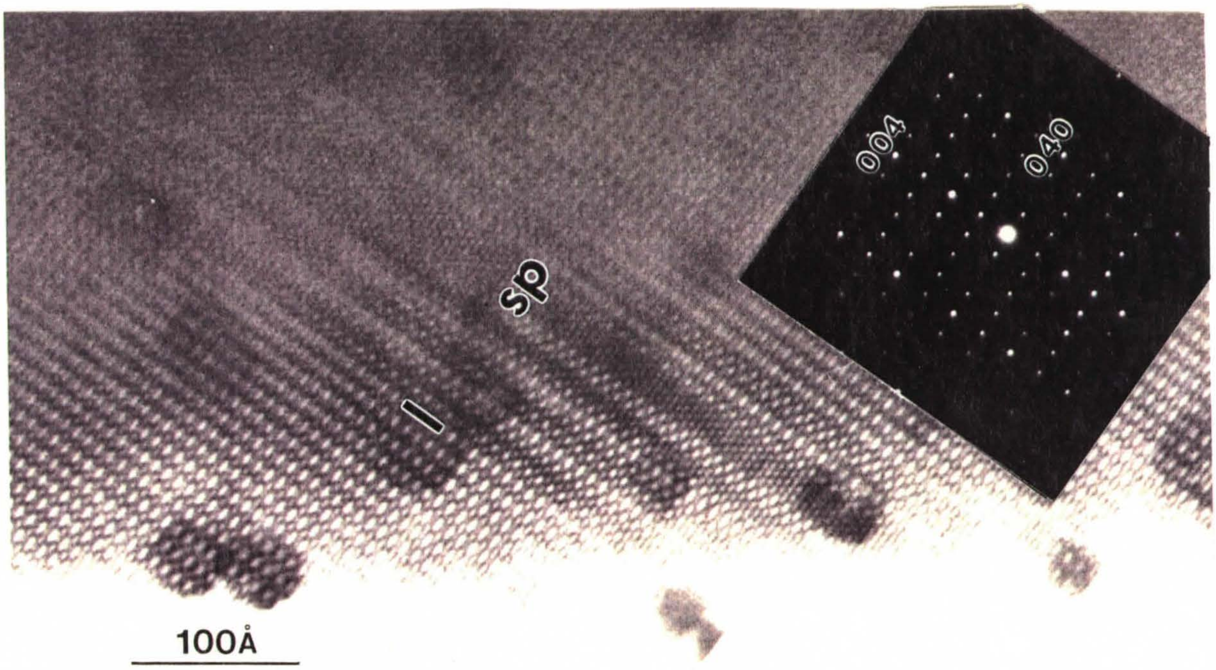

Fig. 4.4 High resolution image of nickel gallosilicate I viewed along the [100] zone axis. The image shows thin (less than 50Å wide) regions of spinel (sp) intergrown with the phase I.

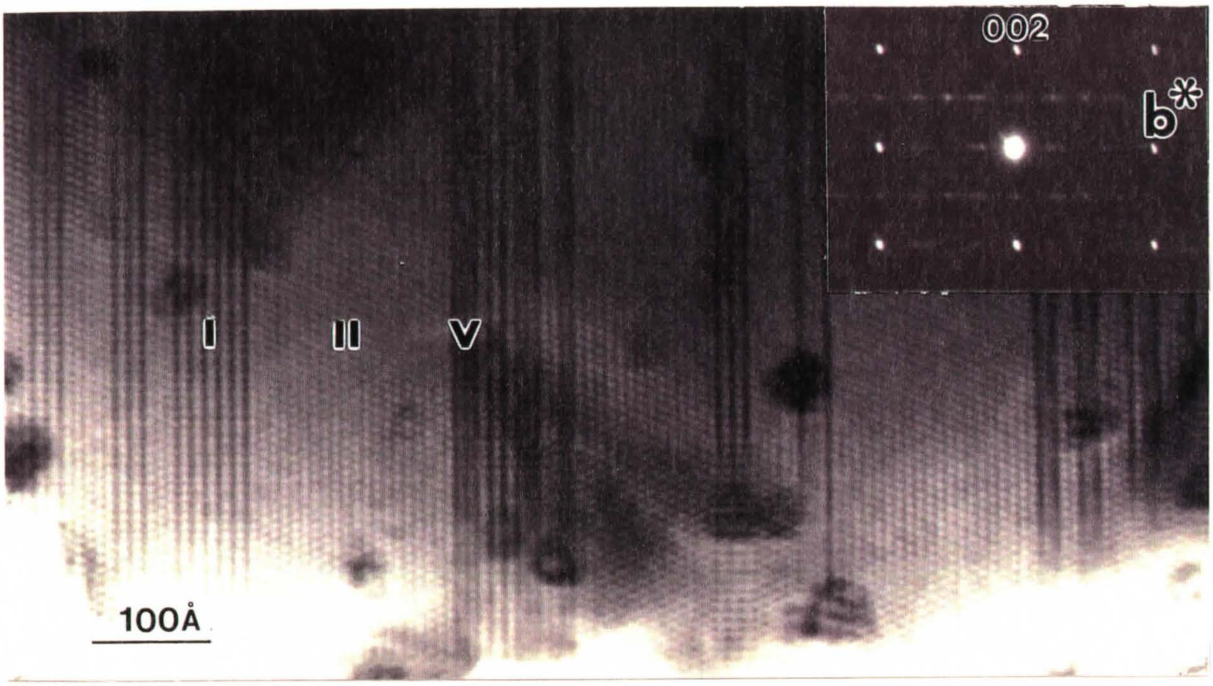

Fig. 4.5 High resolution image of a disordered nickel gallosilicate II crystal viewed along the [100] zone axis. Narrow slabs (less than 100Å wide) of phases I and V (identified by their *b*–axis repeats) are intergrown with the phase II matrix

4.2 Single Crystal Results

The thermodynamic stability of all three spinelloid phases of the $NiGa_2O_4$ – Ni_2SiO_4 system has been confirmed by the successful growth of single crystals from a slowly cooled silica–rich melt, which is expected to produce phases with the richest possible Si content. All samples cooled from 1600° C contained some crystals of spinel, but those cooled from 1575° C showed no evidence of any spinel formation. Samples of 3 NiO : Ga_2O_3 : SiO_2 composition with a 30 wt% excess of SiO_2 added, produced crystals of phase V when cooled to 1540° C. However, crystals of phases I and V were obtained from similar samples cooled further (to 1525° C). This is consistent with the result of the powder experiments, namely, that phase V is the higher temperature phase. Single crystals of phase II were obtained from an Ni_2SiO_4–rich sample of 5 NiO : Ga_2O_3 : 2 SiO_2 composition with a 30 wt% excess of SiO_2 which was heated to 1600° C and then cooled to 1525° C. The cooling rate (i.e 1 or 2° C/hr) had no noticeable effect on the crystal growth.

All crystal structures were solved using direct methods. The structure solutions were complicated by the necessity to refine the occupancies of all of the cation sites. The occupancies of the octahedral sites were particularly difficult to obtain because of the inability to distinguish Ni from Ga, a result of their similar electron densities. In determining the cation occupancies it was assumed that the nickel atoms occupied only the octahedral sites and that the silicon atoms only sat at tetrahedral sites. The stoichiometry of the crystal could then be determined by refining the silicon/gallium occupancy of the tetrahedral sites and assuming that the composition could be written as p $NiGa_2O_4$: q Ni_2SiO_4 (i.e. on the join). The octahedral cation sites were then given a uniform nickel/gallium distribution based on this stoichiometry. After a few cycles of refinement the occupancy of each octahedral site was individually estimated using the current bond lengths, the bond valence / bond length relationship (Brown and Altermatt 1985) and equations [4.1] and [4.2]:

$$n_{Ni}V_{Ni} + n_{Ga}V_{Ga} = 2\,n_{Ni} + 3\,n_{Ga} \qquad [4.1]$$

$$n_{Ni} + n_{Ga} = 1 \qquad [4.2]$$

In these equations n_{Ni} and n_{Ga} are the cation occupancies of the site and V_{Ni} and V_{Ga} are the bond valence sums corresponding to full site occupancy by Ni and Ga respectively. The occupancies were then scaled to provide a stoichiometry consistent with the tetrahedral population. The composition was allowed to vary during a few cycles of refinement and was then kept constant during the final cycles in order to be able to refine the temperature factors.

4.2.1 Phase I

The phase I crystal was a dark green rectangular plate measuring 0.05 x 0.12 x 0.20 mm. It was found to have an orthorhombic unit cell with dimensions *a* = 5.778(2), *b* = 11.723(2), *c* = 8.243(2) Å in space–group *Pmma*, slightly larger than that given by the powder data (c.f. Table 4.1), and a calculated density of 5.857 g/cm^3. The unit cell contents were determined to be $Ni_{10\cdot3(2)}Ga_{11\cdot2(2)}Si_{2\cdot47(2)}O_{32}$. Assuming the composition lies on the spinel – olivine join, this gives a composition of 2.8 $NiGa_2O_4$: 1.2 Ni_2SiO_4 or $Ni_{10\cdot4}Ga_{11\cdot2}Si_{2\cdot4}O_{32}$, which is within the error limits of the refined value. As expected from the crystal growth conditions this is on the Si–rich end of the composition range of the aluminosilicate phase, which has an idealized composition of 3 $NiAl_2O_4$: Ni_2SiO_4 (Ma 1972).

The linear absorption coefficient was calculated as 11.51 mm^{-1} and F(000) = 928. The data was collected over one octant at 28°C and contained 2757 independent reflections. The reflections were measured by a $\theta - 2\theta$ scan over the range from 3° $< 2\theta \leq 70°$ resulting in index ranges of $0 \leq h \leq 11$, $0 \leq k \leq 24$, and $0 \leq l \leq 16$. The cell parameters were obtained using 23 reflections over 12.9° $\leq 2\theta \leq$ 45.0°. A numerical absorption correction was made by approximating the crystal using six boundary planes, with (0 1 0) planes bounding the top and bottom of the plate, (1 0 0) planes bounding the shorter (0.12 mm) faces of the plate's side, and (0 0 1) planes bounding the longer (0.20 mm) faces. Three standard reflections, (4 0 0), (0 8 0), and (0 0 8) were checked every 100 reflections. Of the 2757 reflections measured, 1265 were treated as observed ($|F| > 6\sigma|F|$). The model was refined by full–matrix least–squares calculations minimizing Σ $w(F_o - F_c)^2$ using 95 parameters, which included anisotropic temperature factors (see Appendix 1). The final residual error values, using only observed reflections, were R = 0.057 and wR = 0.072. Using all data, the final residual errors were R = 0.129 and wR = 0.118. The final difference Fourier map had $\rho_{min} = -4.10$ e/Å^3 and $\rho_{max} = 4.23$ e/Å^3. A secondary extinction correction (χ = 0.0014(2)) was applied to the data. The scattering factors for the neutral atoms were taken from the *International Tables for X–ray Crystallography.*

The final atomic coordinates, temperature factors, and cation distributions are listed in Table 4.5. The bond lengths and oxygen–metal–oxygen (O–M–O) bond angles are listed in Tables 4.6a and 4.6b respectively. The solution of the crystal structure was not trivial to obtain due to a difficulty in properly correcting for the absorption of the crystal. The inability to make this correction properly for phase I manifests itself primarily in the large errors on some oxygen coordinates and some metal – oxygen bond lengths (c.f. Tables 4.5 and 4.6a). It is also responsible for the large amounts of electron density which remain in the Fourier difference maps and for the somewhat high values of R and wR. Attempts to solve the structure of phase I in *Pmm2*, taking the loss of the *a*–glide into account, were very unsatisfactory.

A projection on (0 0 1) of the phase I structure is shown in Fig. 4.6. Phase I is based on a distorted cubic close – packed oxygen array, with the greatest distortions occurring for those oxygen atoms lying on the twin plane (T) boundaries within the structure. The structure contains two types of tetrahedral groups, isolated TO_4 units and T_3O_{10} chains. As shown in Fig. 4.6 each T_3O_{10} chain contains two crystallographically distinct tetrahedral sites. There is a correlation between the average bond lengths of the three tetrahedral sites and their occupancies (see Table 4.11), corresponding to the Si – O bond being much shorter than the Ga – O bond (1.64 Å vs. 1.85 Å, Shannon 1976). There is a similar, but less obvious, correlation between the mean bond lengths of the five octahedral sites and their Ni/Ga ratios which can be ascribed to the difference in the Ni – O and Ga – O bond lengths (2.09 Å vs. 2.02 Å, Shannon 1976).

4.2.2 Phase II

The phase II crystal used for the structure determination was a dark green rod measuring 0.11 x 0.15 x 0.32 mm. It was found to have an orthorhombic unit cell in the *Imma* space–group with dimensions a = 5.762(2), b = 17.618(2), c = 8.239(2) Å, which closely match those obtained from the powder data (c.f. Table 4.1), and has a calculated density of 5.797 g/cm³. The unit cell contents were determined to be $Ni_{16\cdot2(2)}Ga_{15\cdot5(3)}Si_{4\cdot3(1)}O_{48}$. Again assuming that the composition lies on the spinel – olivine join, this gives a result of 3.9 $NiGa_2O_4$: 2.1 Ni_2SiO_4 or $Ni_{16\cdot2}Ga_{15\cdot6}Si_{4\cdot2}O_{48}$. This composition is again within the error limits of the refined value and is within the composition range of the corresponding aluminosilicate phase, which has an idealized composition of 3 $NiAl_2O_4$: 2 Ni_2SiO_4 (Ma 1972).

The linear absorption coefficient was calculated as 11.25 mm^{-1} and F(000) = 1380. The data were collected over one octant at 25°C and contained 2050 independent reflections. The reflections were measured by a $\theta - 2\theta$ scan over the range from 3° $< 2\theta \leq 70°$ resulting in index ranges of $0 \leq h \leq 11$, $0 \leq k \leq 36$, and $0 \leq l \leq 16$. The cell parameters were obtained using 20 reflections over $14.9° \leq 2\theta \leq 29.9°$. A semi–empirical correction for absorption was made using the psi–scan technique. Three standard reflections, (4 4 0), (0 3 11), and (3 3 0) were checked every 100 reflections. Of the 2050 reflections measured 1272 were treated as observed ($|F| > 6\sigma|F|$). The model was refined by full–matrix least–squares calculations minimizing Σ $w(F_o - F_c)^2$ using 71 parameters, which included anisotropic temperature factors (see Appendix 1). The final residual error values, using only observed reflections, were R = 0.046 and wR = 0.061. Using all data, the final residual errors were R = 0.079 and wR = 0.082. The final difference Fourier map had $\rho_{min} = -5.76$ e/Å^3 and $\rho_{max} = 4.05$ e/Å^3. A secondary extinction correction (χ = 0.00129(8)) was applied to the data. The scattering factors for the neutral atoms were taken from the *International Tables for X–ray Crystallography.*

The final atomic coordinates, temperature factors, and cation distributions are listed in Table 4.7. It should be noted that the cation distribution of the M1, M2, and M3 sites were not refined independently because the occupancies were not well behaved. Bond valence calculations and comparison of the mean bond lengths showed these three sites to be similar, and so they were fixed at equal compositions. The bond lengths and oxygen–metal–oxygen (O–M–O) bond angles are listed in Tables 4.8a and 4.8b respectively.

A projection on (0 0 1) of the phase II structure is shown in Fig. 4.7. Phase II is also based on a distorted cubic close–packed oxygen array, and has the greatest distortions occurring for those oxygen atoms lying on the twin plane (T) boundaries within the structure. The structure contains only T_3O_{10} units, resulting in only two distinct tetrahedral sites. As in phase I there is also a good correlation in the tetrahedral sites between the Si/Ga ratio and the bond lengths (see Table 4.11), again associated with the difference in the Si – O and Ga – O bond lengths.

4.2.3 Phase V

A dark green rod measuring 0.10 x 0.11 x 0.23 mm was used for the structure determination of phase V. It was found to have an orthorhombic unit cell with dimensions $a = 5.786(2)$, $b = 8.776(2)$, $c = 8.230(2)$ Å in space group *Pmma*, slightly smaller than that obtained from the powder data (c.f. Table 4.1), with a calculated density of 6.014 g/cm^3. The unit cell contents were determined to be $Ni_{7.2(1)}Ga_{9.6(1)}Si_{1.20(1)}O_{24}$. This composition lies on the spinel – olivine join, corresponding to 4.8 $NiGa_2O_4$: 1.2 Ni_2SiO_4 or $Ni_{7.2}Ga_{9.6}Si_{1.2}O_{24}$. This composition is much poorer in Si than the corresponding high–pressure aluminosilicate phase, which has an idealized composition of $NiAl_2O_4$: Ni_2SiO_4 or $Ni_9Al_6Si_3O_{24}$.

The linear absorption coefficient was calculated as 12.2 mm^{-1} and F(000) = 708. The data were collected over one octant at 26°C and contained 1728 independent reflections. The reflections were measured by a $\theta - 2\theta$ scan over the range from 3° $< 2\theta \leq 65°$ resulting in index ranges of $0 \leq h \leq 11$, $0 \leq k \leq 16$, and $0 \leq l \leq 15$. The cell parameters were obtained using 26 reflections over 14.6° $\leq 2\theta \leq$ 30.5°. A semi–empirical correction for absorption was made using the psi–scan technique. Three standard reflections, (2 3 4), (3 3 1), and (1 0 5) were checked every 100 reflections. Of the 1728 reflections measured 983 were treated as observed ($|F| > 6\sigma|F|$). The model was refined by full–matrix least–squares calculations minimizing $\Sigma\ w(F_o - F_c)^2$ using 72 parameters, which included anisotropic temperature factors (see Appendix 1). The final residual error values, using only observed reflections, were R = 0.040 and wR = 0.060. Using all data, the final residual errors were R = 0.079 and wR = 0.080. The final difference Fourier map had $\rho_{min} = -2.97$ e/Å^3 and $\rho_{max} = 3.23$ e/Å^3. A secondary extinction correction (χ = 0.0051(4)) was applied to the data. The scattering factors for the neutral atoms were taken from the *International Tables for X–ray Crystallography.*

The final atomic coordinates, temperature factors, and cation distributions are listed in Table 4.9. The bond lengths and angles are listed in Tables 4.10a and 4.10b respectively.

A projection on (0 0 1) of the phase V structure is shown in Fig. 4.8. The structure contains isolated TO_4 units and T_2O_7 units. Like phases I and II, phase V is based on a distorted cubic close–packed oxygen array and has the greatest distortions occurring for those oxygen atoms lying on the twin plane (T) boundaries within the structure. As in phases I and II there is a good correlation between the tetrahedral site occupancies and mean bond lengths (see Table 4.11). The presence of such correlations in all three structures provides further support for the refined cation distributions.

Table 4.5: Atomic Coordinates and equivalent isotropic temperature displacement coefficients (U_{eq}) with cation distributions for phase I. Cation site distributions are given by dGa + (1–d)Ni for octahedral (M) sites and dGa + (1–d)Si for tetrahedral (T) sites.

	x	y	z	U_{eq}	d
M1	0	0	0	0.0057(3)	0.160(10)
M2	0	$^1/_2$	$^1/_2$	0.0065(3)	0.270(10)
M3	0	0.2480(1)	0	0.0060(2)	0.420(20)
M4	$^1/_4$	0.1247(3)	0.2728(1)	0.0056(2)	0.160(10)
M5	$^1/_4$	0.3755(3)	0.2486(1)	0.0061(2)	0.630(20)
T1	$^1/_4$	$^1/_2$	–0.1220(2)	0.0057(3)	0.917(2)
T2	$^1/_4$	0	0.6352(3)	0.0056(4)	0.366(2)
T3	$^1/_4$	0.2574(1)	0.6229(2)	0.0056(2)	0.741(4)
O1	$^1/_4$	0.1254(12)	0.1245(8)	0.0098(13)	
O2	$^1/_4$	0.3675(10)	0.0044(8)	0.0059(12)	
O3	$^1/_4$	0.1227(13)	0.5221(9)	0.0129(15)	
O4	$^1/_4$	0.3749(15)	0.4934(9)	0.0115(14)	
O5	–0.0149(1)	0	0.2507(1)	0.0115(18)	
O6	0.0052(1)	$^1/_2$	0.2539(1)	0.0157(19)	
O7	0.0129(1)	0.2526(1)	0.2476(1)	0.0038(9)	

Table 4.6a: Bond lengths (Å) for Phase I with standard deviations

M1 – O1	4x	2.071(10)		M5 – O2	2x	2.015(7)
M1 – O5	2x	2.068(1)		M5 – O4	2x	2.018(7)
				M5 – O6	1x	2.033(3)
M2 – O4	4x	2.059(12)		M5 – O7	1x	1.989(3)
M2 – 06	2x	2.029(1)				
				T1 – O2	2x	1.871(11)
M3 – O1	2x	2.048(10)		T1 – O6	2x	1.832(1)
M3 – O2	2x	2.012(9)				
M3 – O7	2x	2.043(1)		T2 – O3	2x	1.715(13)
				T2 – O5	2x	1.652(2)
M4 – O1	2x	2.046(7)				
M4 – O3	2x	2.055(7)		T3 – O3	1x	1.812(13)
M4 – O5	1x	2.124(2)		T3 – O4	1x	1.778(14)
M4 – O7	1x	2.042(2)		T3 – O7	2x	1.825(1)

Table 4.6b: O–M–O Bond Angles (°) for Phase I

O1–M1–O1	2x	89.5(6)		O2–M5–O6	2x	93.2(3)
O1–M1–O1	2x	90.5(6)		O2–M5–O7	2x	87.8(3)
O1–M1–O5	4x	86.1(2)		O4–M5–O6	2x	88.9(4)
O1–M1–O5	4x	93.9(2)		O4–M5–O7	2x	90.1(4)
				O6–M5–O6	1x	88.2(1)
O4–M2–O4	2x	89.2(7)		O6–M5–O7	2x	92.3(1)
O4–M2–O4	2x	90.8(7)		O7–M5–O7	1x	87.1(1)
O4–M2–O6	4x	87.9(2)				
O4–M2–O6	4x	92.1(2)		O2–T1–O2	1x	112.3(6)
				O2–T1–O6	4x	109.3(1)
O1–M3–O1	1x	90.8(6)		O6–T1–O6	1x	107.2(1)
O1–M3–O2	2x	88.9(4)				
O1–M3–O7	2x	83.9(2)		O3–T2–O3	1x	114.1(7)
O1–M3–O7	2x	98.2(2)		O3–T2–O5	4x	108.0(2)
O2–M3–O2	1x	91.8(5)		O5–T2–O5	1x	110.6(2)
O2–M3–O7	2x	86.4(2)				
O2–M3–O7	2x	91.5(2)		O3–T3–O4	1x	111.4(5)
				O3–T3–O7	2x	104.1(2)
O1–M4–O5	2x	85.3(3)		O4–T3–O7	2x	112.0(2)
O1–M4–O7	2x	84.0(3)		O7–T3–O7	1x	112.6(1)
O3–M4–O7	2x	96.3(3)				
O3–M4–O5	2x	94.5(3)				
O5–M4–O5	1x	92.2(1)				
O5–M4–O7	2x	90.7(1)				
O7–M4–O7	1x	84.3(1)				

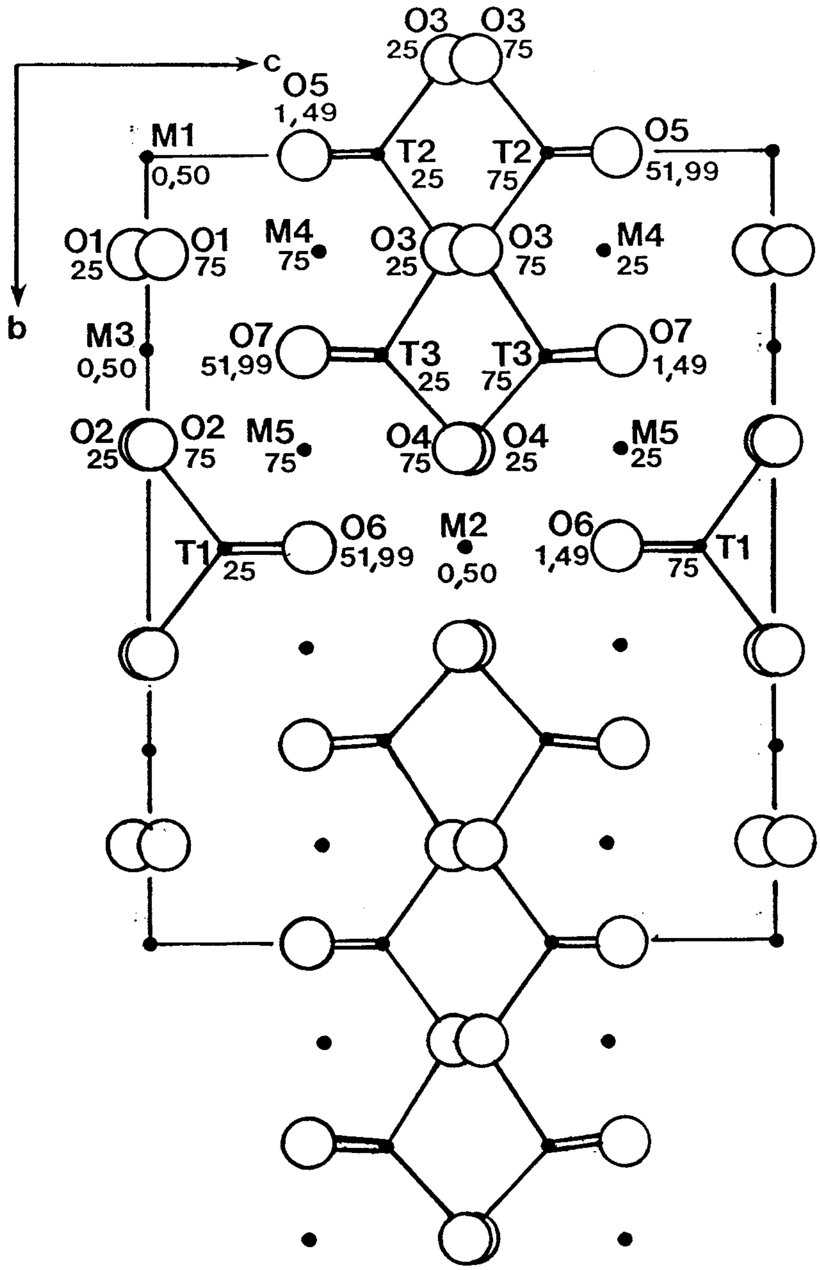

Fig. 4.6 Crystal structure of phase I projected on (100)

Table 4.7: Atomic coordinates and equivalent isotropic temperature displacement coefficients (U_{eq}) with cation distributions for phase II. Cation site distributions are given by dGa + (1–d)Ni for octahedral (M) sites and dGa + (1–d)Si for tetrahedral (T) sites.

	x	y	z	U_{eq}	d
M1	1/4	1/4	3/4	0.0036(2)	0.243(7)
M2	1/4	0.0851(1)	3/4	0.0039(1)	0.243(12)
M3	0	0.1668(1)	0.0221(1)	0.0039(1)	0.243(12)
M4	0	0	0	0.0039(2)	0.732(7)
T1	0	0.4215(1)	0.3786(1)	0.0037(1)	0.782(2)
T2	0	1/4	0.3846(2)	0.0036(2)	0.356(1)
O1	0	0.8319(2)	0.2270(5)	0.0069(7)	
O2	0	–0.0032(3)	0.2450(5)	0.0098(9)	
O3	0	0.1697(3)	0.2702(5)	0.0100(8)	
O4	0.2516(8)	1/4	0.0020(5)	0.0076(9)	
O5	0.2411(5)	0.0818(1)	0.9979(4)	0.0072(6)	

Table 4.8a: Bond lengths (Å) for Phase II with standard deviations

M1 – O1	4x	2.048(3)	M4 – O2	2x	2.020(4)
M1 – O4	2x	2.076(4)	M4 – O5	4x	2.002(3)
M2 – O1	2x	2.061(3)	T1 – O2	1x	1.812(5)
M2 – 02	2x	2.039(4)	T1 – O3	1x	1.839(5)
M2 – O5	2x	2.044(3)	T1 – O5	2x	1.807(3)
M3 – O1	2x	2.052(4)	T2 – O3	2x	1.699(5)
M3 – O3	2x	2.045(4)	T2 – O4	2x	1.709(5)
M3 – O4	1x	2.069(3)			
M3 – O5	1x	2.052(3)			

Table 4.8b: O–M–O Bond Angles (°) for Phase II

O1–M1–O1	2x	89.6(2)	O2–M4–O5	4x	88.4(1)
O1–M1–O1	2x	90.4(2)	O2–M4–O5	4x	91.6(1)
O1–M1–O4	4x	84.9(1)	O5–M4–O5	2x	87.9(2)
O1–M1–O4	4x	95.1(1)	O5–M4–O5	2x	92.1(2)
O1–M2–O1	1x	89.6(2)	O2–T1–O3	1x	113.6(2)
O1–M2–O2	2x	90.4(1)	O2–T1–O5	2x	111.6(1)
O1–M2–O5	2x	84.9(1)	O3–T1–O5	2x	104.2(1)
O1–M2–O5	2x	97.4(1)	O5–T1–O5	1x	111.6(1)
O2–M2–O2	1x	89.0(2)			
O2–M2–O5	2x	92.0(1)	O3–T2–O3	1x	112.6(3)
O2–M2–O5	2x	85.2(2)	O3–T2–O4	4x	107.6(1)
			O4–T2–O4	1x	113.8(3)
O1–M3–O4	2x	85.0(1)			
O1–M3–O5	2x	84.9(1)			
O3–M3–O4	2x	93.5(2)			
O3–M3–O5	2x	96.7(1)			
O4–M3–O4	1x	89.0(2)			
O4–M3–O5	2x	92.0(1)			
O5–M3–O5	1x	85.2(2)			

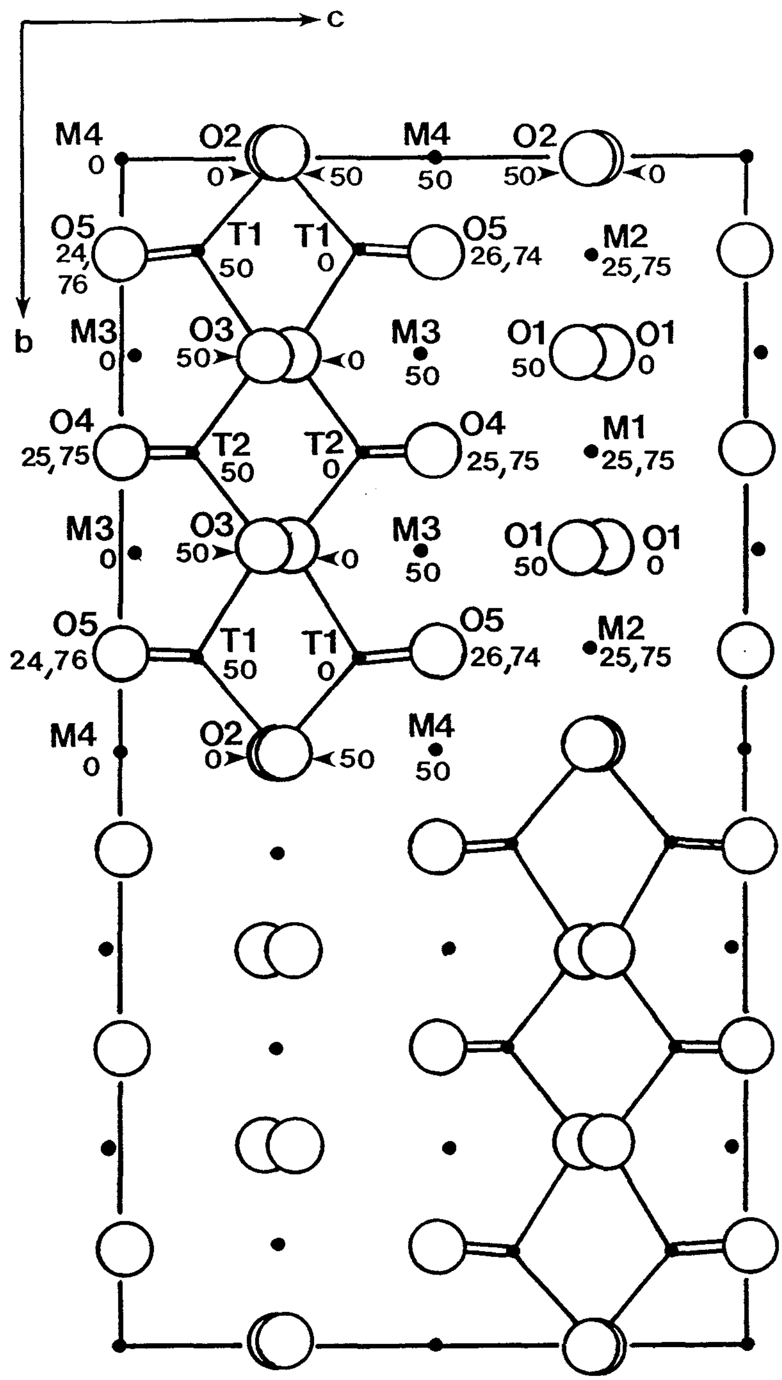

Fig. 4.7 Crystal structure of phase II projected on (100)

Table 4.9: Atomic Coordinates and equivalent isotropic temperature displacement coefficients (U_{eq}) with cation distributions for phase V. Cation site distributions are given by dGa + (1–d)Ni for octahedral (M) sites and dGa + (1–d)Si for tetrahedral (T) sites.

	x	y	z	U_{eq}	d
M1	$^3/_4$	0.01662(2)	0.2509(1)	0.0052(2)	0.615(13)
M2	$^1/_2$	0	0	0.0064(2)	0.424(8)
M3	$^1/_2$	0.3342(1)	$^1/_2$	0.0048(2)	0.322(11)
M4	$^3/_4$	$^1/_2$	0.2258(2)	0.0050(3)	0.307(8)
T1	$^1/_4$	0	0.3767(1)	0.0058(2)	0.949(2)
T2	$^1/_4$	0.3246(1)	0.1292(1)	0.0059(2)	0.726(3)
O1	0.5076(13)	0	0.2464(6)	0.0074(13)	
O2	$^1/_4$	0.1631(9)	0.0016(7)	0.0124(12)	
O3	$^1/_4$	0.1742(6)	0.5049(6)	0.0085(12)	
O4	0.5082(10)	0.3313(5)	0.2514(4)	0.0102(10)	
O5	$^1/_4$	$^1/_2$	0.0163(10)	0.0092(16)	
O6	$^1/_4$	$^1/_2$	0.5238(8)	0.0052(14)	

Table 4.10a: Bond lengths (Å) for Phase V with standard deviations

M1 – O1	2x	2.024(5)		M4 – O2	2x	2.015(7)
M1 – O2	1x	2.052(6)		M4 – O4	2x	2.018(7)
M1 – O3	1x	2.011(5)		M4 – O6	1x	2.033(3)
M1 – O4	2x	2.015(5)		M4 – O7	1x	1.989(3)
M2 – O1	2x	2.028(5)		T1 – O1	2x	1.836(7)
M2 – O2	4x	2.035(6)		T1 – O3	2x	1.858(5)
M3 – O3	2x	2.016(4)		T2 – O2	1x	1.779(7)
M3 – O5	2x	2.047(3)		T2 – O4	2x	1.802(5)
M3 – O6	2x	2.061(1)		T2 – O5	1x	1.789(4)

Table 4.10b: O–M–O Bond Angles (°) for Phase V

O1–M1–O1	1x	87.8(3)		O4–M4–O4	2x	92.6(3)
O1–M1–O2	2x	88.4(2)		O4–M4–O4	2x	86.2(3)
O1–M1–O3	2x	92.5(2)		O4–M4–O5	4x	95.9(1)
O1–M1–O4	2x	92.1(2)		O4–M4–O6	4x	84.1(1)
O2–M1–O4	2x	90.7(2)				
O3–M1–O4	2x	88.4(2)		O1–T1–O1	1x	108.5(4)
O4–M1–O4	1x	88.0(3)		O1–T1–O3	4x	109.4(1)
				O3–T1–O3	1x	110.8(3)
O1–M2–O2	4x	88.8(2)				
O1–M2–O2	4x	91.2(2)		O2–T2–O4	2x	111.3(2)
O2–M2–O2	2x	89.4(3)		O2–T2–O5	1x	112.0(3)
				O4–T2–O5	2x	105.1(2)
O3–M3–O3	1x	91.7(2)				
O3–M3–O3	2x	91.6(2)				
O3–M3–O4	2x	87.4(2)				
O3–M3–O6	2x	89.2(1)				
O4–M3–O6	2x	96.9(2)				
O4–M3–O6	2x	84.1(2)				
O6–M3–O6	1x	90.2(1)				

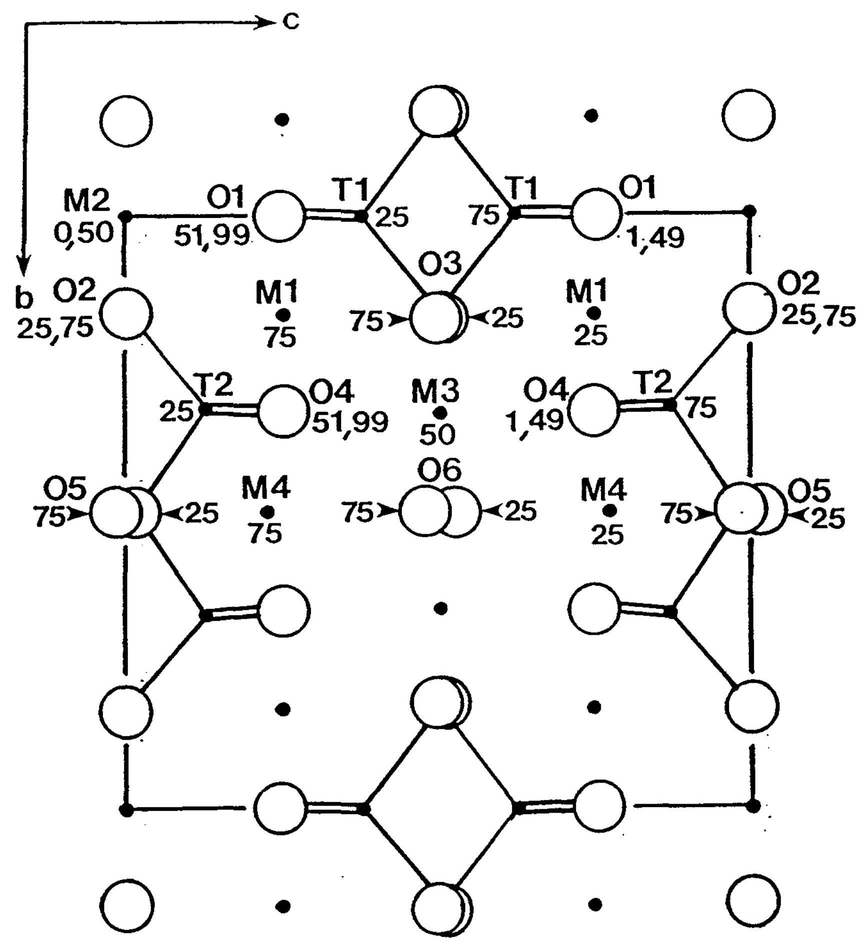

Fig. 4.8 Crystal structure of phase V projected on (100)

Table 4.11: Occupancy (as % Ga) and average bond length (Å) for the tetrahedral cation sites in the nickel gallosilicate spinelloids.

Phase	Cation Site	Occupancy	Average Bond Length
Phase I	T1	91.7	1.852
	T2	36.6	1.684
	T3	74.1	1.810
Phase II	T1	78.2	1.816
	T2	35.6	1.704
Phase V	T1	94.9	1.847
	T2	72.6	1.793

CHAPTER 5

DISCUSSION

The $NiGa_2O_4$ - Ni_2SiO_4 system contains three room-pressure spinelloid phases corresponding to spinelloid phases I, II, and V. Powder sample syntheses and single crystal growth indicate that phase II forms at the lowest temperatures, with phases I and V forming at increasingly higher temperatures. At very high temperatures (1575 to 1600°C) a spinel phase is also formed. Structure refinements using single crystal data indicate an increase in Ni_2SiO_4 content passing from phase V to phase I and phase II. The composition determined for phase V is unexpected since it does not follow the trend observed in the high-pressure aluminosilicate system.

All three spinelloid phases are based on a distorted cubic close-packed oxygen array, with the greatest distortions occurring around those oxygen atoms lying on the twin plane (T) boundaries within the structures. These distortions, associated with the presence of corner sharing tetrahedra, are comparable to those observed in the aluminosilicate phases. A good correlation exists in all three phases between the mean bond lengths of the tetrahedral sites and the gallium content of those sites (cf. Table 4.11). Furthermore, the observed cation distributions clearly show that there is a strong ordering of the Ga/Si atoms on the tetrahedral sites. Specifically, three types of tetrahedral site can be identified in the spinelloid structures: isolated tetrahedra (T_i), tetrahedral sites on the ends of T_2O_7 and T_3O_{10} groups (T_e), and sites in the middle of T_3O_{10} groups (T_m). Examination of the occupancies of these three types of sites shows that they are similar in all three phases: T_i with 92 to 95%Ga, T_e with 73 to 78% Ga, and T_m with 36 to 37% Ga. It is worth noting that such ordering could not be directly determined in the aluminosilicate spinelloids because of the similarity in electron density between Si and Al. However, a similar trend in their tetrahedral occupancies is revealed by a bond valence analysis.

This specific Ga/Si ordering provides some explanation for the observed stoichiometries of the gallosilicate phases. With only isolated tetrahedral sites and T_2O_7 groups, phase V lacks the Si-rich T_m sites and, as a result, its composition is the most spinel-like. Of all the spinelloid phases it is also, in structural terms, the closest relative to spinel (with a ratio of T:S boundaries of 1:2 as opposed to 2:2 for phase I and 2:1 for phase II, see Fig 1.3). Phase I can accommodate a larger amount of Si in its structure because of the presence of the T_m sites in its T_3O_{10} groups (see Fig 1.3a). Phase II, containing T_3O_{10} groups only (see Fig 1.3b), is richest in Ni_2SiO_4 because of the ability of both the T_m and T_e sites to incorporate large amounts of Si. On the other hand, since spinel contains only isolated tetrahedra (T_i) in its structure, the maximum solubility of Ni_2SiO_4 is predicted to be about 5 to 10%. Although the structure of the spinel phase was not refined, the presence of Si in the spinel phase is revealed by the green colour of the spinel crystals obtained, as opposed to the blue colour of pure $NiGa_2O_4$ (the Si displaces the tetrahedral Ni atoms present in $NiGa_2O_4$ spinel resulting in the colour change). There is also a direct relationship between the temperature at which each phase forms and the amount of silicon in its structure.

The amount of silicon that can be incorporated into the gallosilicate spinelloids is probably limited by the large mismatch between the Ga-O and Si-O tetrahedral bond lengths (1.85 and 1.64 Å respectively - Shannon 1976). The disordering of Ga and Si atoms on the same site would cause significant distortions of the lattice due to strain. However, because the longer (and weaker) Ga-O bond is expected to be more compressible than the Si-O bond, it is possible that under pressure, the gallosilicate spinelloids may be able to incorporate more silicon on the tetrahedral sites. A dependence of composition on pressure would explain the disparate stoichiometries of phase V in the gallosilicate and aluminosilicate systems. Unlike phases I and II, which occur at room pressure and have similar stoichiometries in both systems, the aluminosilicate phase V occurs only at very high pressure (above 7.0 GPa - Akaogi et al. 1982) and is much richer in Si than the room-pressure phase in the gallosilicate system (50% vs. 20% Ni_2SiO_4 respectively). By synthesizing the gallosilicate phase V at high pressure, it may be possible to obtain a composition closer to that observed in the aluminosilicate system because of the expected increase in the Si content. A similar argument could also explain the absence of phase V at room pressure in the aluminosilicate system. It is reasonable to assume that such a phase would be poorer in Si, similar in composition to the gallosilicate phase. However, Ni_2SiO_4 has a greater solubility range in $NiAl_2O_4$ (approx. 15 mol % at 1100°C, Akaogi et al. 1982) than in $NiGa_2O_4$, and the spinel solid solution, instead of phase V, may therefore be expected at room pressure. A similar situation occurs in the $MgGa_2O_4$-Mg_2GeO_4 system at 1 atm., where an extensive spinel solid solution includes the compositions of phase I and II (Barbier and Hyde, 1986).

Thermodynamically, the spinelloid phases are stable with respect to a mixture of spinel and olivine. Calorimetric studies of $NiAl_2O_4$-Ni_2SiO_4 and $MgGa_2O_4$-Mg_2GeO_4 spinelloids have found that these phases are very likely stabilized by configurational entropy (Akaogi and Navrotsky 1984, Leinenweber and Navrotsky 1989), indicating that the cation ordering/disordering may play an important role in determining phase formation in these systems. This role of entropy in stabilizing the spinelloid phases is also consistent with our observation that phase V forms at higher temperature than phase I. With fewer cation sites available in the structure of phase V, a higher temperature would be required to compensate for a smaller configurational entropy and maintain a stabilizing $-T\Delta S$ contribution to the free energy. Similarly, the spinel structure, with only one tetrahedral site, will be stabilized at even higher temperatures. Although phase II contains only two tetrahedral and four octahedral sites, the same number as phase V, its structure allows it to accommodate more Si. As a result, phase II has a more disordered structure, possibly allowing to have a greater configurational entropy and permitting its formation at relatively lower temperature. The limitations imposed on the stoichiometry of the spinelloid phases, together with entropy considerations, help explain the absence, in the gallosilicate system, of phase III, previously observed at room pressure in the $MgGa_2O_4$ - Mg_2GeO_4 (Barbier and Hyde 1986) and $MgFe_2O_4$ - Mg_2GeO_4 (Barbier 1989) systems, and of phase IV. If these phases maintained the same degree of occupancies on their tetrahedral sites as phases I, II, and V, they would then have compositions of 2.64 $NiGa_2O_4$: Ni_2SiO_4 for phase III and 3.35 $NiGa_2O_4$: Ni_2SiO_4 for phase IV. Thus the unobserved phases would have compositions lying between those of phases V (4:1) and phase I (2.27:1). As mentioned above, it is not unreasonable for phase V to form because of its structural similarity to spinel and its ability to gain stability from configurational entropy. As well, phase I contains a large number of cation sites and is able to gain a large amount of configurational entropy. On the other hand, phase III, forming at an intermediate composition, would contain only one type of tetrahedral site (see Fig 1.3c) and would be therefore be unable to gain any configurational entropy from Ga/Si disordering, which possibly explains its absence in the system. Phase IV, which contains isolated tetrahedra and T_2O_7 units, also has fewer types of tetrahedral sites than phase I, and hence it too would have less entropy gain from cation disordering.

In conclusion, the discovery of three room-pressure spinelloid phases (I,II, and V) in the $NiGa_2O_4$ - Ni_2SiO_4 system provides more experimental evidence to support the suggestion that such phases are only found in spinel-olivine systems containing an inverse spinel end-member. Of the five known spinelloid structures, four of them (I, II, III, and V), have now been observed *at room pressure* in a number of such systems. This fact and the insights garnered from the structure determinations of the nickel gallosilicate spinelloids clearly indicate that, rather than pressure alone, a combination of pressure-, composition-, and entropy-related effects determine the formation of spinelloid phases. The present study shows in particular that, under conditions of constant pressure, a close relationship exists between the chemical composition and the type of spinelloid structure formed.

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APPENDIX I

ANISOTROPIC TEMPERATURE FACTORS

The anisotropic temperature factors for each of the three nickel gallosilicate spinelloid phases are presented in the following tables.

Table I.1: Anisotropic displacement coefficients ($Å^2$ x10^4) for phase I.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
M1	51(6)	64(5)	54(5)	0	-1(5)	0
M2	58(5)	85(5)	54(5)	0	5(4)	0
M3	62(4)	66(4)	51(4)	0	-8(3)	0
M4	59(4)	59(3)	49(3)	0	0	22(5)
M5	64(4)	59(3)	58(3)	0	0	2(3)
T1	63(5)	64(5)	45(5)	0	0	0
T2	51(8)	59(7)	57(8)	0	0	0
T3	53(4)	58(4)	58(4)	0	0	0(3)
O1	82(24)	113(22)	100(24)	0	0	-82(24)
O2	58(22)	56(21)	62(18)	0	0	54(19)
O3	90(26)	198(30)	100(25)	0	0	-75(29)
O4	74(24)	182(28)	88(22)	0	0	-69(28)
O5	100(37)	83(28)	160(28)	0	-20(22)	0
O6	217(41)	103(28)	151(27)	0	26(23)	0
O7	31(16)	55(17)	28(14)	-21(13)	-7(11)	1(9)

Table I.2: Anisotropic displacement coefficients (Å^2 x10^4) for phase II.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
M1	16	46(3)	45(2)	0	3(2)	0
M2	19	54(2)	45(2)	0	6(2)	0
M3	21	49(2)	47(2)	0	0	0(2)
M4	16	51(3)	50(2)	0	0	5(2)
T1	15	50(2)	45(2)	0	0	0(2)
T2	15	46(4)	46(4)	0	0	0
O1	27	94(13)	84(11)	0	0	-4(10)
O2	50	152(17)	90(11)	0	0	31(11)
O3	50	176(17)	74(12)	0	0	9(11)
O4	84	72(13	71(10)	0	-13(12)	0
O5	66(12)	70(10)	82(8)	-5(9)	14(9)	0(6)

Table I.3: Anisotropic displacement coefficients (Å^2 x10^4) for phase V.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
M1	46(4)	49(3)	61(3)	0	0	-5(2)
M2	52(5)	75(5)	65(3)	0	-10(3)	0
M3	39(3)	49(3)	56(3)	0	8(2)	0
M4	52(6)	53(5)	44(3)	0	0	0
T1	53(4)	56(4)	66(3)	0	0	0
T2	57(3)	59(3)	60(3)	0	0	1(3)
O1	9(26)	126(25)	85(15)	0	6(11)	0
O2	87(23)	165(23)	120(18)	0	0	48(19)
O3	86(22)	90(22)	78(16)	0	0	-6(15)
O4	158(25)	58(15)	91(11)	-16(17)	-2(10)	-6(9)
O5	51(30)	116(29)	107(25)	0	0	0
O6	23(26)	88(26)	45(20)	0	0	0

APPENDIX II

OBSERVED AND CALCULATED STRUCTURE FACTORS

The following tables contain the observed (F_o) and calculated (F_c) structure factors for the three crystals. The tables also include the standard deviation for the observed values. A negative standard deviation indicates that the reflection was treated as unobserved during the refinement.

h	k	l	10Fo	10Fc	10s
2	0	0	1782	-1709	12
4	0	0	4970	5060	23
6	0	0	875	-926	10
8	0	0	2198	2302	19
10	0	0	453	-468	16
2	1	0	245	238	7
4	1	0	179	-189	13
6	1	0	137	66	-23
8	1	0	64	-53	-64
10	1	0	50	46	-50
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2	2	0	28	12	-28
4	2	0	86	-31	-28
6	2	0	37	6	-37
8	2	0	44	-29	-44
10	2	0	49	7	-49
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2	3	0	28	-6	-28
4	3	0	64	-9	-64
6	3	0	37	-1	-37
8	3	0	102	-7	-49
10	3	0	49	3	-49
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2	4	0	3609	3407	16
4	4	0	1112	1113	10
6	4	0	1434	1511	14
8	4	0	534	538	12
10	4	0	715	751	13
0	5	0	340	-333	6
2	5	0	347	334	7
4	5	0	197	-213	14
6	5	0	153	174	23
8	5	0	69	-78	-69
10	5	0	117	76	-62
0	6	0	76	-19	-26
2	6	0	31	-3	-31
4	6	0	36	-21	-36
6	6	0	39	-5	-39
8	6	0	45	-20	-45
10	6	0	50	-1	-50
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4	7	0	36	-7	-36
6	7	0	39	-12	-39
8	7	0	46	0	-46
10	7	0	52	-8	-52
0	8	0	4677	5044	23
2	8	0	1091	-1120	10
4	8	0	3438	3590	26
6	8	0	672	-706	10
8	8	0	1858	1922	16
10	8	0	434	-392	15
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2	9	0	381	382	9
4	9	0	256	-247	13
6	9	0	244	236	17

h	k	l	10Fo	10Fc	10s
8	9	0	135	-91	-40
10	9	0	169	96	-35
0	10	0	36	-9	-36
2	10	0	36	-18	-36
4	10	0	38	-12	-38
6	10	0	69	-17	-69
8	10	0	103	-12	-61
10	10	0	81	-10	-81
0	11	0	41	78	-41
2	11	0	234	-229	13
4	11	0	109	71	-33
6	11	0	147	-80	-30
8	11	0	49	56	-49
10	11	0	55	-53	-55
0	12	0	808	826	10
2	12	0	1526	1575	14
4	12	0	628	647	10
6	12	0	1006	1033	11
8	12	0	394	372	15
10	12	0	589	613	16
0	13	0	243	-268	17
2	13	0	352	350	12
4	13	0	243	-218	16
6	13	0	250	240	19
8	13	0	163	-112	-36
0	14	0	83	13	-55
2	14	0	40	-48	-40
4	14	0	43	10	-43
6	14	0	46	-41	-46
8	14	0	99	5	-99
0	15	0	62	42	-62
2	15	0	103	-64	-50
4	15	0	45	41	-45
6	15	0	165	-53	-28
8	15	0	146	37	-44
0	16	0	2116	2216	19
2	16	0	477	-487	12
4	16	0	1825	1864	16
6	16	0	372	-366	16
8	16	0	1220	1250	13
0	17	0	65	-131	-65
2	17	0	309	297	17
4	17	0	208	-178	23
6	17	0	234	216	24
8	17	0	157	-96	-44
0	18	0	48	21	-48
2	18	0	48	-61	-48
4	18	0	83	18	-83
6	18	0	52	-52	-52
0	19	0	133	104	-43
2	19	0	188	-119	-32
4	19	0	76	97	-76
6	19	0	148	-98	-43
0	20	0	366	357	16
2	20	0	821	825	12
4	20	0	289	307	23

h	k	l	10Fo	10Fc	10s
6	20	0	634	650	15
0	21	0	193	-172	31
2	21	0	250	255	27
4	21	0	181	-124	-35
0	22	0	76	37	-76
2	22	0	53	-80	-53
4	22	0	54	33	-54
0	23	0	173	54	-48
2	23	0	54	-63	-54
0	24	0	1072	1069	13
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2	0	1	1781	1511	13
3	0	1	584	558	8
4	0	1	288	270	12
5	0	1	467	-456	10
6	0	1	766	779	10
7	0	1	243	221	17
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11	0	1	158	85	-44
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3	1	1	98	48	-23
4	1	1	220	229	12
5	1	1	36	1	-36
6	1	1	778	805	9
7	1	1	104	56	-41
8	1	1	115	87	-44
9	1	1	101	4	-65
10	1	1	403	404	17
11	1	1	111	43	-79
0	2	1	321	-323	6
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3	2	1	78	58	-30
4	2	1	171	-172	15
5	2	1	36	34	-36
6	2	1	805	-839	9
7	2	1	41	82	-41
8	2	1	91	-52	-69
9	2	1	47	51	-47
10	2	1	390	-403	18
11	2	1	54	77	-54
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3	3	1	78	39	-30
4	3	1	260	248	11
5	3	1	36	11	-36
6	3	1	700	715	9
7	3	1	41	40	-41
8	3	1	99	98	-60
9	3	1	67	18	-67

h	k	l	10Fo	10Fc	10s
10	3	1	353	363	18
11	3	1	53	24	-53
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2	4	1	1166	1124	9
3	4	1	2559	-2484	18
4	4	1	410	415	8
5	4	1	1655	1737	15
6	4	1	596	618	9
7	4	1	1247	-1299	12
8	4	1	191	199	24
9	4	1	812	837	12
10	4	1	272	313	22
11	4	1	680	-691	15
0	5	1	638	599	6
1	5	1	258	-258	7
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3	5	1	264	287	10
4	5	1	404	411	9
5	5	1	93	-55	-38
6	5	1	586	623	10
7	5	1	148	99	-27
8	5	1	251	214	17
9	5	1	47	-28	-47
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4	6	1	93	-53	-35
5	6	1	36	51	-36
6	6	1	733	-767	9
7	6	1	43	63	-43
8	6	1	45	-34	-45
9	6	1	48	62	-48
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11	6	1	170	63	-37
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2	7	1	1252	1259	11
3	7	1	35	-20	-35
4	7	1	36	2	-36
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6	7	1	712	742	10
7	7	1	44	0	-44
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9	7	1	50	42	-50
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3	8	1	403	412	9
4	8	1	242	227	13
5	8	1	327	-369	13

h	k	l	10Fo	10Fc	10s
6	8	1	575	605	10
7	8	1	164	150	-28
8	8	1	104	98	-62
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10	8	1	369	329	17
11	8	1	173	85	-39
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7	9	1	109	99	-47
8	9	1	176	144	-30
9	9	1	50	-35	-50
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0	10	1	207	-217	15
1	10	1	35	-2	-35
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7	10	1	44	68	-44
8	10	1	115	-67	-51
9	10	1	51	49	-51
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2	11	1	785	802	9
3	11	1	73	-27	-73
4	11	1	79	30	-57
5	11	1	101	59	-43
6	11	1	516	529	12
7	11	1	116	-15	-45
8	11	1	49	29	-49
9	11	1	51	50	-51
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1	12	1	1464	1532	13
2	12	1	567	592	9
3	12	1	1335	-1390	13
4	12	1	289	279	14
5	12	1	1016	1081	11
6	12	1	376	398	14
7	12	1	862	-903	11
8	12	1	170	139	-32
9	12	1	625	623	14
10	12	1	223	238	32
0	13	1	395	403	11
1	13	1	243	-253	14
2	13	1	520	544	10
3	13	1	252	256	15
4	13	1	307	329	16
5	13	1	186	-160	22

h	k	l	10Fo	10Fc	10s
6	13	1	377	378	14
7	13	1	166	127	-31
8	13	1	252	217	23
9	13	1	129	-62	-55
0	14	1	41	-42	-41
1	14	1	39	46	-39
2	14	1	647	-670	10
3	14	1	67	-6	-67
4	14	1	44	-37	-44
5	14	1	45	73	-45
6	14	1	469	-467	13
7	14	1	98	23	-76
8	14	1	52	-24	-52
9	14	1	147	74	-46
0	15	1	125	-74	-32
1	15	1	128	77	-31
2	15	1	646	672	11
3	15	1	69	-57	-69
4	15	1	132	-66	-35
5	15	1	47	78	-47
6	15	1	479	490	14
7	15	1	84	-38	-84
8	15	1	135	-47	-46
9	15	1	55	59	-55
0	16	1	177	207	25
1	16	1	300	-306	15
2	16	1	436	459	13
3	16	1	253	258	19
4	16	1	148	133	-36
5	16	1	264	-266	20
6	16	1	338	340	17
7	16	1	161	139	-40
8	16	1	127	103	-60
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1	17	1	161	-99	-27
2	17	1	392	390	14
3	17	1	128	113	-41
4	17	1	259	255	20
5	17	1	168	-74	-30
6	17	1	297	298	21
7	17	1	211	129	27
8	17	1	200	185	32
0	18	1	128	-104	-43
1	18	1	46	18	-46
2	18	1	354	-366	16
3	18	1	47	14	-47
4	18	1	50	-93	-50
5	18	1	50	44	-50
6	18	1	286	-276	22
7	18	1	107	35	-84
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1	19	1	90	64	-90
2	19	1	434	407	15
3	19	1	48	-50	-48
4	19	1	112	-26	-53
5	19	1	94	66	-94

h	k	l	10Fo	10Fc	10s
6	19	1	324	320	21
7	19	1	114	-39	-75
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1	20	1	695	715	13
2	20	1	252	268	24
3	20	1	677	-690	13
4	20	1	148	158	-48
5	20	1	580	576	14
6	20	1	234	213	29
0	21	1	272	242	21
1	21	1	126	-124	-63
2	21	1	215	267	32
3	21	1	148	132	-45
4	21	1	197	177	-35
5	21	1	55	-98	-55
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1	22	1	129	67	-51
2	22	1	336	-306	21
3	22	1	90	-38	-90
4	22	1	54	-30	-54
0	23	1	112	-92	-77
1	23	1	53	72	-53
2	23	1	355	352	21
3	23	1	56	-61	-56
0	0	2	167	-84	22
1	0	2	747	-613	10
2	0	2	2181	1964	16
3	0	2	535	460	10
4	0	2	93	-8	-70
5	0	2	331	-333	16
6	0	2	1362	1348	14
7	0	2	276	236	18
8	0	2	50	20	-50
9	0	2	140	-103	-46
10	0	2	710	700	14
11	0	2	184	85	-37
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1	1	2	764	-699	8
2	1	2	78	3	-42
3	1	2	531	534	8
4	1	2	38	5	-38
5	1	2	366	-388	12
6	1	2	86	12	-58
7	1	2	278	268	17
8	1	2	76	3	-76
9	1	2	176	-123	-34
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3	2	2	858	-843	8
4	2	2	50	9	-50
5	2	2	606	617	9
6	2	2	97	16	-39
7	2	2	404	-431	13

h	k	l	10Fo	10Fc	10s
8	2	2	45	28	-45
9	2	2	289	302	20
10	2	2	121	30	-56
11	2	2	243	-217	29
0	3	2	123	-148	15
1	3	2	1039	-990	9
2	3	2	30	13	-30
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6	3	2	88	9	-46
7	3	2	421	437	13
8	3	2	45	-19	-45
9	3	2	306	-309	19
10	3	2	52	6	-52
11	3	2	233	221	30
0	4	2	1815	1825	14
1	4	2	774	-726	7
2	4	2	50	23	-50
3	4	2	627	625	7
4	4	2	1560	1583	14
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6	4	2	87	39	-52
7	4	2	350	346	14
8	4	2	841	871	11
9	4	2	256	-246	22
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3	5	2	520	521	7
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7	5	2	286	286	15
8	5	2	46	5	-46
9	5	2	203	-204	27
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11	5	2	173	115	-40
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2	6	2	84	-33	-29
3	6	2	689	-703	8
4	6	2	36	37	-36
5	6	2	518	532	9
6	6	2	40	-8	-40
7	6	2	372	-383	13
8	6	2	46	48	-46
9	6	2	264	275	21
10	6	2	50	12	-50
11	6	2	237	-201	28
0	7	2	141	-149	15
1	7	2	726	-719	8
2	7	2	69	-13	-47
3	7	2	597	612	8

h	k	l	10Fo	10Fc	10s
4	7	2	98	-54	-31
5	7	2	466	-474	10
6	7	2	41	-3	-41
7	7	2	328	342	15
8	7	2	46	-42	-46
9	7	2	211	-248	28
10	7	2	90	6	-90
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3	8	2	290	292	11
4	8	2	104	-23	-30
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8	9	2	87	54	-87
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1	10	2	549	564	8
2	10	2	36	30	-36
3	10	2	467	-495	10
4	10	2	38	-15	-38
5	10	2	392	394	12
6	10	2	43	40	-43
7	10	2	303	-297	18
8	10	2	48	6	-48
9	10	2	207	223	30
10	10	2	53	43	-53
0	11	2	132	-65	-23
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3	11	2	636	673	9
4	11	2	105	-56	-38
5	11	2	523	-546	11
6	11	2	62	-27	-62
7	11	2	418	416	14
8	11	2	125	-36	-46
9	11	2	283	-317	23
10	11	2	91	-19	-91
0	12	2	1237	1278	12
1	12	2	355	-364	11
2	12	2	38	-16	-38

h	k	l	10Fo	10Fc	10s
3	12	2	294	327	14
4	12	2	1010	1042	10
5	12	2	250	-268	18
6	12	2	99	4	-57
7	12	2	240	210	21
8	12	2	659	664	13
9	12	2	118	-121	-68
10	12	2	55	23	-55
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1	13	2	232	-203	15
2	13	2	141	112	-26
3	13	2	156	182	26
4	13	2	41	23	-41
5	13	2	152	-94	-31
6	13	2	87	94	-87
7	13	2	103	82	-75
8	13	2	118	27	-54
9	13	2	202	-91	28
0	14	2	81	52	-65
1	14	2	343	370	13
2	14	2	41	-40	-41
3	14	2	309	-334	16
4	14	2	44	55	-44
5	14	2	267	279	19
6	14	2	125	-21	-41
7	14	2	206	-222	27
8	14	2	52	55	-52
9	14	2	136	141	-60
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1	15	2	464	-463	12
2	15	2	75	-22	-75
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4	15	2	46	-77	-46
5	15	2	334	-355	16
6	15	2	47	-15	-47
7	15	2	294	283	20
8	15	2	157	-60	-39
9	15	2	263	-225	26
0	16	2	44	-65	-44
1	16	2	142	-54	-29
2	16	2	907	913	10
3	16	2	46	51	-46
4	16	2	110	-50	-49
5	16	2	48	-47	-48
6	16	2	686	692	13
7	16	2	143	41	-43
8	16	2	136	-21	-52
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2	17	2	45	40	-45
3	17	2	47	8	-47
4	17	2	48	106	-48
5	17	2	48	-6	-48
6	17	2	50	41	-50
7	17	2	52	5	-52
8	17	2	123	84	-64

h	k	l	10Fo	10Fc	10s
0	18	2	81	-34	-81
1	18	2	221	220	22
2	18	2	47	50	-47
3	18	2	209	-204	25
4	18	2	50	-25	-50
5	18	2	153	135	-41
6	18	2	52	51	-52
7	18	2	161	-120	-42
0	19	2	162	-63	-30
1	19	2	453	-443	14
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2	7	11	234	-218	39
3	7	11	334	267	26
4	7	11	726	661	19
5	7	11	312	-257	31
6	7	11	216	-113	-44
7	7	11	147	143	-92
8	7	11	489	466	26

h	k	l	10Fo	10Fc	10s
0	8	11	920	806	16
1	8	11	625	534	18
2	8	11	248	-211	33
3	8	11	500	-459	21
4	8	11	758	694	18
5	8	11	534	456	20
6	8	11	199	-120	-47
7	8	11	354	-300	28
8	8	11	564	490	22
0	9	11	709	620	16
1	9	11	377	-353	24
2	9	11	114	10	-114
3	9	11	347	311	25
4	9	11	578	540	20
5	9	11	329	-296	29
6	9	11	63	11	-63
7	9	11	235	172	-42
0	10	11	501	-454	19
1	10	11	56	23	-56
2	10	11	58	-22	-58
3	10	11	115	-55	-115
4	10	11	404	-395	24
5	10	11	62	-10	-62
6	10	11	64	-19	-64
7	10	11	124	-68	-124
0	11	11	285	302	32
1	11	11	101	89	-101
2	11	11	61	104	-61
3	11	11	117	-96	-117
4	11	11	268	264	32
5	11	11	63	70	-63
6	11	11	64	91	-64
7	11	11	66	-83	-66
0	12	11	181	147	-46
1	12	11	59	-14	-59
2	12	11	276	271	29
3	12	11	109	37	-109
4	12	11	225	186	37
5	12	11	61	11	-61
6	12	11	212	185	-45
0	13	11	59	76	-59
1	13	11	57	0	-57
2	13	11	447	423	20
3	13	11	59	-10	-59
4	13	11	145	71	-61
5	13	11	143	-9	-64
6	13	11	371	345	25
0	14	11	284	-314	30
1	14	11	58	3	-58
2	14	11	84	-53	-84
3	14	11	59	-32	-59
4	14	11	331	-279	25
5	14	11	99	-24	-99
0	15	11	568	544	17
1	15	11	249	-191	30
2	15	11	252	-220	30

h	k	l	10Fo	10Fc	10s
3	15	11	228	170	34
4	15	11	469	488	22
5	15	11	109	-139	-109
0	16	11	528	506	19
1	16	11	339	340	26
2	16	11	98	-74	-98
3	16	11	319	-295	25
4	16	11	454	456	22
0	17	11	397	391	23
1	17	11	308	-258	26
2	17	11	60	51	-60
3	17	11	278	235	30
0	18	11	268	-261	34
1	18	11	189	38	-39
0	0	12	576	651	15
1	0	12	51	-116	-51
2	0	12	837	944	12
3	0	12	112	122	-62
4	0	12	509	557	15
5	0	12	131	-90	-52
6	0	12	669	704	14
7	0	12	151	104	-50
8	0	12	384	382	21
0	1	12	170	-165	-34
1	1	12	253	-278	22
2	1	12	162	160	-35
3	1	12	243	267	25
4	1	12	126	-150	-61
5	1	12	200	-217	33
6	1	12	149	133	-50
7	1	12	223	201	30
8	1	12	123	-112	-81
0	2	12	131	-2	-41
1	2	12	49	-2	-49
2	2	12	132	-42	-45
3	2	12	50	-10	-50
4	2	12	52	-1	-52
5	2	12	111	-13	-79
6	2	12	55	-36	-55
7	2	12	93	-17	-93
8	2	12	58	3	-58
0	3	12	357	367	17
1	3	12	337	349	19
2	3	12	346	-371	19
3	3	12	295	-314	23
4	3	12	296	316	24
5	3	12	330	289	21
6	3	12	316	-279	24
7	3	12	178	-177	-48
8	3	12	227	222	37
0	4	12	52	9	-52
1	4	12	540	551	15
2	4	12	1485	1547	14
3	4	12	456	-496	18
4	4	12	79	2	-79
5	4	12	459	458	19

h	k	l	10Fo	10Fc	10s
6	4	12	1148	1167	14
7	4	12	375	-351	23
8	4	12	62	-13	-62
0	5	12	423	465	18
1	5	12	274	307	26
2	5	12	432	-433	18
3	5	12	270	-276	28
4	5	12	424	401	20
5	5	12	226	257	37
6	5	12	329	-330	26
7	5	12	236	-198	37
0	6	12	55	14	-55
1	6	12	134	-2	-56
2	6	12	81	-58	-81
3	6	12	108	-9	-108
4	6	12	58	12	-58
5	6	12	61	-13	-61
6	6	12	197	-48	-42
7	6	12	64	-17	-64
0	7	12	331	-311	24
1	7	12	133	-156	-78
2	7	12	267	274	32
3	7	12	168	155	-56
4	7	12	259	-269	36
5	7	12	64	-130	-64
6	7	12	244	168	-41
7	7	12	236	128	-40
0	8	12	641	576	18
1	8	12	159	-110	-64
2	8	12	873	796	17
3	8	12	169	115	-60
4	8	12	582	502	20
5	8	12	228	-85	-43
6	8	12	651	613	21
7	8	12	123	99	-123
0	9	12	138	-73	-93
1	9	12	283	-270	33
2	9	12	119	95	-119
3	9	12	253	261	41
4	9	12	67	-66	-67
5	9	12	255	-219	39
6	9	12	68	77	-68
7	9	12	229	168	-47
0	10	12	179	-41	-50
1	10	12	132	7	-90
2	10	12	64	-7	-64
3	10	12	64	-17	-64
4	10	12	66	-36	-66
5	10	12	108	-4	-108
6	10	12	68	-7	-68
0	11	12	207	163	-46
1	11	12	301	287	30
2	11	12	250	-224	37
3	11	12	331	-263	27
4	11	12	226	150	-44
5	11	12	257	247	39

h	k	l	10Fo	10Fc	10s
6	11	12	212	-146	-48
0	12	12	65	24	-65
1	12	12	423	406	23
2	12	12	1228	1144	15
3	12	12	403	-370	25
4	12	12	64	16	-64
5	12	12	365	353	28
0	13	12	466	419	21
1	13	12	215	160	-42
2	13	12	396	-362	24
3	13	12	233	-148	-40
4	13	12	378	372	27
5	13	12	275	178	33
0	14	12	63	-6	-63
1	14	12	105	6	-105
2	14	12	63	-42	-63
3	14	12	62	-15	-62
4	14	12	63	-5	-63
0	15	12	292	-269	31
1	15	12	199	-95	-43
2	15	12	219	191	-44
3	15	12	64	95	-64
0	16	12	473	432	22
1	16	12	155	-92	-64
2	16	12	541	525	21
0	0	13	51	6	-51
1	0	13	620	-653	14
2	0	13	508	537	15
3	0	13	573	594	14
4	0	13	51	6	-51
5	0	13	513	-541	17
6	0	13	392	421	20
7	0	13	389	421	22
0	1	13	165	134	-35
1	1	13	258	287	22
2	1	13	353	387	18
3	1	13	188	-192	-35
4	1	13	178	124	-35
5	1	13	231	246	30
6	1	13	319	309	22
7	1	13	199	-148	-35
0	2	13	501	-545	15
1	2	13	152	59	-38
2	2	13	71	-64	-71
3	2	13	83	-30	-83
4	2	13	425	-469	18
5	2	13	123	76	-62
6	2	13	146	-46	-50
7	2	13	128	-5	-73
0	3	13	563	632	14
1	3	13	274	-320	24
2	3	13	52	-50	-52
3	3	13	313	311	21
4	3	13	526	555	16
5	3	13	245	-251	28
6	3	13	107	-40	-107

h	k	l	10Fo	10Fc	10s
7	3	13	216	189	-37
0	4	13	441	476	17
1	4	13	173	-98	-34
2	4	13	53	50	-53
3	4	13	101	85	-101
4	4	13	388	420	20
5	4	13	91	-99	-91
6	4	13	142	42	-63
7	4	13	105	64	-105
0	5	13	530	596	16
1	5	13	260	-236	25
2	5	13	128	-8	-59
3	5	13	227	235	32
4	5	13	504	525	18
5	5	13	145	-141	-62
6	5	13	59	-5	-59
7	5	13	95	148	-95
0	6	13	499	-487	17
1	6	13	102	60	-102
2	6	13	159	-73	-43
3	6	13	135	-32	-59
4	6	13	446	-422	20
5	6	13	110	76	-110
6	6	13	105	-54	-105
0	7	13	141	128	-59
1	7	13	209	167	34
2	7	13	306	302	26
3	7	13	59	-118	-59
4	7	13	176	119	-47
5	7	13	102	123	-102
6	7	13	278	247	32
0	8	13	59	1	-59
1	8	13	587	-555	18
2	8	13	485	482	21
3	8	13	541	507	20
4	8	13	114	2	-114
5	8	13	507	-471	21
6	8	13	424	387	24
0	9	13	63	115	-63
1	9	13	339	329	28
2	9	13	396	373	25
3	9	13	337	-296	27
4	9	13	65	106	-65
5	9	13	329	287	29
6	9	13	335	305	31
0	10	13	500	-445	22
1	10	13	65	29	-65
2	10	13	65	-45	-65

h	k	l	10Fo	10Fc	10s
3	10	13	67	-5	-67
4	10	13	440	-392	26
5	10	13	66	48	-66
0	11	13	584	513	22
2	11	13	112	-82	-112
3	11	13	376	301	29
4	11	13	505	461	25
5	11	13	324	-255	34
0	12	13	425	371	27
1	12	13	70	-60	-70
2	12	13	68	50	-68
3	12	13	140	49	-95
4	12	13	326	335	35
0	13	13	516	443	21
1	13	13	143	-102	-101
2	13	13	157	21	-73
3	13	13	165	107	-74
4	13	13	434	401	27
0	14	13	385	-327	29
1	14	13	68	32	-68
2	14	13	165	-68	-66
3	14	13	66	-10	-66
0	15	13	103	101	-103
1	15	13	67	61	-67
0	0	14	325	361	21
1	0	14	249	276	26
2	0	14	311	338	20
3	0	14	167	-194	-43
4	0	14	302	328	23
5	0	14	162	181	-49
6	0	14	215	231	-37
0	1	14	241	284	26
1	1	14	300	301	21
2	1	14	272	-268	23
3	1	14	261	-284	24
4	1	14	216	251	32
5	1	14	230	250	31
6	1	14	141	-166	-62
0	2	14	125	-24	-51
1	2	14	248	-280	25
2	2	14	79	99	-79
3	2	14	278	264	23
4	2	14	98	-18	-98
5	2	14	230	-231	30
6	2	14	58	90	-58
0	3	14	111	-70	-72
1	3	14	54	44	-54
2	3	14	112	48	-77

h	k	l	10Fo	10Fc	10s
3	3	14	54	-44	-54
4	3	14	56	-64	-56
5	3	14	127	35	-72
6	3	14	57	44	-57
0	4	14	526	577	16
1	4	14	162	-15	-37
2	4	14	170	91	-38
3	4	14	117	15	-72
4	4	14	464	514	19
5	4	14	141	-15	-56
6	4	14	133	91	-66
0	5	14	109	-71	-93
1	5	14	120	-16	-65
2	5	14	56	105	-56
3	5	14	104	15	-104
4	5	14	87	-65	-87
5	5	14	58	-16	-58
6	5	14	169	93	-49
0	6	14	131	17	-60
1	6	14	199	-204	-40
2	6	14	57	57	-57
3	6	14	288	256	25
4	6	14	58	18	-58
5	6	14	240	-226	32
0	7	14	234	259	33
1	7	14	325	340	26
2	7	14	325	-305	24
3	7	14	304	-324	28
4	7	14	260	230	31
5	7	14	286	285	31
0	8	14	340	304	23
1	8	14	152	175	-60
2	8	14	309	307	26
3	8	14	270	-217	27
4	8	14	254	280	35
5	8	14	207	157	-41
0	9	14	230	257	37
1	9	14	159	158	-62
2	9	14	194	-134	-42
3	9	14	170	-152	-56
4	9	14	247	232	37
0	10	14	63	-45	-63
1	10	14	236	-201	37
2	10	14	162	112	-57
3	10	14	178	152	-56
4	10	14	64	-38	-64
0	11	14	90	-63	-90

h	k	l	10Fo	10Fc	10s
1	11	14	63	82	-63
2	11	14	66	5	-66
3	11	14	98	-81	-98
0	12	14	510	443	23
1	12	14	102	-31	-102
2	12	14	66	78	-66
3	12	14	112	30	-112
0	13	14	68	-64	-68
1	13	14	68	-62	-68
0	0	15	56	138	-56
1	0	15	53	-58	-53
2	0	15	240	254	27
3	0	15	54	81	-54
4	0	15	104	127	-104
5	0	15	57	-29	-57
0	1	15	169	214	-43
1	1	15	54	-89	-54
2	1	15	110	142	-110
3	1	15	105	77	-105
4	1	15	263	239	25
5	1	15	57	-85	-57
0	2	15	103	-10	-81
1	2	15	109	-25	-86
2	2	15	328	-364	22
3	2	15	54	-5	-54
4	2	15	158	-10	-41
5	2	15	56	-48	-56
0	3	15	98	-57	-98
1	3	15	272	326	27
2	3	15	422	490	19
3	3	15	276	-318	27
4	3	15	157	-52	-45
5	3	15	216	218	-36
0	4	15	122	-111	-65
1	4	15	204	-164	-34
2	4	15	520	547	17
3	4	15	230	232	32
4	4	15	133	-104	-64
5	4	15	147	-122	-57
0	5	15	133	6	-50
1	5	15	291	333	26
2	5	15	434	444	18
3	5	15	257	-324	32
4	5	15	58	7	-58
0	6	15	105	-10	-105
1	6	15	121	-32	-73
2	6	15	349	-339	22

h	k	l	10Fo	10Fc	10s
3	6	15	161	2	-46
4	6	15	59	-10	-59
0	7	15	93	119	-93
1	7	15	94	-105	-94
2	7	15	268	223	27
3	7	15	127	94	-77
4	7	15	62	111	-62
0	8	15	163	136	-54
1	8	15	57	-51	-57
2	8	15	257	216	31
3	8	15	61	72	-61
0	9	15	307	325	28
1	9	15	86	-61	-86
2	9	15	61	87	-61
3	9	15	120	52	-98
0	10	15	61	-20	-61
1	10	15	105	-21	-105
2	10	15	310	-284	27
0	11	15	64	-105	-64
0	0	16	845	913	14
1	0	16	300	-331	24
2	0	16	55	132	-55
3	0	16	333	329	23
0	1	16	350	-387	22
1	1	16	150	-142	-52
2	1	16	356	373	22
3	1	16	173	142	-44
0	2	16	118	-54	-70
1	2	16	56	3	-56
2	2	16	93	7	-93
3	2	16	56	-15	-56
0	3	16	226	254	32
1	3	16	56	108	-56
2	3	16	252	-259	28
3	3	16	173	-97	-40
0	4	16	656	729	16
1	4	16	56	5	-56
2	4	16	303	301	24
3	4	16	57	7	-57
0	5	16	149	161	-55
1	5	16	58	133	-58
2	5	16	58	-151	-58
0	6	16	104	-30	-104
1	6	16	56	-8	-56
2	6	16	101	-17	-101
0	7	16	290	-309	27
1	7	16	170	-154	-47

Observed and calculated structure factors for Nickel Gallosilicate Phase II

h	k	l	10Fo	10Fc	10s
2	0	0	2427	2515	16
4	0	0	6858	7788	33
6	0	0	1437	1474	14
8	0	0	3840	3983	32
10	0	0	917	860	22
0	2	0	434	403	6
2	2	0	417	411	9
4	2	0	297	294	16
6	2	0	295	259	20
8	2	0	210	156	-46
10	2	0	202	153	-82
0	4	0	89	7	-38
2	4	0	38	59	-38
4	4	0	44	-8	-44
6	4	0	53	45	-53
8	4	0	67	1	-67
10	4	0	84	9	-84
0	6	0	2388	-2381	18
2	6	0	4776	5247	25
4	6	0	1662	-1642	15
6	6	0	2427	2525	22
8	6	0	923	-889	17
10	6	0	1439	1485	21
0	8	0	508	-516	8
2	8	0	626	-627	9
4	8	0	309	-340	18
6	8	0	361	-370	21
8	8	0	201	-185	-55
10	8	0	244	-240	-72
0	10	0	42	21	-42
2	10	0	129	-144	-35
4	10	0	50	11	-50
6	10	0	196	-100	-35
8	10	0	71	1	-71
10	10	0	164	-56	-164
0	12	0	7434	7534	35
2	12	0	1569	1642	15
4	12	0	5057	5566	40
6	12	0	1052	1117	14
8	12	0	3137	3352	27
10	12	0	731	715	27
0	14	0	229	393	33
2	14	0	615	619	12
4	14	0	318	313	20
6	14	0	424	431	22
8	14	0	221	189	-58
10	14	0	221	284	-94
0	16	0	332	352	18
2	16	0	478	525	15
4	16	0	240	285	29
6	16	0	393	363	23
8	16	0	167	180	-94
10	16	0	204	225	-112
0	18	0	1196	-1181	13
2	18	0	2598	2504	21
4	18	0	964	-960	14
6	18	0	1727	1783	17
8	18	0	603	-619	26
10	18	0	1185	1250	23
0	20	0	354	-368	21
2	20	0	626	-620	15
4	20	0	290	-307	29
6	20	0	482	-473	23
8	20	0	326	-218	41
0	22	0	69	-210	-69
2	22	0	424	-381	18
4	22	0	246	-180	34
6	22	0	256	-290	-43
8	22	0	83	-129	-83
0	24	0	3567	3382	29
2	24	0	696	699	16
4	24	0	3059	2958	25
6	24	0	550	562	24
8	24	0	2225	2233	22
0	26	0	81	192	-81
2	26	0	475	447	20
4	26	0	207	166	-50
6	26	0	323	367	40
8	26	0	255	119	-61
0	28	0	357	342	28
2	28	0	472	484	23
4	28	0	313	304	36
6	28	0	404	397	34
0	30	0	524	-504	22
2	30	0	1414	1396	16
4	30	0	472	-450	27
6	30	0	1225	1195	19
0	32	0	170	-197	-77
2	32	0	455	-441	27
4	32	0	187	-181	-74
0	34	0	81	-228	-81
2	34	0	342	-351	36
0	36	0	1769	1695	17
1	0	1	1429	1326	11
3	0	1	824	876	9
5	0	1	746	778	12
7	0	1	377	401	24
9	0	1	459	455	30
11	0	1	225	246	-106
0	1	1	920	798	8
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5	15	4	58	29	-58
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3	23	4	185	227	-49
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5	18	5	188	219	-48
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9	18	5	313	163	50
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8	17	7	396	392	38
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3	18	7	1127	1143	13
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h	k	l	10Fo	10Fc	10s
3	22	7	235	256	39
5	22	7	276	270	37
7	22	7	157	187	-157
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2	23	7	612	-649	17
4	23	7	222	207	-45
6	23	7	494	-535	29
1	24	7	272	286	31
3	24	7	217	231	-44
5	24	7	251	289	-46
7	24	7	180	164	-98
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2	25	7	304	294	29
4	25	7	924	-922	17
6	25	7	247	250	-56
1	26	7	63	-88	-63
3	26	7	69	-107	-69
5	26	7	74	-55	-74
0	27	7	201	-114	-44
2	27	7	284	261	34
4	27	7	222	-105	-51
6	27	7	158	232	-158
1	28	7	64	-14	-64
3	28	7	68	4	-68
5	28	7	174	-26	-84
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2	29	7	1075	1074	16
4	29	7	417	423	31
1	30	7	631	628	20
3	30	7	637	638	21
0	31	7	209	191	-55
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2	0	8	588	586	11
4	0	8	3676	3827	29
6	0	8	449	440	20
8	0	8	2636	2555	25
10	0	8	330	299	-61
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3	1	8	1174	-1178	13
5	1	8	956	-957	12
7	1	8	843	-818	17
9	1	8	644	-642	27
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2	2	8	611	622	11
4	2	8	477	504	15
6	2	8	454	445	20
8	2	8	313	336	43
10	2	8	257	311	-87
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3	3	8	47	-31	-47
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7	3	8	105	-43	-105
9	3	8	83	31	-83
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h	k	l	10Fo	10Fc	10s
2	4	8	347	339	15
4	4	8	256	254	24
6	4	8	217	236	-40
8	4	8	193	170	-74
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5	5	8	551	549	17
7	5	8	436	428	26
9	5	8	431	399	39
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4	6	8	1643	-1574	16
6	6	8	1355	1298	15
8	6	8	1094	-1051	19
10	6	8	960	910	27
1	7	8	791	-782	11
3	7	8	687	-690	12
5	7	8	621	-612	16
7	7	8	501	-482	24
9	7	8	511	-446	33
0	8	8	87	68	-87
2	8	8	128	-50	-42
4	8	8	86	57	-86
6	8	8	64	-44	-64
8	8	8	252	41	-52
10	8	8	93	-43	-93
1	9	8	47	16	-47
3	9	8	52	44	-52
5	9	8	59	-11	-59
7	9	8	70	52	-70
9	9	8	85	-19	-85
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2	10	8	207	207	28
4	10	8	220	273	35
6	10	8	189	157	-52
8	10	8	304	194	46
1	11	8	1059	1056	12
3	11	8	1003	980	12
5	11	8	859	824	15
7	11	8	742	725	21
9	11	8	621	583	29
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2	12	8	418	417	17
4	12	8	3181	3113	28
6	12	8	278	321	39
8	12	8	2367	2230	22
1	13	8	984	-977	12
3	13	8	899	-914	13
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7	13	8	718	-693	21
9	13	8	584	-559	33
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2	14	8	713	707	14
4	14	8	488	478	19
6	14	8	569	549	22

h	k	l	10Fo	10Fc	10s
8	14	8	361	345	42
1	15	8	126	30	-56
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7	15	8	105	-15	-105
9	15	8	87	44	-87
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2	16	8	520	534	17
4	16	8	382	364	22
6	16	8	441	414	27
8	16	8	354	267	42
1	17	8	402	415	19
3	17	8	333	379	25
5	17	8	364	361	28
7	17	8	261	296	-52
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4	18	8	1079	-1073	15
6	18	8	1070	1056	17
8	18	8	779	-801	25
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3	19	8	572	-527	17
5	19	8	477	-498	25
7	19	8	426	-418	32
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6	20	8	170	-201	-82
8	20	8	84	-22	-84
1	21	8	140	6	-63
3	21	8	62	26	-62
5	21	8	106	-10	-106
7	21	8	226	38	-60
0	22	8	59	42	-59
2	22	8	174	-91	-50
4	22	8	160	40	-68
6	22	8	210	-72	-60
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3	23	8	590	625	19
5	23	8	518	558	25
7	23	8	516	522	30
0	24	8	2116	2150	19
2	24	8	137	134	-87
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6	24	8	109	118	-109
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3	25	8	539	-564	22
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2	26	8	513	539	22
4	26	8	367	307	28
1	27	8	159	40	-66
3	27	8	70	23	-70
5	27	8	173	49	-84
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2	28	8	466	485	25

h	k	l	10Fo	10Fc	10s
4	28	8	349	341	35
1	29	8	173	232	-67
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1	31	8	355	-376	33
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3	0	9	255	-241	23
5	0	9	141	-155	-59
7	0	9	220	-172	-47
9	0	9	221	-73	-78
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4	1	9	124	-128	-55
6	1	9	1453	-1417	16
8	1	9	76	-81	-76
1	2	9	262	284	18
3	2	9	204	229	27
5	2	9	211	242	-37
7	2	9	69	139	-69
9	2	9	237	174	-71
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4	3	9	53	74	-53
6	3	9	651	-635	18
8	3	9	75	49	-75
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3	4	9	301	340	20
5	4	9	221	249	35
7	4	9	243	262	-48
9	4	9	87	166	-87
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2	5	9	424	-450	15
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6	5	9	335	-337	28
8	5	9	787	744	22
1	6	9	1785	1762	15
3	6	9	1624	1641	17
5	6	9	1427	1368	15
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4	7	9	1190	-1194	14
6	7	9	345	301	27
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3	8	9	507	519	16
5	8	9	398	400	23
7	8	9	391	399	32
9	8	9	263	274	-69
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4	9	9	90	-54	-90
6	9	9	614	596	20
8	9	9	152	-37	-152

h	k	l	10Fo	10Fc	10s
1	10	9	472	445	15
3	10	9	416	389	18
5	10	9	394	372	23
7	10	9	267	268	-45
9	10	9	294	271	-58
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2	11	9	1685	1669	16
4	11	9	170	298	-56
6	11	9	1326	1295	16
8	11	9	248	212	-63
1	12	9	153	-146	-42
3	12	9	307	-156	23
5	12	9	105	-89	-105
7	12	9	143	-115	-143
9	12	9	88	-36	-88
0	13	9	90	107	-90
2	13	9	1462	-1468	14
4	13	9	59	96	-59
6	13	9	1187	-1162	17
8	13	9	145	80	-145
1	14	9	54	66	-54
3	14	9	169	33	-45
5	14	9	64	75	-64
7	14	9	75	2	-75
0	15	9	93	116	-93
2	15	9	608	-590	15
4	15	9	114	100	-114
6	15	9	442	-467	29
8	15	9	164	73	-164
1	16	9	55	45	-55
3	16	9	175	59	-45
5	16	9	66	27	-66
7	16	9	134	61	-134
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2	17	9	349	-327	23
4	17	9	648	654	18
6	17	9	339	-264	33
8	17	9	504	523	35
1	18	9	1177	1176	13
3	18	9	1181	1129	14
5	18	9	991	982	17
7	18	9	947	911	21
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4	19	9	1036	-1034	16
6	19	9	204	196	-64
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3	20	9	423	444	23
5	20	9	374	360	30
7	20	9	370	379	40
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4	21	9	68	-61	-68
6	21	9	406	403	32
1	22	9	440	425	21
3	22	9	338	391	31

h	k	l	10Fo	10Fc	10s
5	22	9	329	377	36
0	23	9	294	332	32
2	23	9	1062	1103	15
4	23	9	268	302	42
6	23	9	954	947	20
1	24	9	176	2	-54
3	24	9	181	-12	-57
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2	25	9	896	-922	16
4	25	9	144	201	-144
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3	26	9	69	-51	-69
5	26	9	166	-10	-89
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2	27	9	371	-306	27
4	27	9	72	106	-72
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3	28	9	128	-84	-128
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2	29	9	70	-182	-70
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4	0	10	152	-99	-46
6	0	10	1251	1211	15
8	0	10	111	-115	-111
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3	1	10	156	118	-41
5	1	10	95	85	-95
7	1	10	162	86	-80
9	1	10	177	42	-136
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2	2	10	192	189	29
4	2	10	154	198	-53
6	2	10	136	145	-92
8	2	10	269	150	-51
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3	3	10	491	481	16
5	3	10	482	433	19
7	3	10	348	353	34
9	3	10	341	316	49
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2	4	10	319	303	19
4	4	10	377	341	20
6	4	10	236	231	-43
8	4	10	181	244	-106
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7	5	10	863	806	19
9	5	10	725	668	29
0	6	10	876	895	12
2	6	10	706	-710	13
4	6	10	819	788	14
6	6	10	566	-572	22

h	k	l	10Fo	10Fc	10s
8	6	10	725	626	24
1	7	10	826	-874	12
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7	7	10	669	-616	22
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8	8	10	183	138	-96
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4	10	10	169	45	-48
6	10	10	177	171	-68
8	10	10	197	40	-86
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3	11	10	270	-239	27
5	11	10	130	-200	-130
7	11	10	77	-177	-77
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2	12	10	1312	1303	13
4	12	10	152	-83	-62
6	12	10	1099	1065	17
8	12	10	84	-104	-84
1	13	10	212	-117	31
3	13	10	60	-102	-60
5	13	10	189	-111	-56
7	13	10	192	-79	-77
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2	14	10	58	100	-58
4	14	10	272	292	35
6	14	10	73	82	-73
8	14	10	216	233	-86
1	15	10	333	335	24
3	15	10	298	309	30
5	15	10	349	290	30
7	15	10	204	243	-76
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2	16	10	236	199	34
4	16	10	338	381	30
6	16	10	145	162	-145
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3	17	10	955	974	15
5	17	10	874	877	18
7	17	10	787	785	24
0	18	10	687	678	16
2	18	10	492	-482	20
4	18	10	653	628	19
6	18	10	407	-424	32
1	19	10	449	-444	20

h	k	l	10Fo	10Fc	10s
3	19	10	439	-430	23
5	19	10	403	-377	27
7	19	10	349	-361	44
0	20	10	61	56	-61
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4	20	10	68	50	-68
6	20	10	312	272	41
1	21	10	251	-263	35
3	21	10	268	-246	36
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6	22	10	259	158	-49
1	23	10	225	-206	-43
3	23	10	102	-197	-102
5	23	10	74	-177	-74
0	24	10	64	-51	-64
2	24	10	875	877	17
4	24	10	74	-60	-74
1	25	10	304	-254	32
3	25	10	71	-238	-71
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2	26	10	67	21	-67
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1	27	10	69	129	-69
3	27	10	71	121	-71
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6	1	11	315	-299	34
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5	2	11	481	441	19
7	2	11	236	318	-59
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6	3	11	175	-31	-63
8	3	11	508	-457	31
1	4	11	234	212	26
3	4	11	230	228	31
5	4	11	64	152	-64
7	4	11	204	198	-66
0	5	11	684	-702	13
2	5	11	573	612	15
4	5	11	608	-614	17
6	5	11	531	508	23
8	5	11	446	-464	36

h	k	l	10Fo	10Fc	10s
1	6	11	102	-18	-102
3	6	11	116	38	-83
5	6	11	64	-64	-64
7	6	11	75	71	-75
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2	7	11	858	-879	13
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6	7	11	773	-725	18
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7	8	11	186	114	-84
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2	9	11	56	11	-56
4	9	11	535	514	19
6	9	11	70	3	-70
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1	10	11	356	358	21
3	10	11	292	310	29
5	10	11	329	326	32
7	10	11	252	229	-55
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2	11	11	513	541	18
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6	11	11	412	428	31
8	11	11	1250	1180	21
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5	12	11	625	587	20
7	12	11	423	400	34
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6	13	11	157	-85	-98
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3	14	11	414	435	24
5	14	11	449	442	26
7	14	11	311	336	48
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2	15	11	60	-14	-60
4	15	11	483	-446	22
6	15	11	76	-8	-76
1	16	11	284	282	29
3	16	11	304	291	31
5	16	11	181	226	-73
7	16	11	255	257	-61
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2	17	11	321	305	27
4	17	11	529	-529	22
6	17	11	283	278	46
1	18	11	61	18	-61
3	18	11	66	60	-66
5	18	11	130	-23	-130
0	19	11	234	288	39
2	19	11	886	-895	16

h	k	l	10Fo	10Fc	10s
4	19	11	191	265	-62
6	19	11	784	-783	22
1	20	11	63	28	-63
3	20	11	130	49	-130
5	20	11	72	8	-72
0	21	11	261	326	39
2	21	11	188	-51	-53
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1	22	11	238	187	39
3	22	11	71	162	-71
5	22	11	221	186	-56
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2	23	11	480	482	24
4	23	11	1053	1101	18
1	24	11	375	396	27
3	24	11	387	346	29
0	25	11	925	-964	17
2	25	11	138	75	-115
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0	27	11	221	-254	-51
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4	0	12	775	747	16
6	0	12	1383	-1301	16
8	0	12	590	582	31
1	1	12	482	-487	16
3	1	12	481	-477	18
5	1	12	379	-407	29
7	1	12	455	-402	30
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2	2	12	281	296	26
4	2	12	262	246	35
6	2	12	308	243	36
8	2	12	236	187	-68
1	3	12	178	32	-37
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5	3	12	66	57	-66
7	3	12	77	-20	-77
0	4	12	512	510	16
2	4	12	499	520	17
4	4	12	442	457	22
6	4	12	448	426	27
8	4	12	376	362	44
1	5	12	905	921	14
3	5	12	870	854	14
5	5	12	807	799	17
7	5	12	690	671	24
0	6	12	107	-136	-107
2	6	12	2178	2207	19
4	6	12	169	-125	-57
6	6	12	1823	1799	19
8	6	12	85	-100	-85
1	7	12	874	-890	13
3	7	12	829	-826	15
5	7	12	768	-776	19
7	7	12	654	-652	25

h	k	l	10Fo	10Fc	10s
0	8	12	701	711	15
2	8	12	610	610	16
4	8	12	657	640	18
6	8	12	503	503	27
1	9	12	114	-14	-114
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5	9	12	166	-39	-70
7	9	12	79	32	-79
0	10	12	449	475	20
2	10	12	379	395	23
4	10	12	415	427	25
6	10	12	387	328	31
1	11	12	311	353	28
3	11	12	332	351	28
5	11	12	297	301	37
7	11	12	345	307	43
0	12	12	739	762	16
2	12	12	1374	-1374	14
4	12	12	707	693	18
6	12	12	1188	-1143	18
1	13	12	464	-492	21
3	13	12	489	-485	22
5	13	12	429	-423	28
7	13	12	445	-418	36
0	14	12	62	45	-62
2	14	12	123	168	-123
4	14	12	170	39	-63
6	14	12	208	146	-67
1	15	12	62	24	-62
3	15	12	109	-2	-109
5	15	12	103	46	-103
0	16	12	101	195	-101
2	16	12	268	282	35
4	16	12	105	181	-105
6	16	12	275	245	-50
1	17	12	694	714	18
3	17	12	640	673	20
5	17	12	682	645	21
0	18	12	228	-185	-42
2	18	12	1667	1646	16
4	18	12	227	-171	-51
1	19	12	636	-650	19
3	19	12	633	-613	20
5	19	12	609	-593	24
0	20	12	652	659	18
2	20	12	516	491	22
4	20	12	597	614	23
1	21	12	143	12	-93
3	21	12	149	33	-91
0	22	12	499	501	23
2	22	12	403	391	28
4	22	12	512	467	25
1	23	12	151	214	-93
3	23	12	165	218	-85
0	24	12	592	615	22
2	24	12	911	-925	18

h	k	l	10Fo	10Fc	10s
1	25	12	387	-394	29
1	0	13	901	877	14
3	0	13	833	820	15
5	0	13	802	761	18
7	0	13	700	655	25
0	1	13	197	209	-37
2	1	13	928	-934	14
4	1	13	177	189	-57
6	1	13	818	-791	20
1	2	13	471	484	18
3	2	13	449	442	21
5	2	13	458	436	26
7	2	13	388	351	38
0	3	13	564	593	16
2	3	13	60	-66	-60
4	3	13	549	532	20
6	3	13	130	-53	-130
1	4	13	349	390	25
3	4	13	413	385	23
5	4	13	336	322	32
7	4	13	375	320	40
0	5	13	1165	1193	13
2	5	13	169	110	-49
4	5	13	1097	1085	15
6	5	13	74	91	-74
1	6	13	432	-465	21
3	6	13	426	-429	22
5	6	13	395	-404	29
7	6	13	296	-336	-52
0	7	13	1095	-1141	13
2	7	13	60	37	-60
4	7	13	1041	-1041	16
6	7	13	202	34	-64
1	8	13	260	241	31
3	8	13	259	247	37
5	8	13	203	192	-59
7	8	13	256	213	-60
0	9	13	509	-536	19
2	9	13	62	75	-62
4	9	13	504	-485	22
6	9	13	75	61	-75
1	10	13	265	282	33
3	10	13	220	257	-46
5	10	13	270	261	44
0	11	13	138	-188	-83
2	11	13	726	731	17
4	11	13	138	-172	-138
6	11	13	643	633	24
1	12	13	771	740	16
3	12	13	713	697	19
5	12	13	642	658	23
0	13	13	66	157	-66
2	13	13	917	-944	16
4	13	13	178	145	-70
6	13	13	844	-822	21
1	14	13	499	516	21

h	k	l	10Fo	10Fc	10s
3	14	13	456	476	25
5	14	13	495	476	26
0	15	13	447	467	23
2	15	13	67	-65	-67
4	15	13	429	429	29
1	16	13	450	439	23
3	16	13	470	430	24
5	16	13	361	382	37
0	17	13	968	969	16
2	17	13	236	193	-43
4	17	13	906	902	19
1	18	13	264	-310	39
3	18	13	294	-289	37
0	19	13	853	-874	17
2	19	13	261	144	38
4	19	13	830	-820	20
1	20	13	68	102	-68
3	20	13	71	115	-71
0	21	13	345	-358	32
2	21	13	70	83	-70
1	22	13	70	81	-70
0	23	13	174	-120	-66
0	0	14	757	-774	15
2	0	14	286	335	32
4	0	14	724	-719	19
6	0	14	343	316	39
1	1	14	601	-626	18
3	1	14	553	-590	22
5	1	14	538	-558	26
0	2	14	298	324	30
2	2	14	302	301	31
4	2	14	311	297	35
6	2	14	285	256	-48
1	3	14	371	-353	24
3	3	14	331	-345	30
5	3	14	261	-306	-48
0	4	14	271	237	31
2	4	14	194	175	-48
4	4	14	273	219	38
6	4	14	205	156	-70
1	5	14	130	-105	-92
3	5	14	67	-92	-67
5	5	14	72	-93	-72
0	6	14	844	875	15
2	6	14	211	-203	-44
4	6	14	822	815	18
6	6	14	298	-202	44
1	7	14	117	-24	-117
3	7	14	67	-34	-67
5	7	14	74	-17	-74
0	8	14	230	194	38
2	8	14	298	278	31
4	8	14	171	180	-75
6	8	14	267	246	-53
1	9	14	306	308	29
3	9	14	330	302	32

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
5	9	14	309	269	40	0	16	14	268	227	39	0	3	15	64	50	-64	2	9	15	408	-404	26	3	1	16	608	-555	22
0	10	14	303	288	30	2	16	14	68	64	-68	2	3	15	427	434	25	4	9	15	160	-38	-93	0	2	16	578	555	21
2	10	14	413	399	25	4	16	14	274	213	44	4	3	15	109	44	-109	1	10	15	314	259	32	2	2	16	587	568	22
4	10	14	353	266	31	1	17	14	138	-139	-122	1	4	15	400	407	26	3	10	15	200	232	-61	1	3	16	68	-22	-68
1	11	14	702	713	18	3	17	14	135	-130	-135	3	4	15	384	413	30	0	11	15	244	274	-42	3	3	16	165	-50	-87
3	11	14	688	680	19	0	18	14	656	691	20	5	4	15	387	349	33	2	11	15	443	-495	27	0	4	16	379	349	27
5	11	14	603	642	25	2	18	14	142	-185	-113	0	5	15	347	281	26	4	11	15	296	263	42	2	4	16	416	358	27
0	12	14	628	-679	19	1	19	14	72	-160	-72	2	5	15	1130	1146	15	1	12	15	210	-140	-49	1	5	16	225	171	-44
2	12	14	230	310	-46	0	20	14	69	124	-69	4	5	15	76	254	-76	3	12	15	220	-174	-55	3	5	16	176	153	-78
4	12	14	638	-640	21	1	0	15	194	-166	-50	1	6	15	180	151	-57	0	13	15	579	-624	22	0	6	16	1293	-1308	15
1	13	14	412	-401	25	3	0	15	276	-202	38	3	6	15	234	185	-46	2	13	15	209	329	-60	2	6	16	326	315	34
3	13	14	329	-376	35	5	0	15	240	-112	-51	5	6	15	76	96	-76	1	14	15	70	139	-70	1	7	16	221	-239	-52
5	13	14	424	-371	31	0	1	15	453	-484	23	0	7	15	67	-115	-67	0	15	15	165	10	-73	3	7	16	202	-218	-64
0	14	14	311	316	33	2	1	15	442	468	24	2	7	15	1040	-1079	16	2	15	15	242	329	-52	0	8	16	210	277	-57
2	14	14	103	161	-103	4	1	15	424	-456	29	4	7	15	257	-101	-45	1	16	15	247	301	-49	2	8	16	251	212	-44
4	14	14	297	295	42	1	2	15	173	201	-61	1	8	15	461	447	24	0	0	16	1236	1231	15	1	9	16	70	17	-70
1	15	14	252	-267	-43	3	2	15	200	174	-57	3	8	15	512	451	23	2	0	16	454	-432	25	0	10	16	385	422	31
3	15	14	285	-264	39	5	2	15	256	202	-49	0	9	15	66	-44	-66	1	1	16	570	-560	22	1	11	16	501	522	25

Observed and calculated structure factors for Nickel Gallosilicate Phase V

h	k	l	10Fo	10Fc	10s
2	0	0	1417	-1398	11
4	0	0	3940	3942	21
6	0	0	745	-756	8
8	0	0	1827	1859	16
10	0	0	409	-375	12
0	1	0	169	147	4
2	1	0	111	-111	11
4	1	0	53	113	-53
6	1	0	76	-49	-36
8	1	0	54	64	-54
10	1	0	72	-9	-72
2	2	0	23	33	-23
4	2	0	51	1	-51
6	2	0	32	33	-32
8	2	0	83	19	-45
10	2	0	41	18	-41
0	3	0	1345	1337	11
2	3	0	2798	2508	15
4	3	0	936	897	9
6	3	0	1164	1163	12
8	3	0	448	442	10
10	3	0	587	631	11
0	4	0	209	214	7
2	4	0	214	-197	7
4	4	0	156	157	13
6	4	0	74	-102	-45
8	4	0	39	96	-39
10	4	0	110	-53	-40
0	5	0	97	-82	15
2	5	0	84	71	-21
4	5	0	104	-47	-20
6	5	0	34	62	-34
8	5	0	82	-15	-49
10	5	0	43	48	-43
0	6	0	3723	3915	21
2	6	0	902	-929	8
4	6	0	2868	2823	23
6	6	0	570	-582	8
8	6	0	1536	1565	14
10	6	0	319	-318	16
0	7	0	189	245	13
2	7	0	248	-261	9
4	7	0	188	194	13
6	7	0	152	-157	19
8	7	0	143	114	-24
10	7	0	139	-76	-32
0	8	0	147	-146	15
2	8	0	114	119	19
4	8	0	57	-102	-57
6	8	0	77	85	-53
8	8	0	82	-39	-71
0	9	0	661	661	8
2	9	0	1186	1194	11
4	9	0	504	520	9
6	9	0	798	815	9
8	9	0	320	308	14
0	10	0	203	217	14
2	10	0	240	-256	12
4	10	0	167	181	18
6	10	0	179	-178	19
8	10	0	44	124	-44
0	11	0	41	-125	-41
2	11	0	116	89	-24
4	11	0	71	-98	-71
6	11	0	76	75	-76
8	11	0	94	-54	-59
0	12	0	1768	1770	15
2	12	0	415	-415	10
4	12	0	1455	1506	13
6	12	0	301	-312	14
0	13	0	165	184	-30
2	13	0	235	-238	15
4	13	0	162	157	24
6	13	0	166	-176	26
0	14	0	41	-117	-41
2	14	0	103	69	-37
4	14	0	140	-94	-28
6	14	0	44	57	-44
0	15	0	302	289	14
2	15	0	667	659	10
4	15	0	260	251	18
0	16	0	163	157	27
2	16	0	202	-221	23
0	0	1	261	-195	4
1	0	1	671	607	6
2	0	1	860	-762	7
3	0	1	409	-355	5
4	0	1	152	-131	12
5	0	1	363	364	8
6	0	1	396	-404	8
7	0	1	158	-154	17
8	0	1	38	-80	-38
9	0	1	215	207	17
10	0	1	194	-212	21
11	0	1	73	-82	-73
0	1	1	374	337	5
1	1	1	94	-47	12
2	1	1	1959	1607	12
3	1	1	46	36	-46
4	1	1	210	201	9
5	1	1	30	-17	-30
6	1	1	850	853	9
7	1	1	34	21	-34
8	1	1	38	105	-38
9	1	1	39	2	-39
10	1	1	431	435	12
11	1	1	45	25	-45
0	2	1	432	389	5
1	2	1	24	3	-24
2	2	1	1674	1451	12
3	2	1	25	-2	-25
4	2	1	248	235	8
5	2	1	43	-5	-43
6	2	1	798	798	8
7	2	1	75	10	-45
8	2	1	94	119	-41
9	2	1	103	4	-35
10	2	1	383	414	14
11	2	1	101	20	-54
0	3	1	348	-310	5
1	3	1	2422	-2290	13
2	3	1	612	-561	6
3	3	1	2043	1976	16
4	3	1	217	-213	10
5	3	1	1334	-1353	13
6	3	1	329	-320	9
7	3	1	1047	1058	10
8	3	1	107	-120	-33
9	3	1	674	-674	10
10	3	1	188	-179	21
0	4	1	478	425	5
1	4	1	185	-176	7
2	4	1	1321	1218	10
3	4	1	154	156	12
4	4	1	305	302	8
5	4	1	67	-110	-44
6	4	1	667	693	8
7	4	1	109	95	-27
8	4	1	168	165	19
9	4	1	56	-49	-56
10	4	1	389	375	12
0	5	1	66	16	-23
1	5	1	68	82	-25
2	5	1	1457	1385	11
3	5	1	71	-82	-32
4	5	1	30	14	-30
5	5	1	55	56	-55
6	5	1	802	813	9
7	5	1	37	-43	-37
8	5	1	39	20	-39
9	5	1	40	37	-40
10	5	1	428	442	13
0	6	1	180	-162	10
1	6	1	401	396	6
2	6	1	519	-504	7
3	6	1	309	-280	8
4	6	1	66	-124	-53
5	6	1	274	295	10
6	6	1	308	-314	11
7	6	1	124	-145	-26
8	6	1	83	-87	-51
9	6	1	195	194	20
10	6	1	159	-183	-28
0	7	1	331	344	8
1	7	1	148	-138	13
2	7	1	873	880	8
3	7	1	122	126	18
4	7	1	253	261	10
5	7	1	129	-87	19
6	7	1	545	563	9
7	7	1	38	81	-38
8	7	1	114	162	-37
9	7	1	42	-40	-42
10	7	1	344	329	14
0	8	1	93	112	-24
1	8	1	108	87	-18
2	8	1	864	889	9
3	8	1	92	-85	-25
4	8	1	80	86	-34
5	8	1	104	55	-26
6	8	1	564	589	9
7	8	1	39	-48	-39
8	8	1	41	62	-41
9	8	1	43	35	-43
0	9	1	200	-196	12
1	9	1	1174	-1208	11
2	9	1	289	-297	9
3	9	1	1076	1115	10
4	9	1	124	-162	-23
5	9	1	841	-858	10
6	9	1	189	-210	18
7	9	1	735	741	9
8	9	1	105	-115	-43
9	9	1	513	-513	12
0	10	1	277	291	11
1	10	1	175	-197	14
2	10	1	550	579	8
3	10	1	183	183	15
4	10	1	210	241	15
5	10	1	167	-142	19
6	10	1	391	414	11
7	10	1	152	129	25
8	10	1	125	164	-38
0	11	1	36	-73	-36
1	11	1	56	84	-56
2	11	1	730	754	8
3	11	1	37	-86	-37
4	11	1	98	-56	-33
5	11	1	95	58	-37
6	11	1	547	548	10
7	11	1	41	-57	-41
8	11	1	43	-24	-43
0	12	1	142	-132	22
1	12	1	216	234	15
2	12	1	203	-232	17
3	12	1	178	-192	19
4	12	1	87	-119	-48
5	12	1	196	213	19
6	12	1	165	-179	23
7	12	1	81	-131	-81
0	13	1	250	244	14
1	13	1	90	-127	-45
2	13	1	386	380	11
3	13	1	129	123	-28
4	13	1	232	215	17
5	13	1	148	-96	24
6	13	1	293	297	15
7	13	1	45	94	-45
0	14	1	129	31	-27
1	14	1	131	69	-27
2	14	1	443	451	11
3	14	1	87	-74	-57
4	14	1	80	31	-80
5	14	1	43	49	-43
6	14	1	347	357	15
0	15	1	191	-144	20
1	15	1	589	-580	11
2	15	1	154	-140	-26
3	15	1	560	566	11
4	15	1	147	-132	-29
5	15	1	487	-474	12
0	16	1	193	192	23
1	16	1	190	-148	21
2	16	1	269	271	17
3	16	1	189	145	22
0	0	2	72	-51	-17
1	0	2	327	-315	5
2	0	2	1486	1482	10
3	0	2	243	236	7
4	0	2	56	-14	-56
5	0	2	171	-167	12
6	0	2	1030	1051	10
7	0	2	118	118	-24
8	0	2	37	19	-37
9	0	2	116	-79	-31
10	0	2	583	566	11
11	0	2	102	58	-51
0	1	2	24	-13	-24
1	1	2	885	817	7
2	1	2	50	-1	-30
3	1	2	654	-651	6
4	1	2	29	-39	-29
5	1	2	464	470	7
6	1	2	32	-10	-32
7	1	2	315	-336	11
8	1	2	37	-54	-37
9	1	2	216	232	18
10	1	2	42	-21	-42
11	1	2	167	-173	28
0	2	2	68	80	-19
1	2	2	1188	1076	9
2	2	2	24	-40	-24
3	2	2	924	-892	8
4	2	2	30	47	-30
5	2	2	659	658	7
6	2	2	32	-62	-32
7	2	2	465	-471	9
8	2	2	37	9	-37
9	2	2	324	326	13
10	2	2	79	-66	-79

h	k	l	10Fo	10Fc	10s
0	3	2	1368	1416	10
1	3	2	385	-374	5
3	3	2	326	322	7
4	3	2	1283	1247	11
5	3	2	257	-242	9
6	3	2	61	33	-61
7	3	2	176	174	17
8	3	2	698	712	9
9	3	2	116	-118	-33
10	3	2	79	43	-79
0	4	2	24	55	-24
1	4	2	730	727	7
2	4	2	109	-105	14
3	4	2	618	-620	7
4	4	2	30	33	-30
5	4	2	463	466	7
6	4	2	72	-89	-43
7	4	2	336	-342	11
8	4	2	38	4	-38
9	4	2	258	240	15
10	4	2	43	-79	-43
0	5	2	77	68	-21
1	5	2	759	784	7
2	5	2	76	8	-25
3	5	2	660	-672	7
4	5	2	65	20	-42
5	5	2	495	512	8
6	5	2	34	-10	-34
7	5	2	364	-379	11
8	5	2	120	-23	-27
9	5	2	269	271	15
10	5	2	43	-22	-43
0	6	2	46	-61	-46
1	6	2	146	-162	13
2	6	2	1309	1289	11
3	6	2	133	140	15
4	6	2	31	-29	-31
5	6	2	84	-107	-32
6	6	2	839	867	9
7	6	2	103	80	-32
8	6	2	39	10	-39
9	6	2	95	-55	-51
10	6	2	509	513	12
0	7	2	112	-85	18
1	7	2	324	325	8
2	7	2	30	14	-30
3	7	2	269	-287	9
4	7	2	88	-80	-28
5	7	2	216	223	13
6	7	2	36	-1	-36
7	7	2	200	-174	16
8	7	2	41	-74	-41
9	7	2	104	126	-45
10	7	2	45	-14	-45
0	8	2	30	71	-30
1	8	2	738	751	8
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3	8	2	642	-666	8
4	8	2	68	46	-48
5	8	2	538	534	8
6	8	2	37	-26	-37
7	8	2	409	-412	11
8	8	2	41	11	-41
9	8	2	327	308	15
0	9	2	984	1014	10
1	9	2	176	-172	14
2	9	2	32	-25	-32
3	9	2	170	154	14
4	9	2	802	839	9
5	9	2	122	-124	-25
6	9	2	38	4	-38
7	9	2	141	96	-26
8	9	2	528	553	11
9	9	2	101	-70	-51
0	10	2	33	26	-33
1	10	2	268	265	11
2	10	2	34	-96	-34
3	10	2	217	-241	14
4	10	2	36	16	-36
5	10	2	190	197	18
6	10	2	68	-86	-68
7	10	2	168	-162	22
8	10	2	42	0	-42
0	11	2	73	56	-48
1	11	2	471	464	9
2	11	2	36	11	-36
3	11	2	414	-423	10
4	11	2	38	34	-38
5	11	2	348	355	12
6	11	2	40	-5	-40
7	11	2	282	-289	16
8	11	2	43	-2	-43
0	12	2	86	-57	-43
1	12	2	113	-30	-25
2	12	2	701	725	9
3	12	2	38	27	-38
4	12	2	87	-38	-47
5	12	2	40	-21	-40
6	12	2	540	564	11
7	12	2	43	18	-43
0	13	2	38	-85	-38
1	13	2	92	71	-44
2	13	2	106	12	-31
3	13	2	86	-67	-51
4	13	2	109	-82	-35
5	13	2	65	56	-65
6	13	2	43	2	-43
0	14	2	41	44	-41
1	14	2	411	408	12
2	14	2	40	15	-40
3	14	2	365	-381	13
4	14	2	79	31	-79
5	14	2	344	333	14
6	14	2	77	-5	-77
0	15	2	553	543	11
1	15	2	62	-41	-62
2	15	2	42	-40	-42
3	15	2	43	39	-43
4	15	2	488	494	12
0	16	2	112	8	-44
1	16	2	44	75	-44
2	16	2	93	-78	-61
3	16	2	103	-72	-51
0	0	3	1058	-967	7
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2	0	3	42	22	-42
3	0	3	1733	1734	13
4	0	3	710	-669	7
5	0	3	1367	-1408	13
6	0	3	32	7	-32
7	0	3	894	887	9
8	0	3	349	-355	11
9	0	3	730	-741	9
10	0	3	43	-15	-43
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1	1	3	174	-170	7
2	1	3	90	70	-15
3	1	3	126	144	13
4	1	3	1288	1218	11
5	1	3	111	-117	-19
6	1	3	32	37	-32
7	1	3	60	74	-60
8	1	3	633	631	9
9	1	3	78	-74	-78
10	1	3	94	30	-50
0	2	3	1423	1363	10
1	2	3	51	27	-31
2	2	3	361	326	5
3	2	3	27	-22	-27
4	2	3	967	965	10
5	2	3	30	20	-30
6	2	3	177	185	15
7	2	3	35	-22	-35
8	2	3	491	502	9
9	2	3	65	2	-65
10	2	3	166	114	23
0	3	3	560	-516	5
1	3	3	418	370	5
2	3	3	325	-296	6
3	3	3	412	-393	6
4	3	3	390	-375	7
5	3	3	176	182	13
6	3	3	171	-177	15
7	3	3	254	-244	12
8	3	3	170	-207	21
9	3	3	82	84	-74
10	3	3	68	-119	-68
0	4	3	1045	1010	9
1	4	3	72	-99	-23
2	4	3	518	504	6
3	4	3	77	76	-28
4	4	3	733	744	8
5	4	3	47	-66	-47
6	4	3	296	295	10
7	4	3	37	31	-37
8	4	3	391	406	11
9	4	3	120	-43	-31
10	4	3	201	176	20
0	5	3	1448	1408	11
1	5	3	50	-17	-50
2	5	3	104	-49	-19
3	5	3	30	23	-30
4	5	3	1073	1058	10
5	5	3	75	-14	-35
6	5	3	34	-32	-34
7	5	3	36	13	-36
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10	5	3	43	-6	-43
0	6	3	671	-652	7
1	6	3	1476	-1544	13
2	6	3	80	-4	-27
3	6	3	1262	1282	11
4	6	3	497	-501	7
5	6	3	1054	-1097	11
6	6	3	58	-12	-58
7	6	3	723	733	9
8	6	3	282	-300	14
9	6	3	648	-642	10
10	6	3	119	-25	-38
0	7	3	1024	1027	10
1	7	3	215	-233	10
2	7	3	156	140	13
3	7	3	182	200	13
4	7	3	774	799	8
5	7	3	175	-173	15
6	7	3	101	96	-32
7	7	3	140	117	23
8	7	3	474	482	11
9	7	3	125	-112	-32
0	8	3	859	896	9
1	8	3	62	86	-54
2	8	3	114	67	19
3	8	3	78	-69	-34
4	8	3	689	712	8
5	8	3	35	66	-35
6	8	3	109	55	-28
7	8	3	40	-45	-40
8	8	3	438	436	11
9	8	3	61	33	-61
0	9	3	271	-258	10
1	9	3	246	242	11
2	9	3	215	-215	12
3	9	3	241	-259	12
4	9	3	225	-213	13
5	9	3	159	152	19
6	9	3	152	-162	23
7	9	3	182	-204	20
8	9	3	163	-142	25
9	9	3	45	88	-45
0	10	3	428	436	9
1	10	3	142	-123	18
2	10	3	379	388	9
3	10	3	58	105	-58
4	10	3	350	362	11
5	10	3	117	-98	-27
6	10	3	294	285	13
7	10	3	117	62	-34
8	10	3	247	242	17
0	11	3	743	747	8
1	11	3	72	35	-59
2	11	3	37	-76	-37
3	11	3	84	-23	-42
4	11	3	612	630	9
5	11	3	59	30	-59
6	11	3	103	-48	-39
7	11	3	68	-12	-68
8	11	3	446	431	12
0	12	3	327	-314	11
1	12	3	754	-770	9
2	12	3	72	-46	-72
3	12	3	664	681	9
4	12	3	264	-273	14
5	12	3	624	-641	10
6	12	3	111	-44	-34
7	12	3	467	474	12
0	13	3	486	472	10
1	13	3	191	-184	18
2	13	3	122	132	-30
3	13	3	148	165	-25
4	13	3	404	414	12
5	13	3	143	-155	-28
6	13	3	108	106	-43
0	14	3	461	474	11
1	14	3	40	61	-40
2	14	3	41	-5	-41
3	14	3	41	-48	-41
4	14	3	428	417	12
5	14	3	91	56	-63
0	15	3	114	-107	-38
1	15	3	166	168	25
2	15	3	154	-169	-28
3	15	3	113	-182	-45
4	15	3	136	-98	-31
0	16	3	170	176	27
1	16	3	44	-98	-44
2	16	3	301	271	16
0	0	4	2324	2401	14
1	0	4	232	-223	6
2	0	4	1167	1158	9

h	k	l	10Fo	10Fc	10s
3	0	4	166	174	11
4	0	4	1513	1491	13
5	0	4	115	-119	19
6	0	4	637	650	8
7	0	4	36	90	-36
8	0	4	741	763	9
9	0	4	40	-66	-40
10	0	4	374	330	12
0	1	4	42	10	-42
1	1	4	327	306	5
2	1	4	26	2	-26
3	1	4	273	-255	8
4	1	4	69	14	-34
5	1	4	206	171	11
6	1	4	33	16	-33
7	1	4	122	-140	-25
8	1	4	38	12	-38
9	1	4	89	90	-54
10	1	4	67	26	-67
0	2	4	121	-101	11
1	2	4	351	-333	5
2	2	4	110	99	14
3	2	4	274	262	8
4	2	4	72	-55	-33
5	2	4	206	-208	11
6	2	4	60	67	-60
7	2	4	110	136	-29
8	2	4	65	-14	-65
9	2	4	138	-118	-26
10	2	4	42	44	-42
0	3	4	989	-1024	8
1	3	4	386	373	6
2	3	4	3828	3846	21
3	3	4	313	-305	8
4	3	4	766	-774	8
5	3	4	244	231	10
6	3	4	2176	2185	17
7	3	4	151	-167	20
8	3	4	415	-423	11
9	3	4	123	128	-32
10	3	4	1188	1199	11
0	4	4	282	-279	7
1	4	4	290	-284	7
2	4	4	250	249	8
3	4	4	227	229	10
4	4	4	201	-201	11
5	4	4	181	-187	13
6	4	4	160	158	17
7	4	4	128	126	-24
8	4	4	130	-99	-27
9	4	4	152	-111	24
10	4	4	44	91	-44
0	5	4	135	163	15
1	5	4	187	199	11
2	5	4	179	-152	11
3	5	4	176	-178	12

h	k	l	10Fo	10Fc	10s
4	5	4	153	132	14
5	5	4	132	126	18
6	5	4	71	-76	-63
7	5	4	54	-111	-54
8	5	4	71	80	-71
9	5	4	123	72	-30
10	5	4	81	-22	-81
0	6	4	1479	1516	13
1	6	4	146	-143	14
2	6	4	831	830	9
3	6	4	94	124	-25
4	6	4	1113	1122	11
5	6	4	89	-95	-31
6	6	4	508	518	9
7	6	4	68	78	-68
8	6	4	665	674	10
9	6	4	42	-60	-42
0	7	4	164	-177	13
1	7	4	208	211	11
2	7	4	134	135	16
3	7	4	195	-191	12
4	7	4	156	-136	15
5	7	4	157	145	17
6	7	4	103	97	-32
7	7	4	141	-126	23
8	7	4	41	-79	-41
9	7	4	82	87	-82
0	8	4	77	36	-32
1	8	4	185	-192	12
2	8	4	57	-49	-57
3	8	4	134	165	19
4	8	4	51	38	-51
5	8	4	200	-147	15
6	8	4	61	-23	-61
7	8	4	96	105	-43
8	8	4	62	36	-62
9	8	4	44	-97	-44
0	9	4	534	-550	8
1	9	4	214	199	12
2	9	4	2061	2136	17
3	9	4	198	-178	14
4	9	4	437	-453	9
5	9	4	147	152	22
6	9	4	1480	1500	13
7	9	4	137	-121	-26
8	9	4	300	-291	15
0	10	4	290	-289	11
1	10	4	118	-143	-24
2	10	4	227	227	13
3	10	4	61	126	-61
4	10	4	219	-236	15
5	10	4	106	-116	-35
6	10	4	171	163	21
7	10	4	101	87	-42
8	10	4	211	-148	19
0	11	4	121	128	-26

h	k	l	10Fo	10Fc	10s
1	11	4	103	91	-31
2	11	4	134	-151	22
3	11	4	112	-89	-29
4	11	4	79	111	-57
5	11	4	39	70	-39
6	11	4	70	-97	-70
7	11	4	43	-70	-43
0	12	4	783	797	9
1	12	4	106	-78	-33
2	12	4	423	412	10
3	12	4	39	74	-39
4	12	4	691	694	9
5	12	4	40	-64	-40
6	12	4	298	303	15
7	12	4	86	58	-86
0	13	4	194	-204	19
1	13	4	137	122	-25
2	13	4	163	138	21
3	13	4	146	-118	-25
4	13	4	188	-177	21
5	13	4	98	99	-50
6	13	4	136	109	-32
0	14	4	42	34	-42
1	14	4	121	-101	-32
2	14	4	103	-74	-41
3	14	4	43	92	-43
4	14	4	117	34	-35
5	14	4	107	-89	-44
0	15	4	240	-228	18
1	15	4	73	102	-73
2	15	4	1140	1105	11
3	15	4	45	-96	-45
4	15	4	232	-205	19
0	16	4	221	-226	21
1	16	4	44	-74	-44
2	16	4	203	154	23
0	0	5	249	-248	7
1	0	5	1794	1903	13
2	0	5	497	-456	6
3	0	5	1514	-1562	13
4	0	5	208	-189	10
5	0	5	1231	1264	11
6	0	5	288	-275	10
7	0	5	845	-862	9
8	0	5	109	-113	-33
9	0	5	699	696	10
10	0	5	127	-166	-36
0	1	5	996	992	8
1	1	5	124	135	13
2	1	5	560	520	6
3	1	5	132	-116	14
4	1	5	742	730	8
5	1	5	100	92	-24
6	1	5	299	306	11
7	1	5	36	-61	-36
8	1	5	375	405	12

h	k	l	10Fo	10Fc	10s
9	1	5	172	60	19
10	1	5	201	182	21
0	2	5	1516	1561	13
1	2	5	151	-158	11
2	2	5	41	-61	-41
3	2	5	113	133	19
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7	2	5	68	80	-68
8	2	5	611	636	10
9	2	5	40	-47	-40
10	2	5	43	-3	-43
0	3	5	692	-674	7
1	3	5	275	-252	7
2	3	5	30	-14	-30
3	3	5	270	257	9
4	3	5	514	-505	7
5	3	5	120	-135	20
6	3	5	34	-19	-34
7	3	5	143	171	23
8	3	5	296	-290	13
9	3	5	90	-78	-55
10	3	5	44	-27	-44
0	4	5	1278	1269	11
1	4	5	29	-20	-29
2	4	5	98	87	-20
3	4	5	30	21	-30
4	4	5	970	963	10
5	4	5	33	-10	-33
6	4	5	83	58	-39
7	4	5	37	19	-37
8	4	5	550	556	10
9	4	5	41	5	-41
10	4	5	44	47	-44
0	5	5	810	840	9
1	5	5	30	-21	-30
2	5	5	340	344	8
3	5	5	30	11	-30
4	5	5	640	652	8
5	5	5	33	-15	-33
6	5	5	210	221	15
7	5	5	37	7	-37
8	5	5	387	386	12
9	5	5	42	0	-42
10	5	5	151	143	-29
0	6	5	172	-178	12
1	6	5	1336	1380	11
2	6	5	366	-358	8
3	6	5	1157	-1182	12
4	6	5	125	-144	20
5	6	5	992	1003	9
6	6	5	211	-243	17
7	6	5	715	-719	9
8	6	5	139	-95	-25
9	6	5	607	606	11

h	k	l	10Fo	10Fc	10s
0	7	5	596	608	7
1	7	5	212	220	11
2	7	5	415	423	8
3	7	5	207	-191	11
4	7	5	478	487	8
5	7	5	142	165	22
6	7	5	288	292	13
7	7	5	83	-115	-55
8	7	5	322	308	14
9	7	5	104	107	-48
0	8	5	1006	1036	10
1	8	5	152	-170	16
2	8	5	133	-134	18
3	8	5	125	144	-22
4	8	5	824	841	9
5	8	5	102	-129	-32
6	8	5	101	-84	-35
7	8	5	41	92	-41
8	8	5	541	539	11
9	8	5	132	-71	-31
0	9	5	363	-382	9
1	9	5	186	-180	14
2	9	5	34	-48	-34
3	9	5	191	190	15
4	9	5	317	-319	11
5	9	5	111	-125	-30
6	9	5	40	-44	-40
7	9	5	184	155	20
8	9	5	220	-217	20
0	10	5	654	646	8
1	10	5	76	69	-47
2	10	5	141	130	20
3	10	5	37	-58	-37
4	10	5	533	545	9
5	10	5	63	58	-63
6	10	5	119	102	-31
7	10	5	59	-34	-59
8	10	5	381	377	14
0	11	5	472	473	10
1	11	5	38	-64	-38
2	11	5	177	160	18
3	11	5	108	49	-30
4	11	5	389	404	11
5	11	5	94	-54	-42
6	11	5	117	129	-35
7	11	5	43	32	-43
0	12	5	121	-86	-26
1	12	5	712	722	9
2	12	5	231	-239	15
3	12	5	660	-652	9
4	12	5	91	-78	-45
5	12	5	622	603	10
6	12	5	167	-193	26
0	13	5	257	287	17
1	13	5	212	187	16
2	13	5	297	281	14

Observed and calculated structure factors for Nickel Gallosilicate Phase V

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
3	13	5	183	-170	20	9	3	6	42	34	-42	5	9	6	39	9	-39	5	1	7	67	-50	-67	1	7	7	71	16	-39
4	13	5	264	254	16	10	3	6	45	36	-45	6	9	6	39	6	-39	6	1	7	414	430	10	2	7	7	372	376	9
5	13	5	139	159	-31	0	4	6	30	53	-30	7	9	6	81	-1	-81	7	1	7	37	29	-37	3	7	7	34	-18	-34
6	13	5	233	228	20	1	4	6	281	-290	8	8	9	6	494	492	12	8	1	7	175	210	23	4	7	7	365	373	10
0	14	5	577	570	11	2	4	6	75	-89	-29	0	10	6	37	50	-37	9	1	7	43	-42	-43	5	7	7	38	4	-38
1	14	5	80	-109	-80	3	4	6	231	245	10	1	10	6	55	-66	-55	0	2	7	130	-152	15	6	7	7	284	269	13
2	14	5	160	-105	23	4	4	6	59	35	-59	2	10	6	94	-109	-34	1	2	7	203	207	10	7	7	7	40	-15	-40
3	14	5	131	93	-31	5	4	6	178	-208	15	3	10	6	38	56	-38	2	2	7	1190	1165	11	8	7	7	253	254	18
4	14	5	508	507	12	6	4	6	37	-81	-37	4	10	6	39	37	-39	3	2	7	175	-182	13	0	8	7	177	-165	15
5	14	5	187	-96	21	7	4	6	160	144	20	5	10	6	40	-57	-40	4	2	7	152	-117	15	1	8	7	125	95	-21
0	15	5	193	-194	22	8	4	6	129	7	-25	6	10	6	80	-97	-80	5	2	7	138	147	19	2	8	7	820	832	9
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2	3	11	130	-157	-26
3	3	11	55	-46	-55
4	3	11	190	-165	17
5	3	11	39	-28	-39
6	3	11	101	-132	-45
7	3	11	43	-66	-43
0	4	11	323	319	11
1	4	11	37	90	-37
2	4	11	286	299	13
3	4	11	57	-87	-57
4	4	11	283	274	14
5	4	11	60	71	-60
6	4	11	254	233	17
7	4	11	44	-72	-44
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1	5	11	192	-202	17
2	5	11	169	-131	18
3	5	11	151	190	23
4	5	11	617	609	10
5	5	11	148	-166	-28
6	5	11	98	-93	-45
7	5	11	45	140	-45
0	6	11	420	-420	11
1	6	11	508	-516	10
2	6	11	160	97	19
3	6	11	437	442	11
4	6	11	389	-367	11
5	6	11	461	-450	11
6	6	11	68	62	-68

h	k	l	10Fo	10Fc	10s
0	7	11	544	566	10
1	7	11	245	-261	15
2	7	11	67	-9	-67
3	7	11	249	240	15
4	7	11	521	496	11
5	7	11	222	-220	18
6	7	11	44	-3	-44
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2	8	11	113	88	-32
3	8	11	41	-123	-41
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5	8	11	131	114	-31
0	9	11	119	-121	-36
1	9	11	83	22	-66
2	9	11	137	-152	-29
3	9	11	41	-53	-41
4	9	11	148	-110	-28
5	9	11	43	-6	-43
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1	10	11	42	39	-42
2	10	11	308	302	15
3	10	11	43	-41	-43
4	10	11	163	143	27
0	11	11	519	502	12
1	11	11	132	-126	-32
2	11	11	145	-117	-29
3	11	11	132	125	-35
0	12	11	283	-272	17
1	12	11	346	-342	15
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1	0	12	60	-86	-60
2	0	12	649	641	9
3	0	12	115	85	-29
4	0	12	475	466	11
5	0	12	79	-72	-79
6	0	12	497	485	11
7	0	12	44	71	-44
0	1	12	152	198	24
1	1	12	184	188	18
2	1	12	156	-198	23
3	1	12	197	-187	17
4	1	12	156	174	22
5	1	12	146	146	-26
6	1	12	110	-147	-42
7	1	12	177	-153	24
0	2	12	229	-242	15
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2	2	12	210	228	17
3	2	12	203	205	17
4	2	12	212	-207	17
5	2	12	191	-202	21
6	2	12	174	185	24
7	2	12	45	146	-45
0	3	12	188	-180	17
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h	k	l	10Fo	10Fc	10s
2	3	12	1279	1287	12
3	3	12	263	-270	14
4	3	12	158	-153	23
5	3	12	256	243	16
6	3	12	994	992	11
0	4	12	316	-307	12
1	4	12	203	-213	17
2	4	12	288	281	13
3	4	12	174	189	21
4	4	12	243	-265	17
5	4	12	202	-188	20
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0	5	12	217	244	16
1	5	12	118	135	-30
2	5	12	254	-244	14
3	5	12	110	-138	-37
4	5	12	216	217	18
5	5	12	42	105	-42
6	5	12	167	-184	26
0	6	12	483	476	11
1	6	12	40	-81	-40
2	6	12	552	544	10
3	6	12	41	80	-41
4	6	12	421	423	12
5	6	12	74	-69	-74
6	6	12	446	425	13
0	7	12	160	81	20
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2	7	12	114	-104	-33
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4	7	12	42	75	-42
5	7	12	157	154	-29
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2	8	12	118	133	-37
3	8	12	174	168	23
4	8	12	113	-134	-42
0	9	12	152	-104	25
1	9	12	226	218	19
2	9	12	987	966	11
3	9	12	213	-204	20
4	9	12	99	-92	-56
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2	10	12	274	252	18
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1	0	13	558	559	10
2	0	13	243	-262	16
3	0	13	491	-513	11
4	0	13	41	-10	-41
5	0	13	480	467	12
6	0	13	211	-214	21
0	1	13	294	292	13
1	1	13	185	185	19
2	1	13	281	281	14
3	1	13	180	-174	20

h	k	l	10Fo	10Fc	10s
4	1	13	252	257	16
5	1	13	160	155	26
6	1	13	255	230	16
0	2	13	600	619	10
1	2	13	214	-244	18
2	2	13	118	-49	-30
3	2	13	226	226	17
4	2	13	542	547	11
5	2	13	225	-201	18
6	2	13	44	-33	-44
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2	3	13	40	-25	-40
3	3	13	40	-42	-40
4	3	13	210	-222	19
5	3	13	110	47	-37
6	3	13	45	-23	-45
0	4	13	553	560	10
1	4	13	159	-178	23
2	4	13	40	-6	-40
3	4	13	141	167	-30
4	4	13	498	498	11
5	4	13	151	-146	-28
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2	5	13	240	234	16
3	5	13	43	-107	-43
4	5	13	259	248	16
5	5	13	156	92	-27
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2	6	13	277	-241	14
3	6	13	460	-442	12
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1	7	13	219	221	19
2	7	13	265	265	17
3	7	13	205	-207	21
4	7	13	197	202	23
0	8	13	529	521	12
1	8	13	241	-232	18
2	8	13	103	-69	-46
3	8	13	224	215	20
0	9	13	184	-195	24
1	9	13	108	22	-44
2	9	13	146	-36	-28
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1	0	14	117	126	-34
2	0	14	314	316	14
3	0	14	144	-114	-27
4	0	14	191	183	22
5	0	14	171	106	24
0	1	14	190	-214	21
1	1	14	321	-305	14
2	1	14	176	194	23
3	1	14	279	279	16

h	k	l	10Fo	10Fc	10s
4	1	14	203	-196	21
5	1	14	284	-261	17
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1	2	14	166	-151	23
2	2	14	113	-82	-34
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1	3	14	41	34	-41
2	3	14	125	75	-31
3	3	14	42	-28	-42
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2	4	14	43	-121	-43
3	4	14	124	78	-33
4	4	14	71	84	-71
0	5	14	171	-198	25
1	5	14	339	-334	14
2	5	14	210	200	21
3	5	14	318	308	16
0	6	14	187	169	22
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2	6	14	295	290	16
3	6	14	149	-92	-28
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2	7	14	167	147	26
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2	0	15	158	-168	-28
3	0	15	110	-62	-40
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2	1	15	260	257	16
3	1	15	44	118	-44
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2	2	15	417	433	13
3	2	15	221	-213	20
0	3	15	43	56	-43
1	3	15	274	267	17
2	3	15	272	-283	17
0	4	15	101	-4	-47
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2	4	15	386	392	14
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