

### CHARACTERISTIC ENERGY LOSSES OF CHanneLED ELECTRONS; RECIPROCIty AND DOUBLE ALIGNMENT

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When electron channeling is used in crystal studies, most experiments have so far been based on observing the element characteristic x-ray emission with an energy dispersive x-ray spectrometer (EDX). However, electron energy loss spectroscopy, EELS, offers several advantages. In addition to the possibility of observing details associated with the electronic structure [1], EELS under channeling conditions differs from EDX in two significant ways [2]: Firstly, it is possible to take advantage of the reciprocity principle which tells us that the crystallographic direction of the outgoing electrons to be energy analyzed is just as important as the crystallographic direction of the incident beam. Thus, there is a dramatic increase in the orientation dependence in EELS compared to EDX, because with this double alignment we benefit from the blocking effect in addition to the channeling effect. Secondly, we can in EELS improve the localization by increasing the scattering angle. In particular, under planar channeling conditions, we have the freedom of selecting equivalent crystallographic directions for the incident electrons and the electrons to be energy analyzed, while at the same time varying the scattering angle, and thus, the localization.

#### References

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**Keywords:** CHANNELING, RECIPROCIty, ELECTRONS

### ELECTRON CHANNELING X-RAY SPECTROSCOPIC STUDY OF PARTIALLY DISORDERED MAGNESIUM-ALUMINATE SPINEL

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Ion configuration in magnesium aluminate spinel has been examined by measurements of characteristic x-ray emission as a function of incident electron beam direction at high angular resolution, a technique that we termed HARECXs. In the experiment, TEM disk specimens of  $(\text{MgO})(\text{Al}_2\text{O}_3)_n$  with compositions  $n = 1.0, 2.4$  and  $3.0$  were prepared after annealing at 1470 K for 2 days. HARECXs profiles of Mg-K, Al-K and O-K x-ray intensities were obtained at 120 kV with incident beam rocking between  $-4g$  and  $+4g$  ( $g = 400$ ) orientations. The theoretical simulations on the dynamical scattering formulation derived by Rossouw et al. clearly demonstrated that the HARECXs profiles depend sensitively on the ion configuration in the crystal lattice as well as the mean-square displacements of ions and specimen thickness. The experimental results were therefore analyzed to determine the ion configurations through detailed comparison with the simulations as a function of the above parameters. It was confirmed that the stoichiometric compound with  $n = 1.0$  has a tendency to form the normal structure, where Mg and Al ions occupy preferentially the tetrahedral (IV) and the octahedral (VI) sites, respectively. As is well known, however, the cation ordering takes place only partially in the annealed compound, so that 60 % of Mg ions are located in the IV sites while the remaining are on the VI sites. The tendency to form the normal structure disappears in the non-stoichiometric compounds with  $n = 2.4$  and  $3.0$ , where the occupation probability of Mg on the IV sites is about 1/3 or less. It has been indicated that the structural vacancies in the non-stoichiometric case are mostly located on the IV sites. Thus HARECXs is useful for quantitative determination of atom configuration in local areas of crystalline materials.

**Keywords:** MGAL04 SPINEL DISORDERING ALCHEMI

### THE ORDERING STATE OF $\text{Ti}_2\text{AlNb}$

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$\text{Ti}_2\text{AlNb}$  is an intermetallic compound with a wide composition range of existence. It has an orthorhombic structure with three independent sublattices: Wyckoff 8g, 4c1 and 4c2. In the classical or ideal scheme each of these contains one only of the three elements. The real ordering state of  $\text{Ti}_2\text{AlNb}$  has been determined here using a combination of ALCHEMI and neutron diffraction. We have chosen to use systematic, or planar, ALCHEMI to minimize delocalization effects. There are two types of superlattice systematic row in  $\text{Ti}_2\text{AlNb}$ . In one type of row, 8g alternates with  $4(c1+c2)$ . In the other,  $4(g+c1)$  alternates with  $4(g+c2)$ . Using both types of row for ALCHEMI enables the shape of the ordering tie triangle to be determined. (The ordering tie triangle is the compositions of the three sublattices plotted out on a ternary cross-section.) An iterated Bloch wave calculation enables the absolute tie triangle to be determined. High resolution time-of-flight powder neutron diffraction data were collected at HRPD at the ISIS Facility (UK) and analyzed using the Rietveld full pattern method with atomic positions and site occupancies treated as variable parameters. An alloy of composition Ti-23Al-21Nb-2Mo-0.35Si was solution treated at 1140°C for 2 hours and quenched, then annealed at 915°C for 72 h and quenched again. The ALCHEMI and neutron diffraction results are shown in the figure below.

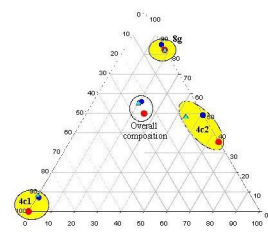


Figure 4-26: Ternary plot of sublattice occupancies determined by neutron diffraction showing good agreement with the ALCHEMI results from a variety of different compositions.

**Keywords:** ALCHEMI  $\text{Ti}_2\text{AlNb}$  SUBLATTICE OCCUPANCY

### PROSPECTS FOR ALCHEMI USING ELECTRON ENERGY LOSS SPECTROSCOPY IN THE TRANSMISSION ELECTRON MICROSCOPE

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Electron energy loss spectroscopy (EELS) in the Transmission Electron Microscope (TEM) is a microanalytical technique that analyses the energy spectrum of the electrons transmitted through a thin solid sample. It can provide information on the concentration and distribution of light and medium atomic number elements at high spatial resolution and, in addition, can often facilitate the identification of local site symmetries (i.e. co-ordinations), and valence states of atomic species via analysis of the electron energy loss near-edge structure (ELNES) associated with core level ionization edges [1]. In recent years, detailed modeling of ELNES has been performed via calculation of the site- and symmetry-projected unoccupied density of states (DOS) using *ab-initio* electronic structure calculations.

Channeling-based X-ray emission experiments in the TEM are a relatively well established technique for the site location of atomic species within the crystalline unit cell. Some of the first experiments using EELS-ALCHEMI were published by Tafto and Krivanek [2] and Tafto [3] who elegantly demonstrated the ability to isolate both the vacancies and local co-ordinations of species on various crystal sites within minerals such as spinels and silicates. However, since that time virtually no similar studies have been published, despite the fact that there have been significant improvements in both EELS detection systems and general understanding of localized inner shell ionization processes over the intervening period.

We shall review the experimental procedures for EELS ALCHEMI and highlight the possibilities in some important application areas including the site location of dopant species in ferroelectric materials based on the perovskite structure.

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**Keywords:** ALCHEMI EELS ELECTRONIC STRUCTURE