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THE COMBINED EFFECT OF ACCELERATION VOLTAGE AND INCIDENT BEAM ORIENTATION ON THE CHARACTERISTIC X-RAY PRODUCTION IN THIN CRYSTALS

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ABSTRACT

A systematic experimental study has been carried out to determine the combined effect of acceleration voltage and incident beam orientation on the characteristic x-ray production in thin crystals. For $MgAl_2O_4$ it has been shown that the orientation dependence undergoes a reversal in character above a particular voltage which is referred to now as the "inversion" voltage. This "inversion" voltage has been experimentally determined to be ~270kV for $MgAl_2O_4$ compounds with a spinel structure and is in agreement with theoretical predictions based on a highly localized scattering model for characteristic x-ray production in thin crystals. Further, in combination with theoretical calculations, this "inversion" voltage behavior has been shown to be different from the conventional critical voltage effect. From the microanalysis point of view, it has been experimentally shown that in order to obtain an analysis independent of the incident beam orientation or the acceleration voltage it is essential to systematically tilt the crystal to an orientation at which no lower order Bragg reflections are excited.

INTRODUCTION

One of the principal products of the interaction of a fast electron (an electron accelerated through a potential of 100kV or more) with a crystalline solid in the form of a thin film is characteristic x-ray emission. These x-ray photons with energies characteristic of the elements constituting the crystalline solid and peak intensities proportional to their concentrations in the material can then be detected by standard energy dispersive x-ray spectrometers to give a chemical microanalysis of the sample (Goldstein, 1979). However, the precise interpretation of these x-ray intensities in terms of their concentrations in the solid (chemical microanalysis) in an analytical transmission electron microscope is limited by a number of instrument and specimen related factors (Zaluzec,

1979; Goldstein and Williams, 1978) which include among others the acceleration voltage and incident beam orientation (particularly for single crystals).

In general for crystalline materials, an incident plane wave of electrons under conditions of strong dynamical scattering sets up a standing wave within the crystal. The intensity modulations of this standing wave within the crystal unit cell are a function of the incident beam orientation and the acceleration voltage. As the scattering events (like inner shell excitations) that lead to characteristic x-ray production are highly localized, the x-ray intensities in turn, are strongly determined by the orientation and the acceleration voltage. For a given acceleration voltage or wavelength of the incident wave, it has been shown that this orientation dependence of the characteristic x-ray emission can also be used as a probe for determining specific site occupations of elemental additions in single crystals (Spence and Tafto, 1983; Krishnan, Rabenberg, Mishra and Thomas, 1984; Krishnan and Thomas, 1984; Krishnan, Rez and Thomas, 1985). The implications of this orientation dependence of the x-ray productions on energy dispersive x-ray microanalysis in a transmission electron microscope have also been briefly discussed (Cherns et al., 1973). However, an interesting voltage dependence of this orientation dependence has also been suggested earlier (Krishnan, Rez and Thomas, 1983). In this paper we present the result of a systematic experimental investigation of the combined effect of the orientation and the acceleration voltage on characteristic x-ray production in thin crystals and discuss its ramifications on conventional energy dispersive x-ray microanalysis.

THEORY

We outline here the derivation of an expression for characteristic x-ray production in thin crystals in the conventional dynamical-theory formulation of electron diffraction (Hirsch et al., 1965).

It has been shown (Heidenreich, 1962) that the incorporation of an imaginary crystal potential $iP(\bar{r})$ in the Schrödinger equation

$$\nabla^2\phi + \frac{2m}{\hbar^2}(E + V + iP)\,\phi = 0 \qquad (1)$$

leads to a rate of energy loss per unit volume at the point $\bar{r}$ proportional to $P(\bar{r})|\phi(\bar{r})|^2$. The rate of "absorption" of electrons in a volume V is then given by

$$\frac{m}{hk}\int \bar{\nabla}.\bar{s}\, d^3r = \frac{2m}{\hbar^2}\int P(\bar{r})\,|\phi(\bar{r})|^2\, d^3r \qquad (2)$$

and could approximate the characteristic x-ray production rate if $P(\bar{r})$ is chosen appropriately.

The scattering processes that lead to characteristic x-ray production in thin crystals are highly localized (Krishnan et al., 1985; Bourdillon et al., 1981; Howie, 1979) and hence we assume that this imaginary part of the crystal potential is a delta function at the mean atomic positions. Under this assumption, the rate of characteristic x-ray production given by equation (2) for any element 'z' and crystal thickness 't' reduces to

$$N_z = \sum_{RCS} \int_0^t \phi^*\phi\, dz \qquad (3)$$

and the summation is over the relevant crystallographic sites (RCS) where the element z is distributed in the unit cell.

For an incident plane wave of electrons, the scattered wave amplitudes ϕ within the crystal can be expressed as a linear combination of Bloch waves (Hirsch et al., 1965),

$$\phi(\bar{r}) = \sum_j \psi^j \sum_h C_h^j \exp[\,i(\bar{k}^j + \bar{h}).\bar{r}\,] \qquad (4)$$

where ψ^j are the excitation amplitudes of the jth Bloch wave, C_k^j are the Bloch wave coefficients and $\bar{k}^j$ are components of the wavector for the electrons.

For centrosymmetric crystals, $\psi^j = C_0^j$. Neglecting absorption, one can then derive an expression for characteristic x-ray productions per unit thickness from equations (1), (2), (3), and (4) as

$$N_z = \sum_{RCS} \sum_{g,h} \exp[i(\bar{h}-\bar{g}).\bar{r}] \sum_{j=\ell} C_0^{j*} C_g^{j*} C_0^{\ell} C_h^{\ell} + \sum_{j\neq\ell} C_0^{j*} C_g^{j*} C_0^{\ell} C_h^{\ell} \frac{\sin[(k^j - k^{\ell})t]}{[(k^j - k^{\ell})t]} . \qquad (5)$$

This expression for characteristic x-ray production is composed of two parts: a thickness-independent term of individual Bloch-wave contributions and a thickness-dependent term of Bloch-wave interference contributions. Further, it has been shown (Krishnan, Rez and Thomas, 1985) that the contributions from the thickness dependent term is small compared to the thickness independent term.

A more detailed elaboration of this derivation is published elsewhere (Krishnan et al., 1985). The inclusion of absorption in a similar treatment is given by Cherns et al., (1973) and a more complete description of ionizing events in crystals including (e,2e) scattering kinematics has also been published (Maslen and Rossouw, 1983).

CALCULATIONS

The spinel crystal structure is a layered one, for in the [100] projection, it can be resolved into alternating layers of parallel non-identical [004] planes. One of these planes is composed of the octahedrally coordinated Al^{3+} ions and the other of the tetrahedrally coordinated Mg^{2+} ions. In this crystallographic projection, if one sets up a two-

dimensional diffraction condition by exciting a $\bar{g}$ = 004 systematic row, then the standing wave that is set up in the crystal under strong dynamical scattering conditions can be preferentially localized on one of these two planes with varying intensities by suitably altering the specimen tilt along the $\bar{g}$ = 004 direction with concomitant changes in the characteristic x-ray emissions (Spence and Taftø, 1983).

Hence, a 15-beam ($-7\bar{g}$ to $+7\bar{g}$) calculation for characteristic x-ray production intensities was carried out for $MgAl_2O_4$ (normal spinel) for the $\bar{g}$ = 004 systematic row and over a range of incident beam orientations specified by the parameter k_x/g (defined such that $k_x/g = 0.5$ for the first order Bragg diffraction condition). For an acceleration voltage of 100kV this theory predicts (Krishnan et al., 1985) that there is an enhancment of characteristic x-ray production for the tetrahedrally coordinated Mg atoms and the octahedrally coordinated Al atoms for positive excitation errors ($k_x/g > 0.5$) and negative excitation errors ($k_x/g < 0.5$) of the first order Bragg diffraction conditions respectively. This is in good agreement with the experimental results of Taftø and Spence (1982).

Similar calculations using equation (5) have been carried out for $MgAl_2O_4$ spinels over a range of incident beam orientations ($0 \leq k_x/g \leq 1.0$) and acceleration voltages (60keV - 400keV) by Krishnan, Rez and Thomas (1983). In order to isolate the role of the acceleration voltage alone, the variation of the ratio of the characteristic x-ray intensities as a function of the acceleration voltage were calculated for a number of different orientations and plotted in Figure 1. The ordinate $\mathbf{R}$ is a normalized measure of the orientation dependence and is defined as $\mathbf{R} = (R_1 - R_2)/(R_1 + R_2)$ where $R_1 = N_{Al-k}/N_{Mg-k}$ at the orientation of interest and $R_2 = N_{Al-k}/N_{Mg-k}$ at a symmetric orientation. From this plot, one can infer that at the "inversion" voltage, the normalized ratios of intensities does undergo a reversal in character, i.e. change in sign. These calculations were all done for a sample thickness of 100Å.

EXPERIMENTAL DETAILS AND RESULTS

Experiments were performed on single crystal $MgAl_2O_4$ with a spinel (Fd3m) structure. The single crystal was oriented and thin slices with a [100] normal were cut using a diamond wafering saw. They were mechanically thinned to a thickness of 35-50 microns and then ion-milled to perforations using argon ions accelerated through a potential of 5-6kV. This gave uniformly thin electron transparent regions of the specimen for TEM observations.

Experiments were performed on the Osaka analytical atomic resolution electron microscope fitted with a LaB_6 filament and a Tracor Northern energy dispersive x-ray analyser and capable of operating over an acceleration voltage range up to 400kV. A standard type two-axis side entry holder was used in all experiments. A specimen thickness that indicated a dynamical scattering condition judged by the appearance of the Kikuchi line pattern was selected. As the orientation dependence presupposes dynamical scattering, it is essential that the sample be of sufficient thickness ($t \geq \xi_g/2$). However, there is an upper limit of thickness ($t \leq 2000\mathring{A}$) corresponding to the attenuation distance of the poorly transmitted bloch waves. At distances greater than this, the electrons are diffusely scattered through small angles and effectively behave as plane waves in producing further x-rays. Therefore, all spectra were collected at an estimated specimen thickness of ~500Å, i.e. between half and one extinction distance thick (which of course changes with voltage).

Experimental orientations corresponding to the ones for which x-ray intensities were calculated were used. Hence a strong $\bar{g}$ = 004 systematic row was excited and spectra collected at five different orientations of the incident beam (specimen tilt/excitation errors):

(1) First order Bragg diffraction ($\bar{g}$ = 004) with large negative excitation error ($s \ll 0$)

(2) First order Bragg diffraction with small negative excitation error ($s < 0$)

(3) Exact first order Bragg diffraction condition ($s=0$)

(4) First order Bragg diffraction condition with small positive excitation error ($s > 0$)

(5) First order Bragg diffraction condition with large positive excitation error ($s \gg 0$)

Apart from this, spectra were also collected at a symmetric [100] zone axis pattern orientation (a reference orientation which would lend itself naturally to computation of the x-ray intensities) and at an orientation in which no lower order Bragg diffraction vectors were excited. The specimens were oriented using the Kikuchi line method.

At any particular acceleration voltage, spectra were collected at each of the above orientations for a period of approximately 300-600 seconds at a counting rate of ~500 cts/sec in order to ensure proper statistics. Parallel illumination was used throughout the experiment. It was also ensured that the specimen thickness remained uniformly constant for the different spectral acquisitions. This entire experimental procedure was repeatedly carried out at the following acceleration voltages: 125kV, 175kV, 225kV, 270kV, 300kV, 350kV and 400kV.

The results of the experiment are shown in Table 1. At each orientation and acceleration voltage the ratios of the k intensities of aluminum to magnesium, i.e. $r = N_{Al,k}/N_{Mg,k}$, is shown. This ratio is a convenient measure as it eliminates any dependence on experimental parameters such as counting time, beam spreading, etc. However, it is impossible to reproduce the orientations of the incident beam accurately at each acceleration voltage. Further, it is also likely that in some cases the incident beam

orientation corresponding to the first order Bragg diffraction condition with large positive excitation errors ($s \gg 0$) might have been very close to the second order Bragg diffraction condition ($\bar{g}$ = 008). These might explain some of the fluctuation in the data.

Figure 2 is a typical set of EDXS spectra collected at an acceleration voltage of 300kV. The accompanying diffraction patterns show the precise orientation of each acquisition.

DISCUSSION

The experimentally observed integrated elemental ratios show a clear variation with the orientation of the incident beam (specimen tilt) at each acceleration voltage and with acceleration voltage for each orientation (except for the non-Bragg case, at which no lower order diffraction vectors were excited). Further, these variations are statistically significant. However, in order to interpret the data and be able to compare spectra collected at different acceleration voltages, it is essential to normalize the data. We define a normalized measure ($\mathbf{R}_s$) of the orientation dependence at each acceleration voltage and incident beam orientation (s) as

$$\mathbf{R}_s = \frac{r_s - r_{zap}}{r_s + r_{zap}} \qquad (6)$$

where r_s is the ratio of the intensities $N_{Al,k}/N_{Mg,k}$ at the orientation s, and r_{zap} is the same ratio at a symmetric zone axis pattern orientation. Any variation in the value of $\mathbf{R}_s$ at constant acceleration voltage would then indicate an orientation dependence attributable to the preferential localization of the standing wave at the different planes within the crystal unit cell. The effect of voltage can be isolated by comparing values of $\mathbf{R}_s$ at a specific orientation (specimen tilt) specified by the excitation error, s. Further, $\mathbf{R}_s$ is a normalized thickness averaged intensity ratio. For the range of thickness that is

relevant the variation of $\mathbf{R}_s$ is only weakly dependent on thickness (Krishnan, Rez and Thomas, 1985) and would be negligible when compared to the statistical fluctuation in the data. Based on the experimental data, values of this normalized measure have been calculated, tabulated in Table 2 and plotted in Figure 3. The error bars were derived from the original counting statistics based on three standard deviations and the standard deviation was calculated as the square root of integrated counts after background subtraction.

From the experimental data (Figure 3) it can be seen that this orientation dependence of the characteristic x-ray emission exhibits an interesting reversal in character for negative excitation errors of the first order Bragg diffraction condition. Above some voltage (~270kV), which is referred to now as the "inversion" voltage the value of $\mathbf{R}_s$ undergoes a change in sign. Physically this could be interpreted as follows: Below the "inversion" voltage, $V < V_I$ one observes that the localization behavior of the standing wave within the crystal unit cell is such that for negative excitation errors ($s < 0$) of the first order Bragg diffraction condition ($g = \bar{4}00$), the strongly excited Bloch wave localization is enhanced on the octahedrally coordinated aluminum sites and for positive excitation errors ($s > 0$) of the first order Bragg diffraction condition, there is an enhanced localization of the strongly excited Bloch wave on the tetrahedrally coordinated magnesium sites. The concomitant variation in the characteristic x-ray production for the elements gives a negative value of $\mathbf{R}_s$ when normalized with respect to the symmetric orientation (Table 2) and is in agreement with the results of our calculations (Figure 1). However, the symmetric zone axis pattern orientation itself is a channelling orientation (S. J. Pennycook and J. Narayan, 1985) with an enhancement in the localization of the aluminum sites below the inversion voltage greater than the corresponding enhanced localization for negative excitation errors of the first order Bragg diffraction condition. Therefore, at these acceleration voltages ($V < V_I$) the decrease in the localization of the significant Bloch wave on the octahedral sites for positive excitation errors of the first

order Bragg diffraction condition (Table 1) gives rise to a <u>more</u> negative value of $\mathbf{R}_s$ at these orientations (Table 2). Further, if the normalization were to be carried out with respect to a non-symmetric, i.e. non-Bragg orientation the data would give a positive value of $\mathbf{R}_s$ in agreement with earlier experimental results (Taftø and Spence, 1982). On the other hand, above the inversion voltage ($V > V_I$), the experimental results indicate that $_s$ has a non-zero positive value, particularly for $s < 0$ and $s \ll 0$. A simple interpretation would be to attribute this to a change in the localization of the Bloch wave on the two crystallographic sites, i.e. localization on the tetrahedrally-coordinated magnesium sites for negative excitation errors and on the octahedrally-coordinated aluminum sites for positive excitation errors of the first-order Bragg diffraction condition respectively. This would lead to a considerable reduction of r_s for $s < 0$ such that $\mathbf{R}_s < 0$. However, for the symmetric orientation condition, there is a significant lowering in the value of r_{zap} for $V > V_I$ (Table 1), possibly due to a considerable drop in the localization of the Bloch waves on the aluminum sites. If this were to be the influential factor the parameter, $\mathbf{R}_s$ normalized with respect to r_{zap}, should increase considerably for all s. Therefore, it could be concluded that this orientation dependence is largely due to a change in the localization with incident beam orientation for the systematic orientation condition. This explanation, though consistent with the experimental observation, does not agree with the results of the theory (Figure 1) which predicts an enhanced inversion for positive excitation errors of the first-order Bragg diffraction conditions. This anomaly is not understood and is being pursued at present. It should be emphasized, however, that the inversion behavior predicted by the theory is indeed real and confirmed by the above observations.

This observation of the "inversion voltage" behavior seems to be quite different from the conventional critical voltage behavior observed for a centro-symmetric crystal set at the second order Bragg diffraction condition. This critical voltage behavior is attributed to an interchange of the symmetries and excitation amplitudes of the Bloch waves 2 and 3, that is, below the critical voltage wave 2 is symmetric and wave 3 is antisymmetric but

above the critical voltage wave 3 is symmetric and wave 2 is antisymmetric (Humphreys and Fisher, 1971). This would lead to a degeneracy of the eigenvalues of the Bloch waves (Lally et al., 1972) at the critical voltage. Our calculations (Krishnan, Rez and Thomas, 1983) of the dispersion surfaces for the different acceleration voltages at and around the above inversion voltage reveal no degeneracies, suggesting that this effect cannot be interpreted as an interchange of the symmetries of the Bloch waves. Further one can also calculate the critical voltage for the $MgAl_2O_4$ (spinel) using the following simple 3 beam expression given by Lally et al.:

$$V_c = \frac{2.26 \times 10^4}{[(\xi_{2g}^{100})^2 - (\xi_g^{100})^2]} (\xi_g^{100})^2 \ \xi_{2g}^{100} \ g^2 - 5.12 \times 10^5 \qquad (7)$$

where ξ_g^{100} and ξ_{2g}^{100} are two beam extinction distances for 100kV electrons. For $MgAl_2O_4$, $\xi_g^{100} = 850 \mathring{A}$ and $\xi_{2g}^{100} = 1531 \mathring{A}$. Using these values, the critical voltage for $MgAl_2O_4$ calculated using equation (7) is approximately 3.27 ± 0.325MeV. These arguments conclusively show that this "inversion voltage" is significantly different from the critical voltage effect.

From the microanalysis point of view it can be seen from Table 1 that only in the case in which no lower order Bragg diffraction vectors are excited is there no statistically significant variation with voltage of the characteristic x-ray intensities. This should be borne in mind when performing routine microanalysis of crystalline materials and the crystal systematically tilted to obtain such a diffraction condition before collecting energy dispersive x-ray spectra to avoid artifacts due to this kind of diffraction effects.

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TABLE 1

Integrated elemental intensity ratio $r = N_{Al}/N_{Mg}$

Acceleration Voltage	←— Orientation —→						
	s<<0	s<0	s=0	s>0	s>>0	zap	No Bragg
125kV	4.54	4.55	3.49	3.43	3.42	5.59	3.83
175kV	4.55	4.13	3.78	3.47	3.59	5.77	3.87
225kV	5.61	5.83	3.19	3.17	3.48	6.11	3.78
270kV	4.45	4.63	4.45	3.23	3.42	4.37	3.71
300kV	5.07	4.51	3.50	3.18	3.45	4.32	3.82
350kV	5.47	5.39	4.61	3.24	3.38	4.29	3.77
400kV	5.00	5.07	3.89	3.14	3.41	4.27	3.72

TABLE 2

Normalized measure of orientation dependence--**R**

Orientation (Specimen tilt/excitation errors)

Acceleration Voltage	$s \ll 0$	$s < 0$	$s = 0$	$s > 0$	$s \gg 0$
125kV	-0.10	-0.10	-0.23	-0.24	-0.24
175kV	-0.12	-0.17	-0.21	-0.25	-0.23
225kV	-0.04	-0.02	-0.31	-0.32	-0.27
270kV	0.01	0.03	0.01	-0.15	-0.12
300kV	0.08	0.02	-0.10	-0.15	-0.11
350kV	0.12	0.11	0.04	-0.14	-0.12
400kV	0.08	0.09	-0.05	-0.15	-0.12

Fig. 1. Variation of the electron induced characteristic x-ray emissions as a function of the acceleration voltage for fixed excitation errors. Normalization is done with respect to a symmetrical orientation.

Fig. 2. A typical set of energy dispersive x-ray spectra as a function of orientation of the incident beam at an acceleration voltage of 300kV. Parallel illumination conditions were used. In the insets the precise orientation of each acquisition is shown.

Fig. 3. Characteristic x-ray production intensities in $MgAl_2O_4$ (spinel) as a function of acceleration voltage and incident beam orientation. The normalized measure R_s (see text for definition) is defined such that any non-zero value indicates an orientation dependence. Note the change in the sign of R_s as a function of incident beam orientation for voltages greater than 270kV.

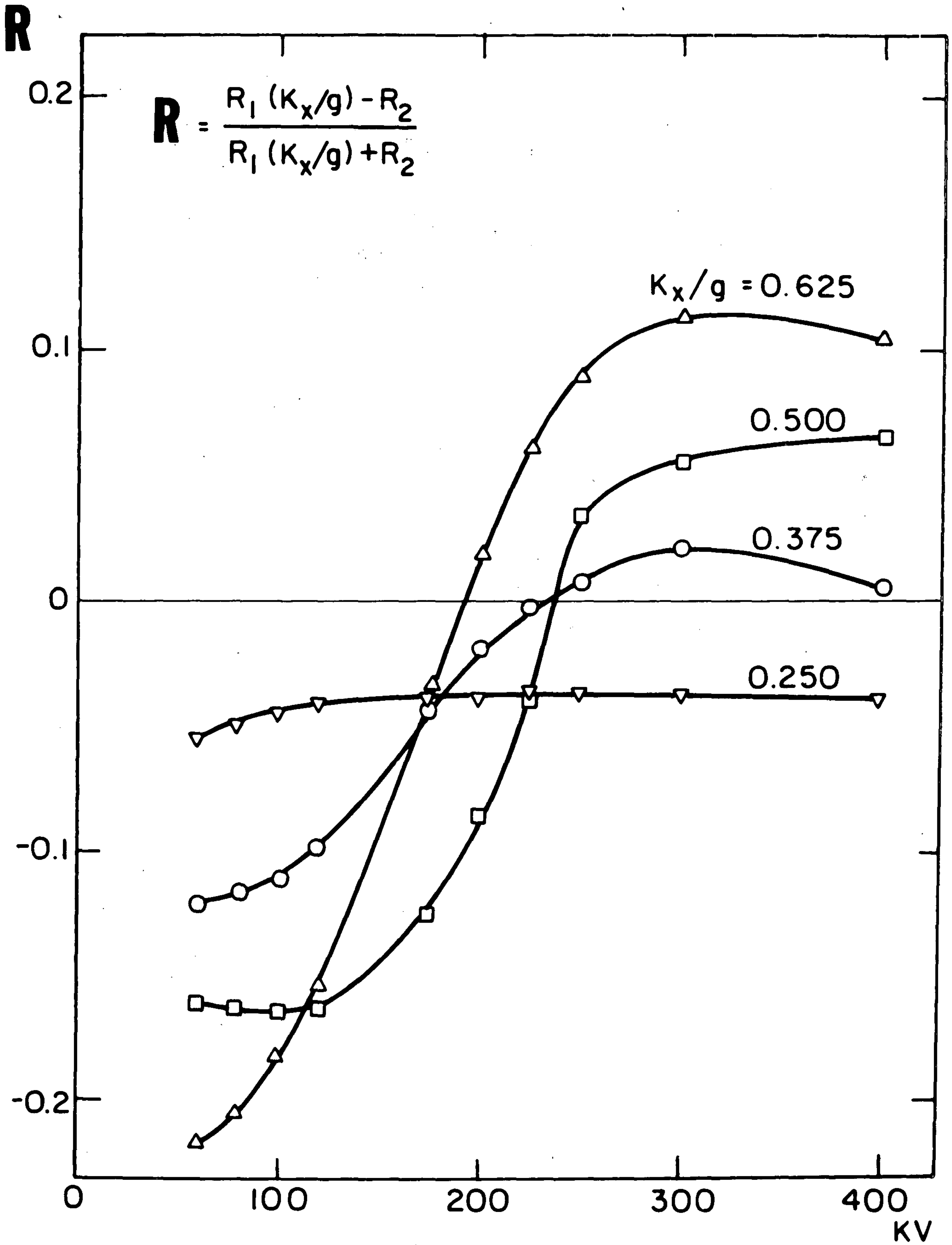
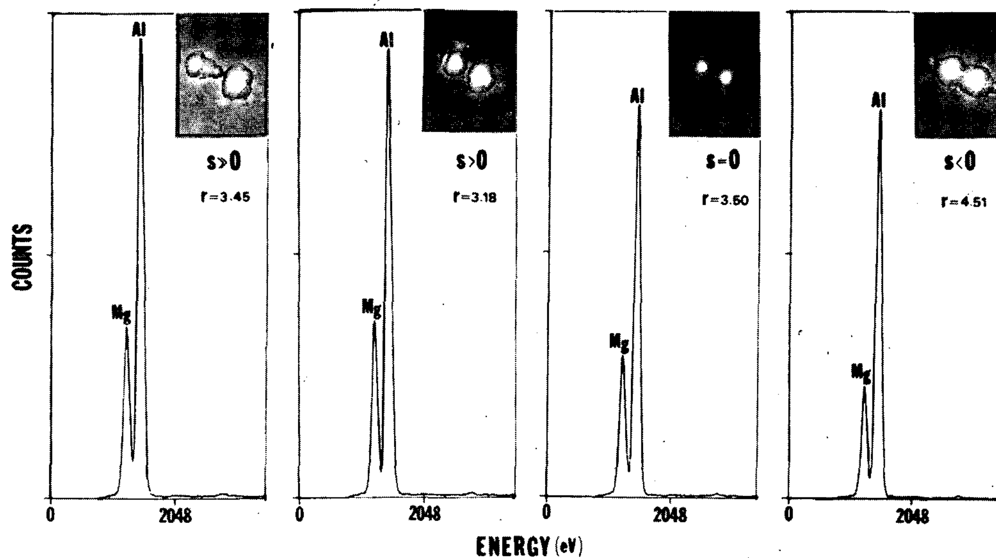
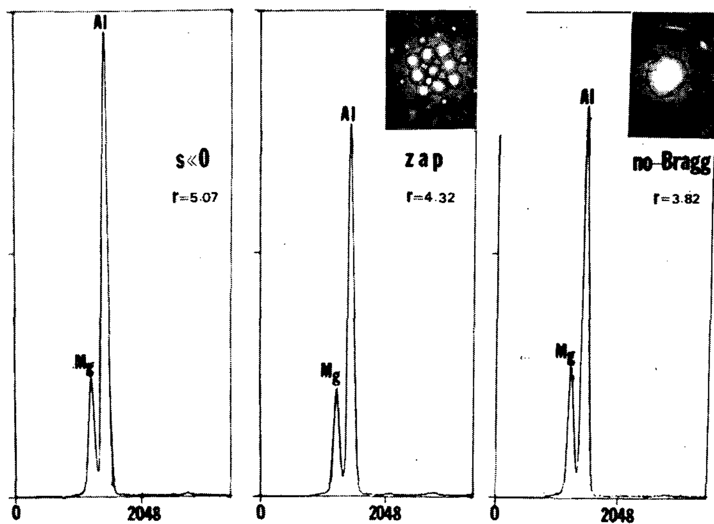


Fig. 1

XBL 859-4025





$MgAl_2O_4$

300 Kv

$r = N_{Al}/N_{Mg}$

XBB 855-3698

Fig. 2

XBL 859-4024

Fig. 3

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