

Difference in spectral reflectivity between grains of homogeneous and exsolved titanomagnetite

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SUMMARY. The spectral reflectivities of homogeneous and exsolved titanomagnetites of the same chemical composition were measured in air and in oil. The reflectivities of the two kinds of titanomagnetite are closely similar in the blue part of the spectrum, but become increasingly different when proceeding towards the red end. The refractive indices (n) and the absorption coefficients (k) were calculated from the reflectivity values. The n -values of the two kinds of titanomagnetite show a pattern of separation similar to that of the reflectivity values. Regarding possible differences between the k -values no reliable conclusions could be drawn. Some possible explanations of the observed separations are discussed.

THE present paper forms the first part of an investigation of the relations between chemical composition and spectral reflectivity in the magnetite-ulvöspinel series. Reflectivity measurements have earlier been made on a homogeneous titanomagnetite (Cervelle, 1967), magnetites from different localities (Demirsoy, 1968), lunar ulvöspinel (Cameron, 1970), and on homogeneous titanomagnetites of varying compositions (Prévoit and Mergoil, 1973). From these measurements reflectivity values for some compositions in the magnetite-ulvöspinel series have been obtained. However, to establish a relation between reflectivity and composition, additional data are needed.

Normally in plutonic rocks a primary homogeneous phase of titanomagnetite has changed by oxidation and exsolution to Ti-poor magnetite and ilmenite. However, if a lower oxygen fugacity prevails a significant part of the original content of Ti remains in the magnetite phase. At lower temperature this phase becomes unstable and since cooling proceeds slowly in this kind of rock exsolution into one magnetite and one ulvöspinel phase takes place. Though the two phases are not pure end components the names magnetite and ulvöspinel are used in this paper for simplicity. Exsolved titanomagnetites are reported from numerous localities, and since homogeneous titanomagnetites usually do not occur in plutonic rocks, the investigation of the relations between reflectivity and composition will essentially be carried out on exsolved titanomagnetites. The purpose of the present work was to study whether exsolution results in a change of reflectivity as compared with the primary homogeneous phase.

Material and methods

The titanomagnetite investigated was derived from a single hand specimen taken from an ore concentration in the Ulvö dolerite, a flat-lying layered intrusion in the

eastern part of central Sweden. Microscopical analysis of a polished section demonstrated that the titanomagnetite was exsolved into two phases.

Cube-like crystallites of magnetite with a diagonal usually less than $0.4 \mu\text{m}$ were found in a matrix of ulvöspinel. Virtually no inclusions of ilmenite or any other mineral were observed. Reflectivity measurements on several grains at a wavelength of 546 nm showed values ranging from $R 0.167$ to $R 0.170$. This small variation in reflectivity was regarded as an indication of a rather constant composition of the titanomagnetite within the hand specimen, though this has to be checked by electron-probe analysis. From these observations it was concluded that it should be possible to separate and homogenize the titanomagnetite without changing its composition. The composition of the obtained homogeneous grains should then be approximately equal to that of the unhomogenized grains in the polished section. One fraction of the separated grains was analysed by X-ray diffraction, the other fraction was homogenized by heating in evacuated and sealed silica tubes. Part of the homogenized material was analysed by X-ray diffraction and from the rest of it a polished section was prepared.

Preparation of polished sections. Cut slices or separated grains were moulded in an epoxy resin. Grinding was then carried out in four different stages on a cast-iron lap in a slurry of SiC-powder and water; the grades of SiC-powder used were: SiC-280, SiC-400, SiC-600, SiC-1000. The rough polishing stages were performed on a petrodisc, a cast aluminium disc coated with a partly self-lubricating hard plastic layer. This disc gives rise to practically no relief. On a first petrodisc $7\text{-}\mu\text{m}$ diamonds and on a second $3\text{-}\mu\text{m}$ diamonds were used as abrasive. Minute amounts of paraffin oil were added as lubricant.

The remaining polishing stages were all carried out on discs covered with a synthetic cloth called pella pan-W. A stage using $3\text{-}\mu\text{m}$ diamonds was followed by one using $1\text{-}\mu\text{m}$ diamonds. In both a mixture of distilled water and ethanol was used as lubricant. In the final stage the specimens were polished in a slurry of MgO-powder and distilled water. The two first grinding steps were carried out manually. All other stages until the final polishing stage were carried out on a Struers DP-U2 machine equipped with a PDM-force specimen holder. Interstage cleaning was performed with an ultrasonic cleaner. The quality of the polished surfaces was examined under a $16/0.22$ interference contrast objective. All grains on which reflectivity measurements were performed showed perfectly plane and scratch-free surfaces.

Reflectivity measurements. Apparatus used: Leitz Microscope Photometer MPV on an Ortholux-Pol polarizing microscope. Light-measuring device Type BN 5002 T with high-stability power supply Type NSHM from Knott Elektronik. $60\text{W}/12\text{V}$ lamp stabilized by current stabilizer from Knott Elektronik. Norma Lichtmarkengalvanometer 1705 GB 2 D. Running interference-filter type Veril-200, with the following pass bands given by the manufacturer: 12 nm at 550 nm , 13 nm at 450 and 650 nm . The specimens were fixed on a Leitz Levelling Table. A SiC-crystal polished and macroscopically calibrated by Firma Carl Zeiss was used as reflectivity standard (SiC 47 42 51 no. 121). This standard was mounted in a holder, equipped with three levelling screws, which can be placed at the top of the levelling table, making it possible to level the grain to be measured and the standard separately.

Precautions to minimize the errors in the reflectivity measurements were taken in accordance with the results obtained by Piller (1967), Demirsoy (1968), and Galopin and Henry (1972). To minimize errors due to wrong settings of the running filter only positions at scale marks were used. Therefore the measurements were carried out at the following wave-lengths: 458, 481, 499, 525, 546, 559, 578, 596, 617, 640, and 655 nm. The measurements were performed in air using a 16/0.40 objective. In addition one grain from each of the homogenized and the unhomogenized specimens were measured in oil in order to calculate the refractive indices (n) and the absorption coefficients (k). In these measurements a 25/0.65 immersion objective was used. A Zeiss immersion oil filling the requirements of COM (Commission of Ore Microscopy) was used. The n -values of this oil are found in Table II (Min. Mater. News Bull. Quant. Microsc. Meth. 1974, 3).

The part of the illuminated area of a grain corresponding to the opening of the photometer stop was always set to an order of $30\ \mu\text{m} \times 30\ \mu\text{m}$. No significant changes in the galvanometer reading occurred on slightly displacing the measured grains. Regarding the grains of exsolved titanomagnetite this shows that the illuminated area was so large compared to the single exsolution bodies that effects due to variations in the distribution of these bodies were averaged out.

At every second wave-length, starting with 458 nm, four to five repeated measurements were carried out on each grain. At the remaining wave-lengths only two repeated measurements were performed. Each of the repeated measurements were usually performed on different days, and the adjustments of the apparatus were always reset.

The relative standard deviations of these measurements varied from 0.3 % to 0.5 %. The variation in the reflectivity value of the same area of a grain received from repeated measurements expresses the resultant effect of the nonsystematic errors. Assuming normal distribution of the variation, the confidence interval at the 95 % level for the mean value of five measurements is almost equal to $\pm\sigma$ (σ = standard deviation). Taking into account the resulting reflectivity values of two, four, and five measurements a relative precision of the order of ± 0.6 % seems probable.

The refractive index of the immersion oil decreases by 0.000 34 per °C of temperature rise, and the ideal temperature at which the measurements should be performed is 23 °C. Measurements of the temperature close to the microscope showed variations between 21 and 24 °C. The reflectivities of the specimen and standard also decrease with temperature, and the error introduced by using the standard value of 23 °C when the working temperature was no more than 2° from the ideal was calculated to be less than 0.1 % and can thus be disregarded.

In order to estimate the effect of bad polishing, including possible differences in polishing hardness of the two exsolved phases, the reflectivity of one grain from the unhomogenized specimen (no. 1) was measured repeatedly after different polishing stages. After polishing with 3- μm diamonds on a petrodisc the surface still looked rather rough under an ordinary objective; $R = 0.156$ at 546 nm. After polishing with 1- μm diamonds the surface looked almost perfect under an ordinary objective, but rough under the interference contrast objective; $R = 0.166$ at 546 nm. After the final polishing in a slurry of MgO-powder and distilled water the surface looked completely

even and scratch-free, $R = 0.169$ at 546 nm. Though there was a large difference in polishing quality between the two last stages their reflectivities only differed by $R = 0.003$. Differences in polishing finish so small that they cannot be detected under the interference contrast objective should thus hardly lead to any significant differences in the measured reflectivity values.

Primary glare (see Piller, 1967) was measured using a 'black hole' box. With the diaphragm settings used no glare was detected; with improper settings, however, a measurable glare was observed.

Secondary glare was calculated using the formula given by Piller (1967) and assuming a reflectivity of $R = 0.02$ for the objective (measurements on the flat lens surface of the 16/0.40 objective resulted in values lower than that used in the calculation at all wave-lengths). The calculation showed that this type of glare is completely negligible in the present investigation.

Piller (1972) measured the variation in reflectivity of micro-areas in SiC-standards, and found that the relative standard deviation from the macroscopically calibrated value was of the order of $\pm 0.4\%$. The relative accuracy, taken as $\pm 2\sigma$, is $\pm 0.8\%$. The relative precision of the measurements in the present work was not influenced by such a variation since the same micro-area of the standard was used in all measurements.

Of the errors mentioned only the resultant nonsystematic error and the error in the standard value seem to be of importance. Since they are surely independent the relative accuracy is $\pm(0.6^2 + 0.8^2)^{\frac{1}{2}} = \pm 1.0$. The minor systematic errors, neglected in the above estimation, probably give rise to a resulting error not larger than $\pm 0.5\%$. Taking such an error into account the estimate is $\pm(0.6^2 + 0.8^2 + 0.5^2)^{\frac{1}{2}} = \pm 1.12$. A relative accuracy of $\pm 1\%$ is therefore assumed.

Electronmicroprobe analysis. As part of the investigation of the relations between chemical composition and reflectivity, complete analyses of four grains in the unhomogenized specimen were carried out. The conditions under which these analyses were performed will be described in a following paper. The compositions of two homogenized grains were compared with those of the four unhomogenized grains: The number of counts during ten seconds for Fe, Ti, Mg, and Al were measured in each of the six grains. To ensure that the number of counts could be compared in a meaningful way, all grains were analysed during one single run, without changing the settings of the electronmicroprobe analyser.

Results

Electronmicroprobe analysis. The four grains in the unhomogenized specimen were similar in composition. Expressed in atoms % Fe varied from 30.1 % to 30.3 %, Ti from 7.3 % to 8.0 %, Mg from 2.1 % to 2.3 %, and Al from 1.7 % to 1.9 %. The two grains from the homogenized specimen gave counts for each of these elements falling within these intervals. For the purpose of this work the grains of the two specimens may consequently be regarded as isochemical.

X-ray diffraction of the unhomogenized material showed spinel reflexions that were separated into broadened double peaks. According to Basta (1960) this pattern is

obtained in most X-ray diffraction analyses of titanomagnetites with exsolution texture. The analysis of the homogenized material showed only well-defined single reflexions situated at spinel positions, indicating that the homogenization was complete. Completion was also confirmed by microscopical examination.

Reflectivity measurements. Table I gives all obtained reflectivity values. In fig. 1 the values in air are plotted against the wave-length. Plots 1 to 3 refer to the grains in the unhomogenized specimen. The values of the fourth grain are not plotted. Plots 1h and 2h refer to the grains in the homogenized specimen. All the plotted values lie close to

TABLE I (left). *Spectral reflectivity values measured in air and oil. Nos. 1 to 4 denote grains from the unhomogenized, nos. 1h and 2h from the homogenized specimen*

TABLE II (right). *R_a and R_o values of grains 1 and 2h, estimated from the freehand curves in figs. 1 and 2. Calculated values of the refractive indices (n) and absorption coefficients (k), and refractive indices (n_o) of the immersion oil*

	Air						Oil		R_a		R_o		n		k		n_o	
	1	2	3	4	1h	2h	1	2h	1	2h	1	2h	1	2h	1	2h		
458 nm	0.160	0.158	0.160	0.159	0.158	0.159	0.047	0.047	460 nm	0.1606	0.1591	0.0474	0.0487	2.249	2.235	0.401	0.408	1.5264
481	0.161	0.159	0.160	0.160	0.158	0.160	0.048	0.047	480	0.1609	0.1599	0.0478	0.0475	2.253	2.239	0.397	0.411	1.5240
499	0.162	0.161	0.162	0.162	0.159	0.161	0.049	0.049	500	0.1626	0.1600	0.0493	0.0485	2.259	2.240	0.412	0.423	1.5218
525	0.166	0.163	0.164	0.165	0.161	0.162	0.051	0.050	520	0.1648	0.1622	0.0511	0.0496	2.267	2.248	0.429	0.427	1.5199
546	0.169	0.168	0.169	0.170	0.164	0.164	0.055	0.052	540	0.1676	0.1640	0.0533	0.0513	2.278	2.250	0.449	0.451	1.5181
559	0.171	0.169	0.171	0.172	0.165	0.167	0.056	0.053	560	0.1713	0.1663	0.0558	0.0528	2.302	2.267	0.457	0.451	1.5166
578	0.174	0.172	0.173	0.174	0.167	0.167	0.058	0.054	580	0.1747	0.1682	0.0580	0.0541	2.327	2.280	0.458	0.455	1.5153
596	0.178	0.176	0.177	0.177	0.169	0.170	0.059	0.055	600	0.1778	0.1699	0.0598	0.0552	2.357	2.294	0.445	0.451	1.5141
617	0.180	0.178	0.179	0.180	0.170	0.171	0.061	0.056	620	0.1800	0.1713	0.0612	0.0560	2.376	2.310	0.438	0.441	1.5130
640	0.182	0.180	0.181	0.182	0.171	0.172	0.062	0.056	640	0.1816	0.1723	0.0621	0.0566	2.395	2.320	0.423	0.434	1.5121
653	0.182	0.181	0.182	0.183	0.172	0.173	0.063	0.057	660	0.1827	0.1730	0.0629	0.0567	2.404	2.338	0.422	0.410	1.5112

each other in the blue part of the spectrum, but towards the red end an increasing difference between the values of the homogenized and the unhomogenized grains becomes evident. The grains no. 1 and 2h were selected to be measured in oil. Fig. 2 shows the resulting curves, which are similar to those in fig. 1.

Calculation of n and k. From Fresnel's formula for reflectivity the following formulae for n and k can be derived:

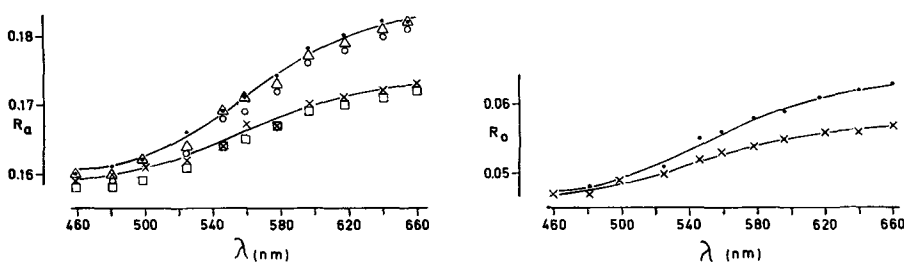
$$n = 0.5(n_o^2 - 1)(1 - R_a)(1 - R_o) / \{n_o(1 - R_a)(1 + R_o) - (1 + R_a)(1 - R_o)\}$$

$$k = n \{2(1 + R_a) / n(1 - R_a) - (1 + 1/n^2)\}^{\frac{1}{2}}$$

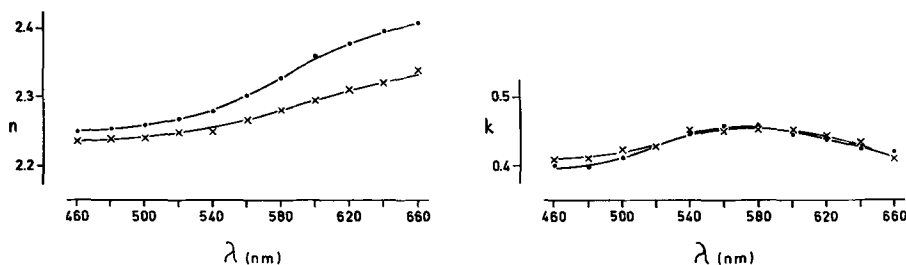
where R_a is the reflectivity in air, R_o the reflectivity in oil, and n_o the refractive index of the immersion oil (in the formula for k given by Piller and Gehlen (1964) the n in front of the square root is missing). In figs. 1 and 2 freehand curves fitting the plotted values of grains no. 1 and 2h were drawn. From these curves the reflectivity values to be used in the calculations of n and k were derived. These reflectivity values and the calculated values of n and k are given in Table II. The n-values are plotted in fig. 3 and the k-values in fig. 4. Before drawing any conclusions regarding these plotted values the accuracy of the calculated values must be considered. If true reflectivity values are R_a and R_o , combinations of relative errors of the order of $\pm 1\%$ in one or

both of these values can be: $(1.01R_a, R_o)$, $(0.99R_a, 1.01R_o)$, and so on. There are eight possible combinations.

Using different values of R_a and R_o , representative of the present material, the relative errors in n ($\Delta n/n$) and k ($\Delta k/k$) were calculated for all eight combinations. $|\Delta n/n|$ varied from 0.6% to 2.9% and $|\Delta k/k|$ from 4% to 38%. (A thorough study of the errors in the n - and k -values derived from reflectivity data is given by Piller and Gehlen (1964)). Returning to the plotted values in figs. 3 and 4 it can be seen that the



FIGS. 1 and 2: FIG. 1 (left). The reflectivity in air (R_a) plotted as a function of the wave-length. Free-hand curves fitting the plotted values of grains no. 1 and 2h. Grain no. 1 black points, no. 2 circles, no. 3 triangles, no. 1h squares, and no. 2h crosses. FIG. 2 (right). The reflectivity in oil (R_o) plotted as a function of the wave-length. Freehand curves fitting the plotted values. Grain no. 1 black points, no. 2h crosses.



FIGS. 3 and 4: FIG. 3 (left). The refractive index (n) plotted as a function of the wave-length. FIG. 4 (right). The absorption coefficient (k) plotted as a function of the wave-length.

n -curves separate in the same way as the reflectivity curves. The k -curves on the other hand almost coincide. However, a slight change in the freehand curves in figs. 1 and 2, in such a way that they still fit the plotted values, leads to significant separation of the curves. Therefore it cannot be concluded whether there are significant differences between the k -values of the two types of titanomagnetite or not.

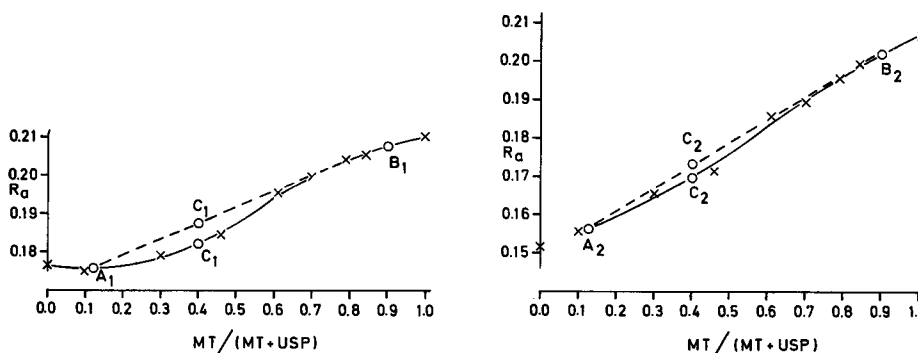
Regarding the n -curves it is possible to bring them to coincide by changing the reflectivity values by $< \pm 1\%$. This is most efficiently done by moving the R_a - and the R_o -curves in opposite directions. In the red part of the spectrum each curve must be moved by an order of $\pm 0.5\%$, and further each of the curves of grain no. 1 must be moved in the opposite direction compared with the corresponding curve of grain

no. 2h. It seems, however, rather improbable that resultant errors of the order of 0.5 % systematically should be directed in opposite directions for spectral curves so similar as those in question. It is therefore assumed that the n -values of the two types of titanomagnetite are significantly separated in the red part of the spectrum.

Discussion and conclusions

The obtained results imply that any simple relation between chemical composition and reflectivity that is valid for grains showing exsolution texture can be applied to homogeneous grains only in the blue part of the spectrum.

The rest of this paper is devoted to a discussion of three possible ways in which the observed difference in reflectivity behaviour can be explained:



FIGS. 5 and 6: FIG. 5 (left). The reflectivity in air (R_a) at 617 nm of synthesized titanomagnetites of varying compositions plotted as a function of the magnetite/(magnetite + ulvöspinel) ratio, $Mt/(Mt + Usp)$. For further explanation see the text. FIG. 6 (right). The reflectivity in air (R_a) at 499 nm of synthesized titanomagnetites of varying compositions plotted as a function of the magnetite/(magnetite + ulvöspinel) ratio, $Mt/(Mt + Usp)$. For further explanation see the text.

A. The observed difference may be caused by a slightly uneven surface due to different polishing hardness of the two exsolved phases. This interpretation, however, is improbable in the light of the polishing experiments described earlier in this paper.

B. The reflectivity values of homogeneous synthetic titanomagnetites, measured by the present author, are plotted against the magnetite/(magnetite + ulvöspinel) ratio in figs. 5 and 6. (The study of these synthetic specimens will be reported in a later paper.) Fig. 5 shows the reflectivity at 617 nm and fig. 6 at 499 nm. Consider a homogeneous titanomagnetite having a composition and reflectivity values fixed by point C_1 in fig. 5 and C_2 in fig. 6. If this titanomagnetite exsolves into two phases with compositions and reflectivity values given by points A_1 and B_1 in fig. 5 and A_2 and B_2 in fig. 6, the reflectivity values of the exsolved titanomagnetite are given by C'_1 and C'_2 . The difference between C'_1 and C_1 is significantly larger than the difference between C'_2 and C_2 . This is in accordance with the results we are trying to explain. At present, however, it is impossible to state whether the difference in spectral reflectivity

between the homogenized and the unhomogenized titanomagnetites from the Ulvö dolerite is completely explained by this model, because the synthetic titanomagnetites have compositions strictly on the Fe_3O_4 - Fe_2TiO_4 join, whereas the investigated titanomagnetites from the Ulvö dolerite contain significant amounts of Al and Mg. The spectral reflectivities of magnesioferrite, spinel, and hercynite (Demirsoy, 1968) indicate that the presence of these elements influences the shapes of the curves in figs. 5 and 6.

C. From the discussion under point *B* it follows that at present we do not know whether part of the difference in spectral reflectivity between homogeneous and exsolved titanomagnetites still remains to be explained. Additional influences may depend on the following circumstances: Reflectivity depends on the interaction of the incident light wave with the electrons in the reflecting medium, which is in turn a function of the geometric and energetic configuration of all the electrons. If the configuration is changed the reflectivity is usually changed too. If the exsolution is coherent, and if there is a difference in cell edge between the two phases, this difference is compensated by elastic strain (Brett, 1964). Elastic strain in the lenses of microscope objectives is known to change linearly polarized light to elliptically polarized light. Significant changes in optical behaviour can thus result from elastic strain. The two exsolved phases in titanomagnetites both have spinel structures, the difference in cell edge being of the order of 1 % only. In the present material the crystallites of magnetite are seldom larger than $0.4 \mu\text{m}$ in diagonal. They are always orientated with their sides parallel to each other (exsolution along the (100)-planes according to Ramdohr, 1953). All these circumstances are in favour of a coherent exsolution. The X-ray diffraction analyses clearly demonstrate two phases with different cell-edges. The broadened double peaks can be explained in at least two ways. Broadening may be caused by a continuous change in Fe/Ti ratio in the boundary zones of the two phases leading to a continuous change in cell edge. In this case no compensation by elastic strain is necessary. On the other hand, if there is a chemical discontinuity across the phase boundary, compensation by elastic strain gives rise to a continuous change in cell edge. The fact that there is an exsolution into two phases with different compositions implies that all intermediate compositions are thermodynamically unstable. A continuous change in composition in the boundary zones could then occur only if the rise in free energy is less than the elastic strain energy occurring in the alternative case. In an investigation by Vincent, Wright, Chevallier, and Mathieu (1957) measurements of saturation magnetization at various temperatures indicate that the Ti-poor phase remains constant in composition during the homogenization process, but decreases in amount by dissolving into the Ti-rich phase. The second alternative, which suggests a discontinuous change in composition across the phase boundary, is consequently the only possible one. The strain necessary to compensate a difference in cell edge of 1 % may appear too small to cause any significant changes in the electron configuration. There are, however, variables important for the electron configuration that are very sensitive to changes in the cell edge.

A strict treatment of the relations between elastic strain, electron configuration, and reflectivity requires quantum mechanic calculations beyond the scope of this paper.

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Errata: Table II, the right-hand column of each pair should be headed 2h. Figs. 5 and 6, the upper curve in fig. 5 should be labelled C'_1 , and in fig. 6, C'_2 .