JOURNAL OF MATERIALS SCIENCE 23 (1988) 2021–2028

Structure studies of non-crystalline materials by electron diffraction

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The ability of electron diffraction to determine the structure of non-crystalline materials has been critically revised on the basis of the main sources of error: the premature temination of the experimental intensity curve and the problems associated with the elimination of the inelastically scattered intensity. A method of studying the problem is presented and the efficacy of the most representative procedures for reducing errors in the final correlation functions is checked.

1. Introduction

It is well known that liquids and amorphous solids do not have periodic atomic arrangements. However, because atoms have well defined sizes and closest distances of approach, both liquids and amorphous solids have definite structures relative to an origin at the centre of an average atom. This structure is described by

$$4\pi R^2 \varrho(R) = 4\pi R^2 \varrho_0 + \frac{2R}{\pi} \int_0^\infty S\, i(S) \sin(SR)\, dS$$

in which $\varrho(R)$ is the density of atoms, $4\pi R^2 \varrho(R)$ the radial distribution function (RDF(R)), ϱ_0 the mean density of atoms, $S = 4\pi \sin \theta/\lambda$ and the kernel of the integral

$$F(S) = S\, i(S)$$

is sometimes called the reduced interference function. Another useful correlation function, $Q(R)$, is defined as

$$Q(R) = 4\pi R[\varrho(R) - \varrho_0]$$

In practice, $i(S)$ is calculated directly from the corrected and normalized intensity observable in a diffraction experiment as

$$i(S) = \frac{I_n - \langle f^2 \rangle}{\langle f \rangle^2}$$

where I_n is the coherent-scattering intensity in absolute units, $f(S)$ are the scattering factors and $\langle\ \rangle$ refers to composition averages.

In general, information about the short-range structure can be obtained from the position and area of maxima in RDF curves: nearest-neighbour spacings, average number of nearest neighbours, the mean dihedral angle. However, the final result is subject to many sources of error depending on the experimental radiation used, although the termination of the experimental data at finite scattering vectors is the main challenge to obtaining reliable structural data for these disordered systems.

Most experimental work has involved neutron and X-ray scattering experiments. As pointed out by Cargill [1] electron-scattering studies on amorphous solids have been limited to thin film samples and, although some authors used electron diffraction [2–10], this technique has not been widely used because it presents some difficulties. Major problems are the correction of inelastic scattering and the very limited range ($S_{\max} \simeq 80\,\mathrm{nm}^{-1}$) over which rather noise-free intensity data can be obtained.

In spite of the above considerations, we think that electron diffraction can give an important contribution to the knowledge of the structure of liquids and amorphous solids taking into account the following considerations:

(1) X-ray and neutron-diffraction experiments can provide only an averaged picture of the structure of disordered systems (about 10^{20} to 10^{21} atoms are sampled) and then local information is lost. In fact, the correlation functions evaluated from these experiments are insensitive to local phenomena such as phase separation [11]. On the other hand, we are interested in glasses based in selenium, some of which contain regions with extensive chemical segregation [12, 13] and we think that electron-scattering measurements, involving about 10^3 atoms, can distinguish multi-phase compositions [14].

(2) Along with reciprocal-space scattering techniques, TEM experiments provide complementary information in real-space over the same region.

(3) Electron diffraction is the most suitable technique to study thin foils.

Although we believe that the above considerations are very convincing, one has to bear in mind that if we attempt to get a further insight into atomic arrangements and to devise a model of the amorphous structure, highly accurate and meaningful RDF functions are needed. Thus, the question is raised: are quantitatively worthwhile the electron diffraction data or otherwise, their results are only qualitative?

The present work attempts to answer this question and to arouse interest in electron-diffraction measurements. In the next section, the main sources of error are discussed, then we present our method studying the problem and, finally the validity of the main

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procedures for the reduction of errors on severely limited raw data are analysed.

2. Sources of error in electron scattering

The major problems in this studies have been multiple scattering [15], uncertain scattering factor [15, 16], "aliasing" [17] due to the discrete character of Fourier transformation and some systematic errors as the normalization error [18], but the main amount are due to truncation errors and a wrong inelastic-scattering correction. We follow with both main sources of error.

2.1. Truncation errors

Owing to the small wavelength associated with electrons, one would expect it to allow the exploration of large distances in reciprocal space. However, because of the S-dependence of scattering factors, $f(S)$, and the usual system of data recording, the range is limited to about $S_{\max} < 120\,\text{nm}^{-1}$ [1]. However, our own experience shows that beyond $S_{\max} \simeq 80\,\text{nm}^{-1}$ it is not possible to determine faithful data with conventional instruments. This truncation of data causes two errors in the RDF functions: (i) loss of resolution, (ii) spurious ripples of wavelength $2\pi/S_{\max}$ (sidebands).

2.2. Inelastic scattering

From the totally scattered intensities one has to eliminate the inelastic contribution which gives a continuous background under these curves. Errors in the background corrections are responsible for mistaken determination of the radii r_i of the coordination spheres and of the coordination numbers, n_i.

Several procedures have been used [6, 7] to determine this correction, but they are affected by a remarkable amount of arbitrariness; accordingly, we only retain two procedures: the analytic background correction method of D'Antonio *et al.* [19] and the "crystalline phase" method.

In the first method, the background line $B_m(S)$ is generated as

$$B_m(S) = [u_m(S)B_{m-1}(S) + v_m(S)b_m(S)], \quad m = 1, \ldots, n$$

where m represents the number of a segment in a set of n segments, $u_m(S)$ and $v_m(S)$ are weighting coefficients in the overlap range of two segments, $b_m(S)$ is a function given by

$$b_m(S) = \exp(\alpha_m + \beta_m S^{\gamma_m})$$

the background constants α_{m}, β_{m} and γ_{m} being determined on the basis of some positivity and area criteria.

Although this method has been successfully applied [19], we have chosen the other one because for many cases the background cannot be described via a set of analytic functions [7], and as we have less confidence on final results coming from sophisticated corrections and complicated refinements.

The "crystalline phase" method is based upon the assumption that the smooth curve through the minima of the corresponding polycrystalline phase (obtained by heating the amorphous films) represents the inelastic background, which has to be substracted from the totally scattered intensity. The method was first suggested by Kommik [5] and justified by Gandais [9]. We have used an improved version [20, 21] which allows a simultaneous normalization and inelastic contribution correction, the interference function, $i(S)$, being directly obtained as

$$i(S) = \frac{I(S) - I_{\text{med}}(S)}{I_{\text{med}}(S) - \alpha I_{\text{c}}^{\text{b}}(S)}$$

where $I(S)$ is the scattered intensity by the amorphous system in arbitrary units, $I_{\text{med}}(S)$ is a smooth curve through the inflection points of $I(S)$ and represents its medium level signal, $I_{\text{c}}^{\text{b}}(S)$ is the "crystalline" background obtained in similar conditions that $I(S)$ and α is an adjustable parameter which give account that both measurements (amorphous and crystalline phase) correspond to separated recordings. The knowledge of α is related to N, the normalization constant via

$$I_{\text{med}}(S) = N\langle f(S)\rangle^2 + \alpha I_{\text{c}}^{b}(S)$$

From the above equation we evaluate α and N by means of a least squares procedure and then, we calculate the $i(S)$ function.

On the other hand, very accurate electron-diffraction experiments have been realized with energy filters and electronic-intensity detection [4, 22–24]. However, we are dealing with conventional electron microscopes which are not fitted with a velocity filter.

3. Method of analysis and experimental procedure

In order to obtain a quantitative evaluation of the effects, on the final RDF, of the termination error, and to check the efficiency of the methods consecrated to restore the true unaffected RDF, we have devised a simple method: we start with the published $i(S)$ data of Waseda [25] on liquid selenium at 523 K, which extend to a high upper limit ($S_{\max} = 148\,\text{nm}^{-1}$ and $S_{\min} = 5\,\text{nm}^{-1}$) and we calculate the corresponding distribution functions (which will be our reference functions for comparison). Then, we purposely truncated the experimental data at the node in $S = 80\,\text{nm}^{-1}$ to create a new data set of much shorter length. Another RDF was computed using this new data. Fourier transformations were performed using Filon's [26] quadrature with basic intervals of $1\,\text{nm}^{-1}$ for S. For both sets of data the reduced interference function was previously extrapolated to zero, in the experimentally inaccessible region of very small S, by means of the usual procedure [27]. Subsequently, we try and evaluate the different correction strategies.

A residual factor largely used [28–30], namely

$$\Delta = \frac{\sum_i |A_i - B_i|}{\sum_i |A_i|}$$

where A_i and B_i refer to the reference and trial RDF, respectively, was used to check the agreement between both distribution functions. Note that we have chosen RDF functions as a reference for comparison instead of the $Q(R)$ ones based on two reasons: (i) with regard to the effects of the finite upper limit in S, RDF becomes worse because of the implicit convolution does not distort or shift peaks in $Q(R)$ and, peak shapes are affected in the RDF by the extra factor of R. Thus, this reference is a more severe test. (ii) The RDF is directly used to get the short-range order information.

In every case the internal consistency of the $i(S)$ data was checked according to the method proposed by Rahman [31], which showed that $i(S)$ must satisfy the following relation:

$$4\pi\varrho_0 l^3 \left(\frac{J_1(\mu_l)}{\mu_l}\right) = 1/(\mu\pi)\int_0^\infty Si(S) \times \{J_0[(S+\mu)l] - J_0[(S-\mu)l]\}\,\mathrm{d}S$$

J_0 and J_1 being spherical Bessel functions, μ an arbitrary parameter in nm^{-1} and 1 must be less than the radius R_c of the sphere such that for any $R < R_c$, the function RDF(R) is effectively zero.

Although the determination of the radii of the coordination spheres from the RDF is straightforward, nevertheless, the measurement of the coordination number is not so evident because: (i) different criteria in determining the area under the coordination peak [25, 32], (ii) overlapping between neighbouring maxima and, mainly, (iii) the area being dependent of the maximum scattering vector, $S_{\max}$. Therefore, we have used the method of Stetsiv [33], based on the theoretical relation between the height of the first maxima and $S_{\max}$. This method allows us to obtain the coordination number, n, and the mean-square amplitude of vibration of the atoms in the first coordination shell, free from the aforementioned drawbacks.

It is obvious that this proposed method allows us to study the influence of the termination of data, but it is unable to evaluate errors due to a bad inelastic scattering correction since we start with the $i(S)$ data. In order to quantify this influence we have obtained electron-diffraction raw data of amorphous selenium up to $S_{\max} = 80.5\,\mathrm{nm}^{-1}$. Then we compare the RDF corrected for termination errors by the most efficient method and whose inelastic scattering was corrected by the "crystalline phase" procedure, with another one obtained by means of a very large set of X-ray data for which the inelastic scattering correction is more simple by far.

Other experimental precisions follow:

(a) The thin films of amorphous selenium were obtained by thermal evaporation.

(b) The electron diffraction experiments were performed inside a Phillips EM 300 electron microscope operated at 80 KV.

(c) Diffraction patterns were recorded photographically on Kodak film and then scanned with a Joyce Loebl-3cs microdensitometer. The photographic calibration was carried out by the Karle method [34].

4. Systematic errors

In order to take into account only the termination error, we have corrected the original data set and the truncated one (which we refer to as data 1 and data 2, respectively, in the following discussion) from the effects of systematic residual errors. Since these errors usually appear as low frequency perturbations which affect the low-R region, it can be eliminated following a method of repeated Fourier transforms [9, 18, 35]: near the origin the correlation function $Q(R)$ must be represented by a straight line with a slope $m = -4\pi\varrho_0$. Then, the $Q(R)$ curve deduced from the experimental data is modified setting the values in the region of subatomic distances equal to $-4\pi R\varrho_0$, and a new interference function is obtained by the reverse transformation. The difference between both interference functions show up the systematic errors which can be removed giving a corrected interference function, whose Fourier transform will give the corrected correlation function. This procedure can be iterated although no substantial improvement was observed beyond two iterations.

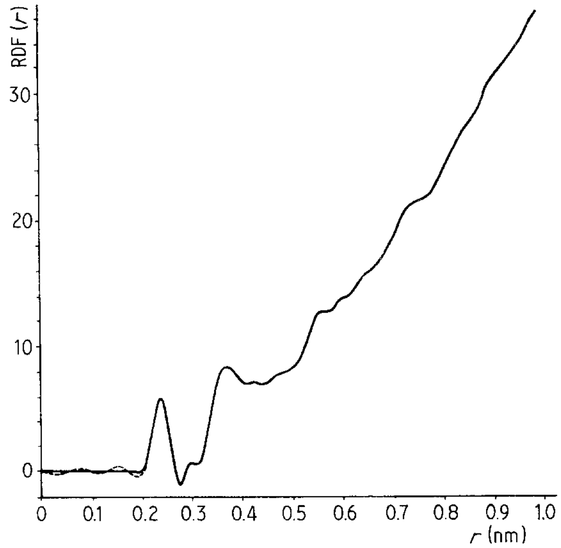

Figure 1 RDF functions derived from the complete original data of Waseda (– – –) and that corresponding to the corrected one from systematic errors (——).

The radial distribution function derived from data 1, that we set up as "reference", is shown in Fig. 1 along with the corrected one from systematic errors. We can see that the only effect of this correction take place on the low-R ripples, leaving unaffected the maxima. In Table I we show the results of the Rahman test relative to the 'reference' data.

In Fig. 2 we show the RDF derived from data 2 and corrected from systematic errors, along with the reference. It can be seen from comparing both curves the remaining low-R riples and a remarkable loss of

TABLE I Test of the original $i(S)$ data (corrected from systematic errors) by the Rahman method. The last columns list, respectively, the left and right hand-side of the Rahman equation

L	μ		
0.500	0.350	0.156 598 E − 01	0.156 942 E − 01
	0.850	0.154 261 E − 01	0.154 597 E − 01
	1.350	0.150 038 E − 01	0.150 358 E − 01
1.000	0.350	0.124 131 E + 00	0.124 106 E + 00
	0.850	0.118 816 E + 00	0.116 795 E + 00
	1.350	0.104 203 E + 00	0.104 189 E + 00
1.500	0.350	0.412 539 E + 00	0.411 914 E + 00
	0.850	0.359 054 E + 00	0.358 554 E + 00
	1.350	0.273 824 E + 00	0.273 515 E + 00

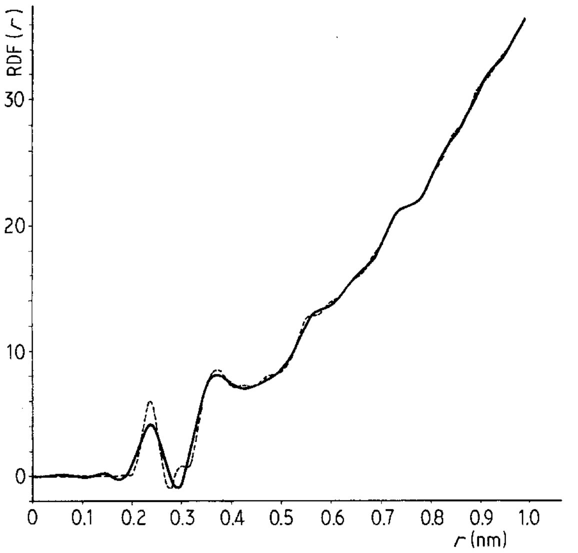

Figure 2 RDF derived from the limited data and corrected from systematic errors (——) and the "reference" (– – –).

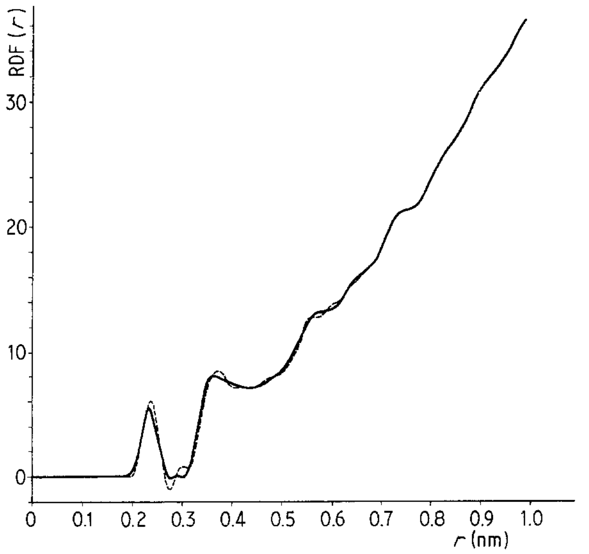

Figure 4 Comparison of the RDF obtained by the Narayan method and the "reference" (– – –).

resolution on curve 2. However, the corrections account for a considerable improvement in the residual factor (from $\Delta = 2.36\%$ to 1.72%).

5. Extension of the observed scattered data

The traditional approaches to obtain reliable RDF functions are either to extend the observed data to large values of S or to replace $S\,i(S)$ by $S\,i(S)\,M(S)$, where $M(S)$ is a convergence factor. We now consider the first case.

Various methods of extrapolating the experimentally inaccessible data have been suggested. We have used the negative cut-off procedure of Narayan [36], based upon the strict positivity of the $G(R) = \varrho(R)/\varrho_0$ function and avoiding the biasing of other methods.

The extended $i(S)$ function is plotted in Fig. 3. The bar in $S = 79\,\text{nm}^{-1}$ denotes the separation between the extended data and the original ones corrected from systematic errors. It is worthwhile indicating that this previous refinement was a necessary condition for the convergence of the Narayan method. Fig. 4 shows the obtained RDF, which is a close fit to the reference. Data were extended up to $300\,\text{nm}^{-1}$ and we have performed 45 iterations (the residual factor was lowered until $\Delta = 1.26\%$).

We have also tried the extension method due to Shevchik [37], which has been applied by D'Anjou [38], but our results were very poor. In this method the $S\,i(S)$ function is assumed a weighted sum of sine terms. For the large-S oscillation in $S\,i(S)$ only one term may be retained yielding to a damped sine wave which is fitted by least squares to these oscillations. In our case, the last two oscillations of the reduced interference function do not correspond strictly to the large-S region and more terms of the sum should be considered.

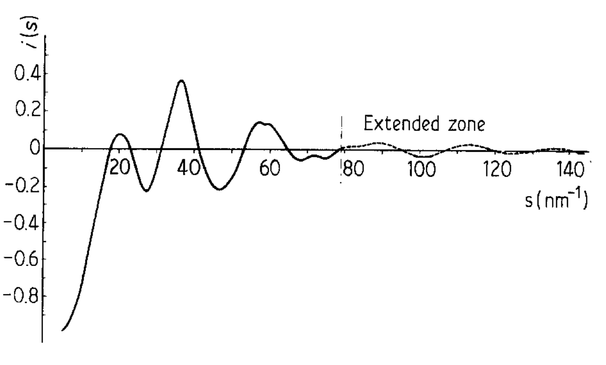

Figure 3 Extended $i(S)$ function obtained using the iterative procedure of Narayan.

6. Convergence factors

Some convergence factors, $M(S)$, are frequently used to suppress the spurious termination maxima. The change of $S\,i(S)$ by $S\,i(S)\,M(S)$ has two purposes: (i) it reduces the importance of the less accurate high-angle data, (ii) it decreases the sharp cut-off of data at $S_{\max}$. The more used $M(S)$ functions have been the Gaussian (artificial temperature factor) [39–41]

$$M(S) = \exp(-bS^2)$$

and the resolution function or Lanczos window [11, 17, 42]

$$M(S) = \frac{S_{\max}}{\pi S}\sin(\pi S/S_{\max})$$

The latter has a clear physical basis in that limitation of data to $S < S_{\max}$ is equivalent to a resolution in the direct space of $2\pi/S_{\max}$ and, moreover, it has been suggested [11] that the more well behaved results are obtained by means of this function. Furthermore, the practical efficacy of the Gaussian window is restricted because of the existence of an upper bound of the parameter b which corresponds to a "superheating" of the phase [43].

Whenever we use a convergence factor, the calculated correlation function, $Q_c(R)$, becomes [39]

$$Q_c(R) = Q(R)*C_W(R)*C_M(R)$$

where * denotes convolution and, the smearing functions $C_W(R)$ and $C_M(R)$ are, respectively, the Fourier transforms of the observation rectangular window and the $M(S)$ discriminative window.

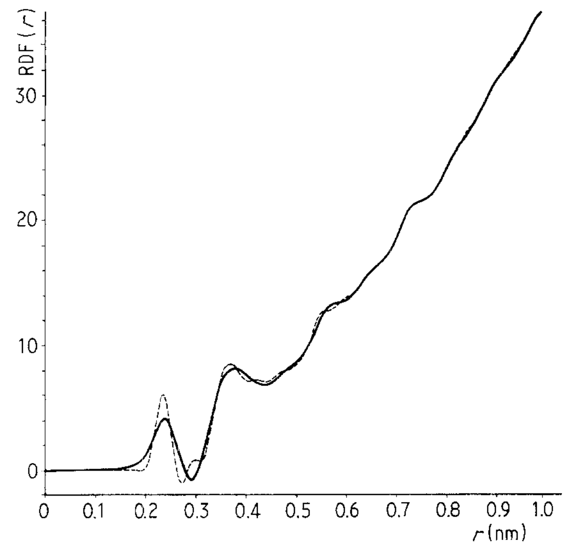

Figure 5 RDF obtained using a Gaussian convergence factor, $M(S_{\max}) = 0.1$, and deconvoluted by the iterative method of Van Cittert–Ergun.(– – –) "Reference".

As it has been previously discussed [39, 44], this double convolution involves an important broadening of the maxima and such artifact is the price we must pay in order to remove the spurious features. The classical deconvolution methods [45–47] are unable to retrieve the true $Q(R)$ function from the smeared $Q_c(R)$; for example, Fig. 5 shows the result of the iterative method of Van Cittert–Ergun [45], where we have used a Gaussian convergence factor so that $M(S_{\max}) = 0.1$ (no improvement was observed beyond six iterations), and a poor agreement is obtained. In fact, the method improves discreetly the raw result obtained by simply applying the convergence factor but it is unable to correct the broadening coming from the limitation of data because of the involution property [48] exhibited by $C_w(R)$ (the sampling function).

However, the approximate Mencik method [49] allow us to overcome this problem by supposing an analytical form to the $Q_c(R)$ maxima, by means of a trial-function procedure. A first insight into the potentiality of the method was carried out by Warren [39] which, however, dealt with the less efficient Gaussian discriminative window [44]. In the latter reference we have presented and tested a useful version of Mencik's method where $M(S)$ is the Lanczos function and it is very appropriated for the case of sharp peaks on a smooth background.

At this point we outline the main steps of this approximate treatment:

(i) Separation of $Q_c(R)$ in a slowly varying background, $Q_0(R)$, plus the relevant maxima, $Q_{c_i}(R)$,

$$Q_c(R) = Q_0(R) + \sum_i Q_{c_i}(R)$$

so that each $Q_{c_i}(R)$ maxima, centred at R_i, undergoes the correction procedure while the background is unaffected by the convolution.

(ii) Analytical approximation of the ideal peak profile

$$Q_i(R) \simeq Q_{a_i}(R)$$

We have tried Gaussian and Lorentzian profile functions, which have been largely used in the Rietveld refinement of crystal structures from powder diffraction data [29].

(iii) Determination of the smearing function, $C(R)$

$$C(R) = [C_W(R)] * [C_M(R)]$$

(iv) Convolution of the assumed $Q_a(R)$ function with $C(R)$ to yield a convoluted function, $F(R)$

$$F(R) = [Q_a(R)] * [C(R)]$$

(v) Subtraction of the convoluted function, $F(R)$ from the calculated $Q_c(R)$ one

$$e(R) = Q_c(R) - F(R)$$

This difference function contains some information on the corrections to be applied to the assumed Q_a function.

Evaluation of a better approximation to the ideal $Q(R)$ maximum, $Q'(R)$:

$$Q'(R) = Q_a(R) + e(R) = Q_a(R) + Q_c(R) - F(R)$$

With regard to $Q_a(R)$ we have distinguished two cases:

6.1. Gaussian fit

$$Q_a(R) = A \exp(-a^2R^2)$$

(for simplicity the peaks are supposed as centred at the origin). Then, we have showed [44] that

$$Q'(R) = A \exp(-a^2R^2) + Q_c(R) - A(F(R)/\mathrm{A})$$

and

$$F(R)/A = \exp(-a^2R^2)\, C(R)$$

where

$$C(R) = (S_{\max}/\pi)\{[Si(\pi - S_{\max}R) + Si(\pi + S_{\max}R)]/2\pi\}$$

$Si(X)$ being the sine integral

$$Si(X) = \int_0^x \frac{\sin t}{t}\,\mathrm{d}t$$

and the parameters A and a are evaluated in such a way that the $F(R)$ peak matches the width and height of the corresponding Q_c maxima.

6.2. Lorentzian fit

$$Q_a(R) = A/(1 + a^2R^2)$$

The obtained result for this case is similar to the precedent one except that now $F(R)/A$ is

$$F(R)/A = (S_{\max}^2/2\pi^2 a) \times \left\{\mathrm{sinc}\,(S_{\max}R) * \left[\arctan\frac{2a\mu}{1 + a^2(R^2 - \mu^2)}\right]\right\}$$

where $\mu = \pi/S_{\max}$ and $\mathrm{sinc}(X) = (\sin X)/X$.

In Fig. 6 we show the first maxima of the RDF(R) (data 1) together with that calculated via the Lanczos function as weighting window and the best fit obtained by the deconvolution method above exposed (with Gaussian fit and the back-shift correction referenced in [44]). The residual factor, for the case of Lanczos function without deconvolution was $\Delta = 2.96\%$, and

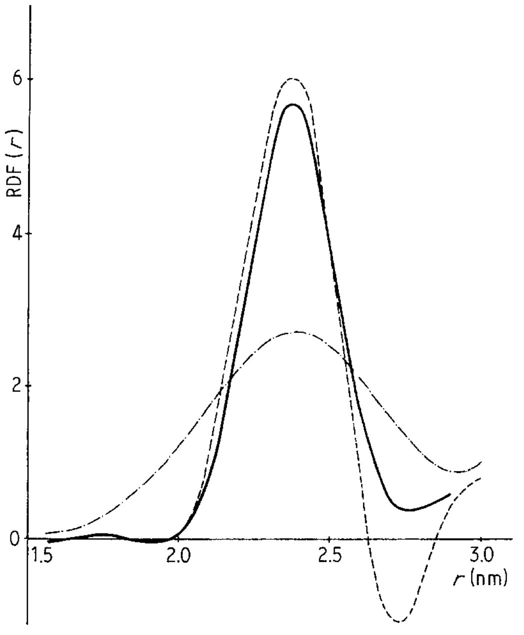

Figure 6 First maximum of the RDF: (a) (—·—) calculated using the Lanczos function as convergence factor, (b) (——), deconvoluted from the previous one, and (c) (– – –), "reference".

remarkable improvements (1.68% and 1.40%, depending whether only the first or the two first maxima were deconvoluted) were obtained.

Nevertheless, the result for the Lorentzian fit was in poor agreement with the reference, suggesting that the shape of the considered maxima departs drastically from Lorentzian.

7. The maximum entropy method

Very recently, Wei has published [50] an original insight into the problem by proposing a method based upon the information theory and which avoids the subjective judgements implicit in other methods. The maximum entropy method (MEM) showed that the optimum $\varrho(R)$ has the form

$$\varrho(R_j) = \exp\left[-1 - v_0 - \sum_{l=1}^{L} v_l \operatorname{sinc}(R_j S_l)\right]$$

where the $(L + 1)$ set of v parameters are found by substituting the above equation into the equation

$$\sum_{j=1}^{M} 4\pi R_j^2 \varrho(R_j) = \sum_{j=1}^{M} 4\pi R_j^2 \varrho_0$$

which express the normalization condition for the radial density, and into the L equations

$$\sum_{j=1}^{M} 4\pi R_j^2 [\varrho(R_j) - \varrho_0] \operatorname{sinc}(R_j S_l) = i(S_l),$$

$$\text{for } l = 1, 2, \ldots, L$$

corresponding to the Fourier inversion and, $i(S_l)$ being the L observed values of the interference function.

The resulting equations are highly non-linear and have been solved by a hybrid algorithm based on Newton's method [51].

It is apparent from Fig. 7 the excellent quality of the RDF obtained by this method ($\Delta = 1.54\%$) in the following conditions.

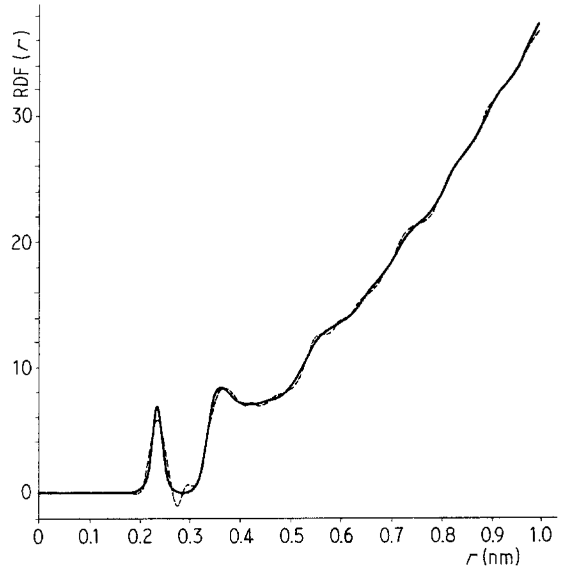

Figure 7 RDF restored via the MEM method, along with the "reference" (– – –).

(1) The intervals in reciprocal space were $\Delta S = 2\,\mathrm{nm}^{-1}$.

(2) In real space we have sampled $M = 250$ spherical shells by steps of $R = 1$ nm.

Moreover, the following considerations (not contemplated in the original paper of Wei) can be drawn:

(1) Unstable solutions and unreliable results were obtained whenever the input data were not refined. High quality of data is a prerequisite.

(2) The limit of the Nyquist images, $2\pi/\Delta S$, must be avoided.

On the other hand, we propose an alternative variant of the Wei method which consists of evaluating the $\varrho(R)$ function and then, by using the inverse Fourier equations, we may obtain the $i(S)$ function over an extended range. This extension of the truncated data can be observed in Fig. 8.

Although reliable RDF functions have been obtained by means of this last variant, nevertheless the goodness of the fit ($\Delta = 2.02\%$) is lower than the obtained via the direct method of Wei.

8. Remarks on the precedent methods

A few other methods have also been tried to deal with termination errors, as the sampled transform method of Lovell *et al.* [52] which is a very efficient one but, owing to the severe termination of data, it gives very scarced points in the real space resulting in a poor resolution.

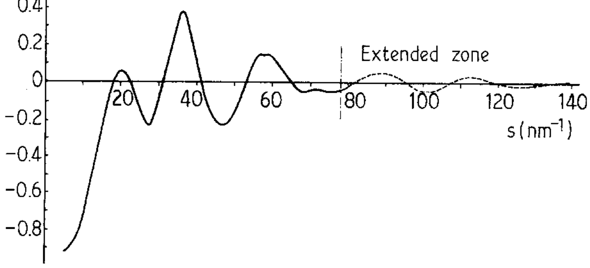

Figure 8 Extended $i(S)$ function obtained by the MEM method.

TABLE II Parameters which define the relative merit of the correction methods: (Δ), residual factor; (R_1), first-neighbour distance; (R_2), second-neighbour distance; (n), coordination number. Procedures: (1) original data; (2) raw truncated data; (3) truncated data corrected from systematic errors; (4) Narayan method; (5) Lanczos function and deconvolution; (6) MEM method.

	Δ	R_1	R_2	n
(1)	–	2.36	3.71	2.04
(2)	2.36	2.38	3.83	2.09
(3)	1.72	2.38	3.65	2.00
(4)	1.26	2.34	3.64	2.05
(5)	1.40	2.36	3.73	2.00
(6)	1.54	2.36	3.65	2.00

With regard to the above mentioned methods we show in Table II a summary of the parameters which define its relative success.

The best approach to the reference (the smallest Δ value) is obtained by the Narayan method. However, in some way, the RDF obtained via the MEM method could be interpreted as the optimum approach in the feeling that its result could be a closer fit to reality than the adopted reference itself.

9. Application to amorphous selenium

In order to evaluate the influence of errors related to a bad inelastic correction, we have applied the "crystalline phase" and Narayan methods to thin film samples of amorphous selenium whose electron diffraction spectrum was recorded up to $S_{\max} = 80.5\,\mathrm{nm}^{-1}$.

Fig. 9 shows both radial distribution functions: that obtained according to the above procedure and that published by Andonov [53], obtained with X-rays and $S_{\max} = 175\,\mathrm{nm}^{-1}$ (the residual factor is $\Delta = 3.6\%$). Provided that inelastic scattering corrections are straightforward for X-rays and that a residual factor $\Delta = 1.2\%$ corresponds to the best fit in the correction of termination errors for liquid selenium, we conclude that inelastic scattering correction errors amount to about twice the magnitude of those for termination.

10. Conclusions

The contribution of this work lies in providing results and arguments to discuss the relative significance of the main sources of error in electron diffraction, in testing the efficiancy of some methods to correct them and in deciding about the quantitative meaning of the obtained results.

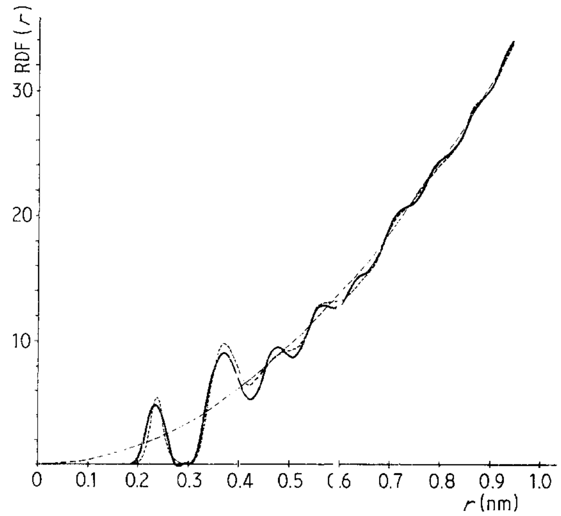

Figure 9 Corrected RDF obtained from data of amorphous selenium (——) and that published by Andonov (– – –).

In earlier sections we have showed that termination errors are less significant that those derived from bad inelastic scattering corrections. We have also made manifest the power of some methods to reduce the truncation errors even for drastic limitation of data. Such results prompt the question: are the obtained RDF functions accurate enough to envisage comparisons with those calculated from models? In this matter we think that the results might be improved if a comparison with fine details of models of the amorphous structure is wanted.

Nevertheless, we suggest that qualitative and semiquantitative deductions are meaningful and that comparison with computer generated models could be very useful with regard to the positions of the main peaks, number of first neighbours and the approximate magnitude of the maxima.

Acknowledgements

We are grateful for many helpful discussions with Dr A. Criado.

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Received 3 March
and accepted 24 September 1987