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Temperature dependence of EXAFS Debye–Waller factors in β - and γ -AgI

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Abstract. EXAFS at the iodine L_3 edge in β - and γ -AgI has been measured. The EXAFS Debye–Waller factors of the first and second coordination shells of iodine have been extracted by a standard analysis based on the harmonic approximation. No difference has been found between the temperature dependence of mean square relative displacements (MSRD) of the two β and γ phases. The harmonic approximation has been checked against more complete anharmonic EXAFS and x-ray diffraction analyses; it has been found adequate to interpret the MSRDs of AgI for temperatures lower than 300 K. The experimental MSRDs for the two distances I–Ag and I–I have been compared with MSRDs calculated on the grounds of the available lattice dynamics models. The sensitivity of EXAFS to phonon polarization properties has been made evident.

1. Introduction

Extended x-ray absorption fine structure (EXAFS) has been recognized to be a useful tool for studying the local vibrational dynamics of crystalline solids. A great deal of experimental tests of the actual potentialities of EXAFS as a vibrational probe have been done for metals [1, 2]: the temperature dependence of the EXAFS Debye–Waller (DW) factor has been successfully described by simple Einstein or Debye models. However, these models proved to be inadequate for more complex systems [3, 4]. For such systems the experimental thermal behaviour of DW has to be analysed by more refined models of vibrational dynamics, which generally rely on harmonic approximation.

A particularly interesting system is AgI whose phonon density of states is far from having a Debye behaviour. AgI is a polymorphic substance, and below 420 K and at atmospheric pressure two crystallographic phases coexist: a HCP form with wurtzite structure, denoted by β -AgI, and a FCC form with sphalerite structure, denoted by γ -AgI. The two modifications of AgI differ for the stacking sequence of atomic planes, respectively in the c direction for the β phase and in the [111] direction for the γ phase. At 420 K AgI undergoes a first-order transition to the superionic α phase. Crystalline structure and dynamic properties of AgI in both β and α phases have been studied extensively by many techniques [5] but only a few reports exist on the γ phase [5, 6]. AgI shows an anharmonic behaviour of both I and Ag atoms even at relatively low temperatures. This anharmonicity has been investigated by various authors [7–11]: EXAFS measurements at the K edge of Ag in AgI carried out by Boyce *et al* [10]

showed that in β -AgI the distribution of Ag-I distances is asymmetric, the extent of asymmetry increasing with temperature. Recently the thermal behaviour of β -AgI was re-investigated by x-ray diffraction by Yoshiasa *et al* [11] at 123, 297, 363 and 413 K; the thermal motion of both Ag and I atoms was shown to be anharmonic even at the lowest temperature.

This work is intended to complete and extend the results of our previous study on the DW factors of AgI obtained by the analysis of the EXAFS at the L_3 edge of iodine [12] in the framework of the harmonic approximation. In our previous paper we studied the temperature dependence of DW factors from 50 K to room temperature in a powdered sample containing a mixture of both β and γ phases. We found that the correlation of atomic motion affects the EXAFS DW factor much more strongly for the I-Ag than for the I-I distance. We succeeded in interpreting the experimental behaviours of the DW factors of the first two coordination shells of iodine by a hybrid Einstein-Debye model which explicitly took into account the phonon eigenvectors of the low-frequency optical modes calculated at the centre of the first Brillouin zone (BZ).

In this paper we present the EXAFS data from room temperature down to 11 K for the two distinct β and γ modifications and compare them with previous data of the mixed sample. No structural measurements on AgI below the liquid nitrogen temperature have been published. Our object is to test, on AgI, the peculiar sensitivity of the EXAFS DW factor to the phonon polarization properties. To this aim, firstly we study the limits of harmonic approximation in extracting and interpreting the EXAFS DW factors of AgI; secondly we compare the experimental DW factors of the first two shells of I in β and γ modifications with the available force constant models taking into account the eigenvectors of vibrational modes of the whole BZ.

2. Experimental details

The β phase of AgI is commonly recognized as stable below 420 K; however, any mechanical treatment, such as grinding β monocrystals, causes a partial transformation to γ phase. γ -AgI has been described as a metastable polytype of β -AgI which transforms to β phase by annealing or aging. It is not easy to prepare β -AgI samples in powder form, as required for absorption measurements (*vide infra*), without the presence of a fraction of γ phase and vice versa. Commercial AgI powders are generally a mixture of the two phases with concentrations that depend on the preparation method. To isolate samples containing only one phase we started our preparation procedures from a high-purity AgI powder (purchased from C Erba, Milan, Italy). The β sample was obtained by adding the commercial powder to a saturated solution of $KI \cdot H_2O$; a small deposit of very fine powder was obtained after two weeks. The γ sample was obtained by pressing the commercial powder at 5 GPa for a few seconds and then milling the pellets in an agate mortar. The purity degree of each phase was determined by x-ray diffraction: accordingly, in what follows the term ' γ -AgI' indicates a sample with a largely prevalent (over 90 %) presence of zincblende phase, while ' β -AgI' indicates a pure wurtzite phase. In order to obtain samples with uniform thickness, as required by x-ray absorption spectroscopy in transmission mode, the very fine powders (less than 5 μm in size) were dispersed in alcohol by means of an ultrasonic mixer. Then they were left to settle down slowly on a polytetrafluoroethylene membrane. Samples with $\Delta\mu x = 0.7$ and total $\mu x \leq 2.3$ at the iodine edge were prepared using one or two

membranes. More details on samples preparation and characterization are given in reference [13].

EXAFS measurements were carried out in transmission mode at the PULS synchrotron radiation facility of the Frascati National Laboratories with an electron beam energy of 1.5 GeV and a maximum stored current of 50 mA. Data were collected with a channel-cut Si(111) monochromator crystal. The standard energy calibration was made at the K edge in Cu metal, $E = 8978.9$ eV. The energy resolution was estimated to be 0.8 eV at 4560 eV, mainly determined by the monochromator rocking curve. Ion chambers filled with Ar were used as detectors. Temperature was changed from 11 K to 292 K by a helium cryostat; helium gas flowed in a cold stream where the sample holder and a silicon diode sensor were mounted. The estimated accuracy of temperature measurements was ± 2 K.

3. Results

The experimental EXAFS signal $k\chi(k)$ for the β and γ phases at low (11 K) and high temperature (250–260 K) are presented in figure 1. The wavevector k of the photoelectron depends on the x-ray energy E according to the relation $k = \sqrt{2m_e E/\hbar^2}$. To extract the EXAFS signal from the absorption coefficient we used the same procedure adopted in our previous work on AgI in mixed phase [12] to which the reader is referred for details. The Fourier transforms of the $k\chi(k)$ functions in the range 2.3–8.1 \AA^{-1} are shown in figure 2; a strong similarity between the spectra of the two phases is evident. We will limit our analysis to the first two shells: the contribution of the four silver atoms of the first shell extends from 1.4 to 3.4 \AA , and the peak at 4.3 \AA is due to the 12 iodine atoms of the second shell. The multiple scattering contribution to the EXAFS signals of the second shell has been evaluated to be negligible [13].

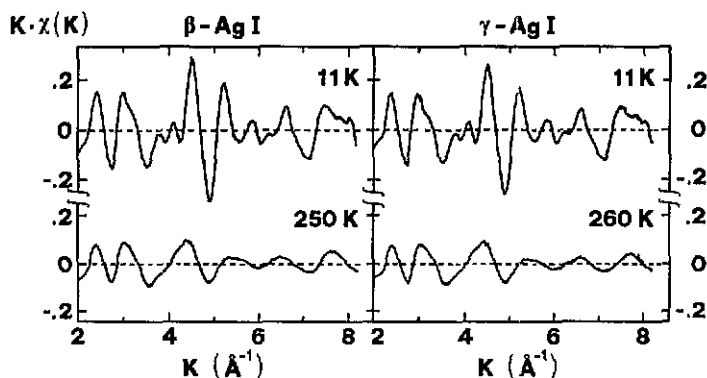


Figure 1. Experimental EXAFS signals $k\chi(k)$ at the L_3 edge of iodine in β and γ -AgI at low and high temperature.

The EXAFS oscillations, within the single-electron, single-scattering, plane wave approximation and in the hypothesis of small harmonic thermal disorder, are described by [14]:

$$\chi(k) = \sum_j A_j \sin(2kR_j + \phi_j) \quad (1)$$

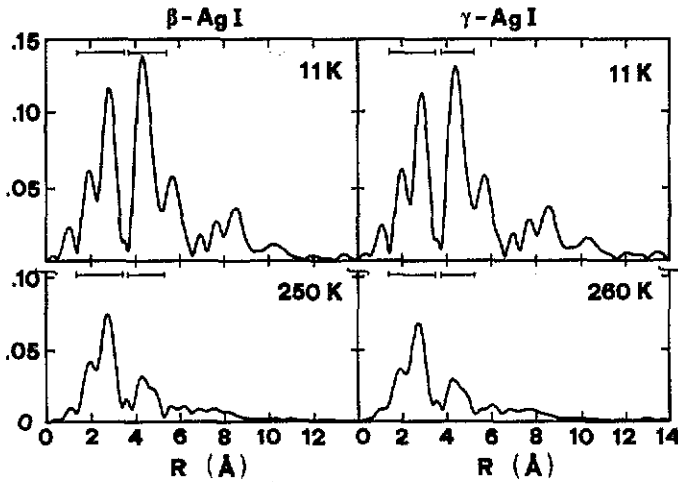


Figure 2. Fourier transforms of the EXAFS signals $k\chi(k)$ at the L_3 edge of iodine in β and γ -AgI at low and high temperature. $k\chi(k)$ signals have been transformed in the k range 2.3–8.1 \AA^{-1} using a Hanning window. The horizontal bars in the upper part of the figure represent the intervals of the inverse transform for the 1st and 2nd coordination shells of iodine.

where the index j labels the shells of scattering atoms surrounding the absorbing atom at distance R_j ; ϕ_j is the total phase shift experienced by the photoelectron. The amplitude A_j of the oscillations is described by

$$A_j = \frac{S_0^2 N_j f_j(k, \pi)}{k R_j^2} e^{-2R_j/\lambda} e^{-2\sigma_j^2(T)k^2} \quad (2)$$

where S_0^2 takes into account the intrinsic inelastic losses, $f(k)$ is the back-scattering amplitude, λ the mean free path of the photoelectron and N_j the j th shell coordination number. Atomic thermal vibrations cause a reduction of the amplitude of EXAFS signal. For small atomic displacements the temperature dependence of EXAFS signal is described by a Debye–Waller-like factor, $\exp(-2k^2\sigma_j^2)$, where σ_j^2 is the temperature-dependent mean square relative displacement (MSRD) of absorber and backscatterer atoms [15]. The MSRDs $\sigma^2(T)$ of AgI were determined using the amplitude ratio method [14]; the 11 K EXAFS spectra of β - and γ -AgI were used as reference for the analysis of the two phases at higher temperatures. The amplitude ratio curve for the first shell (distances I–Ag) could be fitted by a straight line up to about 8 \AA^{-1} in the whole temperature range 11–260 K. The analysis of the second shell (distances I–I) was performed by considering the k range of the spectra in which the amplitude ratio curve was linear; this k range progressively decreased with increasing temperature [12].

The $\Delta\sigma^2(T) = \sigma^2(T) - \sigma^2(11 \text{ K})$ so obtained were refined by a best fit of the whole $k\chi(k)$ signal. The experimental points $\sigma^2(T)$ are shown in figure 3 for the first two shells of iodine in β - and γ -AgI. The data in figure 3 have been vertically shifted to match at 11 K the theoretical values calculated by an Einstein model (*vide infra*). Three sets of measurements were analysed: the modulus of the maximum deviation from the mean value was $\leq 5\%$ for the first shell experimental points and $\leq 10\%$ for the second shell points. The slopes of $\sigma^2(T)$ curves are the same for the β and γ phases and coincide with those found for the mixed β - γ sample [12] in the range of overlap and within the experimental uncertainty.

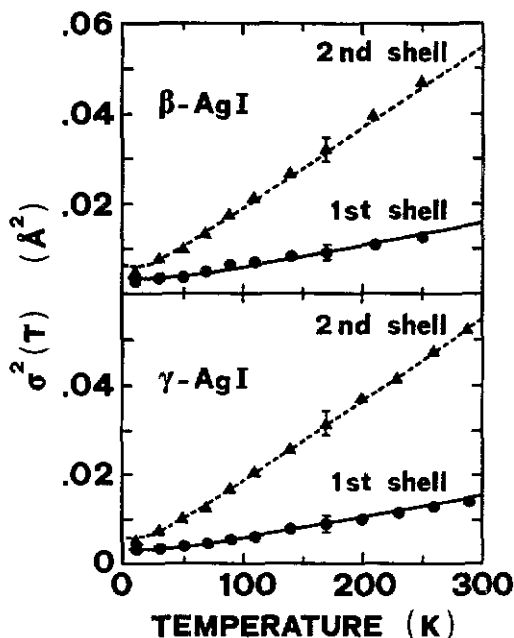


Figure 3. Experimental mean square relative displacements for the 1st (full circles) and 2nd (triangles) shell of I in β - and γ -AgI. The lines represent Einstein models for the 1st shell (full curve) and the 2nd shell (broken curve) which best fit the experimental data. The Einstein frequencies are respectively 2.65 and 1.35 THz. The experimental data have been upward shifted to match the absolute theoretical values at 11 K.

EXAFS formulae (1) and (2) utilized in our analysis are strictly valid only for narrow gaussian distributions of interatomic distances. However this is not the case of AgI which is known to present non-negligible anharmonicity effects even at relatively low temperatures [10, 16]. In the case of not-too-large disorder it was shown that EXAFS analysis can be performed through a cumulant expansion approach [17, 18]. The MSRD σ^2 corresponds to the 2nd order cumulant C_2 of the distribution of distances and the use of formula (2) is equivalent to truncating the cumulant expansion to the 2nd order term. Only if anharmonic contributions are very weak can the higher order cumulants be neglected. Anharmonicity contributes to the experimental damping of EXAFS amplitude with terms proportional to $k^n C_n$, where $n = 2, 4, 6, \dots$. The analysis of the I L_3 EXAFS amplitude was forced in terms of harmonic because the presence of a beating in the signal at 6 \AA^{-1} and the shortness of the k range ($k \leq 8.1 \text{ \AA}^{-1}$), due to the vicinity of the I L_2 edge, did not allow us to obtain reliable information on cumulants of order higher than the second. First-shell σ^2 values extracted on the basis of harmonic analysis of the I L_3 EXAFS are however in good agreement with the corresponding values obtained from a cumulant analysis of the EXAFS at the K edge of Ag [16]. On the grounds of this results, we also considered reasonable the analysis of the 2nd shell signal restricted to the linear part of the amplitude ratio. The reproducibility of the first shell results with changing of the absorption edge, namely with changing sample thickness, experimental conditions, and data reduction procedure, is a good check of the accuracy of results and allows a better understanding of the limits of the standard treatment of EXAFS data in the presence of anharmonicity [16].

Thanks to a wider k range Ag K EXAFS allows a deeper investigation of the anharmonicity of AgI than I L_3 EXAFS but, because of the joint effects of large core-hole width and large Ag–Ag DW factor, provides information only on the first coordination shell. I L_3 EXAFS has the advantage of carrying information about not only the nearest but also the next-nearest neighbours of I.

4. Discussion

From the comparison between the experimental data of β and γ -AgI we deduce that the MSRDS of the first two shells are equal, within the experimental uncertainties, for the two phases. In general the temperature dependence of MSRDS is compared with simple Einstein and Debye models [3, 12] or with more refined force-constant models. In what follows we will try to interpret the data in terms of some of these models.

In figure 3 an Einstein model has been fitted to the experimental data. The Einstein model, in spite of its simplicity, is able to give an estimate of the effective bond-stretching force constants. The Einstein frequencies for both β and γ phases of AgI are 2.65 THz for the first shell and 1.35 THz for the second one. The corresponding effective stretching force constants are 26 Nm^{-1} and 7 Nm^{-1} respectively. These results demonstrate that both AgI modifications, in spite of their different symmetry, show similar vibrational local properties. This fact is not surprising since the first two shells are equivalent for the two phases: four Ag atoms are tetrahedrally coordinated to I at a bonding distance of 2.81 Å and 12 I atoms surround the central I at 4.59 Å. Moreover the vibrational properties of both phases have been shown to be very similar on the basis of a valence shell model [8, 19]: the β and γ AgI phonon densities of states are almost equal and they are both characterized by two sharp peaks centred at about 0.5 and 3.3 THz and by an intermediate broad distribution due to longitudinal acoustical modes.

The MSRDS σ^2 (which gives the variance of the distance distribution) can be influenced by anharmonicity. The anharmonic contribution to MSRDS can be expressed as $\sigma_A^2 = \sigma^2 - \sigma_H^2$, i.e. the difference between the experimental value and the value σ_H^2 expected on the basis of harmonic approximation. In our case σ_H^2 is given by the Einstein model. For weak anharmonicity σ_A^2 can be calculated within the quasi-harmonic approximation. However for AgI the quasi-harmonic contribution in the range 11–300 K has been evaluated to be negligible due to the very low thermal expansion coefficient of AgI. The agreement between experimental values of σ^2 and the harmonic model of Einstein in figure 3 could lead us to consider σ_A^2 negligible with respect to σ_H^2 . However, this result, in the range 11–292 K, is valid only within the limits of experimental uncertainty. In fact, the temperature dependence of the fourth cumulant determined from Ag K EXAFS [16] indicates a gradual increase of anharmonicity already above liquid nitrogen temperature, in agreement with the previous Ag K EXAFS measurements by Boyce and *et al* [10]. The deviation of the DW factor from harmonic behaviour when approaching the superionic transition temperature was determined by Yoshiasa *et al* [11] who performed x-ray diffraction measurements on single β -AgI crystal. In table 1 the uncorrelated mean square displacements of the distance I–Ag, $\langle u_I^2 \rangle + \langle u_{Ag}^2 \rangle$, calculated from the β_{11} and β_{33} coefficients obtained by Yoshiasa *et al* on the basis of both a harmonic and an anharmonic analysis are reported. The difference between the two analyses is $\leq 7\%$ for temperatures ≤ 297 K. In the following discussion when we compare the experimental $\sigma^2(T)$ with the MSRDS

calculated on the grounds of some force-constant models of vibrational dynamics, we will neglect the anharmonic contribution in the range 11–292 K on the grounds that it is comparable to experimental uncertainty.

Table 1. $\langle u^2 \rangle_I + \langle u^2 \rangle_{Ag}$ represents the difference between the MSRDS of the distance I–Ag and the corresponding correlation term [15]. The uncorrelated mean square displacements $\langle u^2 \rangle$ for Ag and I in β -AgI have been calculated at different temperatures from the second-order x-ray diffraction temperature factors reported in reference [10]. The values are expressed in Å^2 .

T	Harmonic analysis $\langle u^2 \rangle_I + \langle u^2 \rangle_{Ag}$	Anharmonic analysis $\langle u^2 \rangle_I + \langle u^2 \rangle_{Ag}$
123	0.048	0.050
297	0.127	0.136
363	0.163	0.174
413	0.192	0.205

EXAFS DW factors are useful to discriminate among different lattice-dynamical models. It is well established that for a given compound different force-constant models can yield very different eigenvectors even though they correctly reproduce the dispersion relations of phonons [20, 21]. In fact, it has been shown that various refined models of the vibrational dynamics of a given binary compound, all best-fitting experimental phonon dispersion curves, can produce very different atomic mean square displacements [21]. A salient characteristic of the curves in figure 3 is the very different slope for the I–Ag and I–I distances; it is due to the very different correlation effects of atomic motion for the two distances [12]. For both β and γ modifications the comparison with the correlated Debye model [12] has been shown to be inadequate for interpreting the MSRDS of the second shell because, as in the case of the mixed compound, it does not take into account the contribution of the low-frequency phonon branch at 0.5 THz. In what follows we will consider separately each structural modification of AgI.

4.1. β -AgI

The interpretative scheme proposed for the temperature dependence of MSRDS in the mixed compound of reference [12] can be likewise applied to β -AgI. According to this scheme the $\sigma^2(T)$ of both I–Ag and I–I distances is reproduced satisfactorily by a hybrid correlated Einstein–Debye model, taking into account the eigenvectors of the low-frequency optical mode at 0.5 THz in the centre of BZ, calculated by Bührer *et al* [8, 19]. The main limitation of this approximation lies in the fact that it considers only the atomic displacement pattern of the low-frequency optical mode at the BZ centre. A rigorous comparison between experiment and theory requires knowledge of eigenfrequencies and eigenvectors of all vibrational modes over all the BZ. A convenient expression of MSRDS is given by equation (7) of reference [12]. The only theoretical calculation of vibrational dynamics that has been published for AgI [8] is based on a valence shell model. It allowed its authors to calculate mean square atomic displacements in good agreement with x-ray diffraction measurements. We calculated the MSRDS of the I–Ag and I–I distances using eigenvectors and eigenfrequencies of all modes calculated by Bührer *et al* by the valence shell model [18, 19] throughout

the entire BZ, but we did not achieve satisfactory agreement with experiment. In particular the strong correlation between I and Ag is not reproduced and the I-Ag MSRD is larger than the I-I MSRD. The disagreement, which progressively grows when considering outer points in the BZ, should be ascribed to a wrong evaluation of phase relationships between eigenvectors which, however, does not affect the uncorrelated mean square displacements. This fact stresses the uselessness of EXAFS as a vibrational-dynamics probe in view of the peculiar sensitivity of MSRD to the phase relationships between eigenvectors.

4.2. γ -AgI

There are only a few experimental and theoretical reports on γ -AgI because it occurs only in powdered form. To interpret the $\sigma^2(T)$ data of γ -AgI we have made reference to CuBr which has physical properties very similar to γ -AgI. CuBr has the same structure and almost the same ionicity as γ -AgI. Bührer *et al* [8] exploiting these similarities compared the calculated phonon dispersion curves for γ -AgI with the experimental phonon frequencies of CuBr. We have followed a similar procedure comparing the experimental $\sigma^2(T)$ for γ -AgI with the $\sigma^2(T)$ calculated for CuBr. The harmonic part of MSRD of CuBr was calculated by Tranquada and Rehr [22] by utilizing the shell model parameters which had been determined by Hoshino *et al* [23] by a least-squares fit to measured phonon dispersion relations. The $\sigma^2(T)$ calculated by Tranquada and Rehr for the first and second shell of Br in CuBr (as reported by Tranquada and Ingalls in figure 5 of reference [17]) are plotted in figure 4 and compared with the corresponding values calculated using Einstein models of frequency ν_{CuBr} . The frequencies ν_{CuBr} have been obtained from the frequencies ν_{AgI} that best fit the γ -AgI experimental data of figure 3 by applying the homology criterion of interatomic forces:

$$\frac{\nu_{\text{CuBr}}}{\nu_{\text{AgI}}} = \left(\frac{(M a^2)_{\text{AgI}}}{(M a^2)_{\text{CuBr}}} \right)^{1/2} \quad (3)$$

where M is the mass number and a the lattice constant of the specific compound [8]. The agreement between the Tranquada and Rehr calculations and the scaled MSRD model appears satisfactory for both shells. In a previous comparison between the x-ray diffraction DW factors measured for 17 materials with zinblende structure and the corresponding values calculated on the basis of several lattice-dynamical models, the shell model was found to best interpret the experimental results as well [24]. With respect to x-ray diffraction DW factors, EXAFS DW factors can be used as more stringent tests of lattice-dynamical models since they are sensitive not only to the magnitude of atomic eigenvectors but also to their phase relationship.

5. Conclusions

The EXAFS Debye-Waller factors of AgI in its non-superionic hexagonal and cubic modifications have been extracted from EXAFS at the L_3 edge of I from 11 to 292 K. From the $I L_3$ EXAFS the MSRDs of the first two distances I-Ag and I-I have temperature range. These results confirm and extend previous results for AgI samples in the mixed β - γ phase. The harmonic approximation, within the uncertainty of experimental data, has been found adequate to interpret MSRDs for temperatures lower than 300 K. Thus an Einstein model can be satisfactorily fitted to the experimental data.

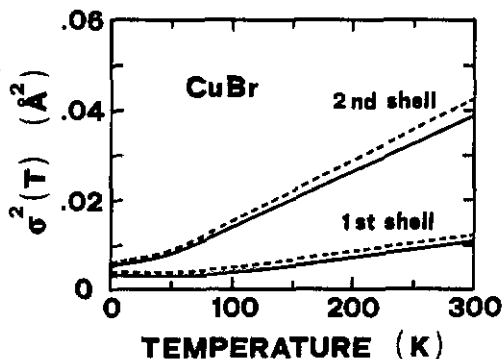


Figure 4. Temperature dependence of the mean square relative displacement for the 1st and 2nd shells of Br in CuBr (full curves) as shown in figure 5 of reference [17]. The broken curves represent Einstein models for CuBr with frequencies obtained from γ -AgI experimental data by means of the homology criterion.

The availability of AgI samples of pure β and γ phases has allowed us to compare the experimental MSRDS to those calculated by some theoretical models of vibrational dynamics.

We have found that while the experimental β -AgI MSRDS cannot be reproduced by the MSRDS calculated according to a valence shell model, the experimental temperature dependence of MSRDS in γ -AgI is in satisfactory agreement with the temperature dependence of MSRDS calculated using a shell model for CuBr, whose vibrational properties are equivalent to those of γ -AgI. The sensitivity of MSRDS to phase relationships between eigenvectors and the relative ease with which the Debye-Waller factors, as a function of temperature, can be extracted from EXAFS, encourages the use of this technique to test the force-constant models of vibrational dynamics of heteroatomic crystals.

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