

Correlated annealing of radiation defects in alkali halide crystals

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Abstract. The kinetics of the correlated annealing of pairs of neutral (F-H) and charged (α -I) Frenkel defects in alkali halides is treated theoretically, taking into account defect diffusion, annihilation at short distances, and elastic or Coulomb interactions. It is shown that elastic interactions considerably affect the annealing kinetics and survival probability even for defect pairs that are third or fourth neighbours in the crystalline lattice. A widely used description of the correlated annealing in terms of the pseudo-first-order reaction fails for close defects, yielding effective energies E^* which in fact differ essentially from the activation energy E_a of diffusion, even when it is corrected by an interaction energy. The effect of the defect initial distribution over relative distances is studied. It is demonstrated that pulsed creation of spatially well correlated defects can lead to the non-exponential decay of concentrations usually expected for an isothermal annealing. The theory developed is compared with actual experimental data for KBr. It is concluded that the several-stage annealing of defect concentrations often observed in thermostimulated experiments does not necessarily mean recombination of close Frenkel defects which differ in their initial distances.

1. Introduction

Low-temperature ionizing irradiation (x-rays, γ -rays, electron beam, etc) of alkali halides produces various types of defects. Among them of key importance are primary Frenkel pairs of F and H centres (a vacancy with an electron and an interstitial halide atom, respectively) which are created via the non-radiative decay of self-trapped excitons (STE) [1-4] and spatially are well correlated within so-called *geminate pairs*. Because of electron tunnelling between electron and hole components of close Frenkel pairs, charged Frenkel defects, i.e. α and I centres (an empty vacancy and an interstitial anion, respectively), arise also; in KBr irradiated at 10 K the ratio of the number of α -I Frenkel pairs to the number of F-H pairs may be about four or five [5, 6].

The stability of Frenkel pairs is controlled by I- and H-centre delocalization, stimulating subsequent recombination with α , F and F' (an F centre with a trapped electron) centres [7]. This recombination leads to the well known *multi-stage thermal annealing*, which was investigated by different methods: optical absorption [7-16], crystal volume change [8], release of the stored energy in the form of heat [17, 18], luminescence [4, 7, 11, 16, 19-23] and gas desorption [7, 11].

Considering KBr crystals as an experimentally well studied example, let us summarize briefly the most reproducible distinctive features of low-temperature annealing.

(i) Thermostimulated reorientation of H centres begins at 16 K [16]. For this reason the dichroism of preferentially aligned H centres was found to decrease abruptly.

(ii) As the motion of I centres is spatially correlated with α centres nearby, it is initiated at about 22 K [24]. Their recombination is not accompanied by a thermoluminescence (TL) peak.

(iii) Delocalization of isolated (non-correlated) I centres is observed at 27–30 K [4, 7, 11–13, 23]. It results in a strong TL peak with the spectrum exhibiting the π - and σ -polarized emission bands [15]. Several workers [15, 22, 25] interpreted the stage at 27–30 K as a process with first-order (monomolecular) kinetics due to close defect recombination within geminate F–H pairs (despite doubts expressed in [11]). According to the thermostimulated conductivity (TSC) data, the activation energy of this stage varies from 0.06 eV at low doses to 0.10 eV at higher doses [21].

(iv) The H-centre annealing in KBr was investigated in detail in [7–9, 11–16, 19, 26, 27]. It is shown to be accompanied by TL [7, 11] as in the case of a random (i.e. uncorrelated with α centres) distribution of I centres. As follows from the studies in [7], H-centre annealing leads to several TL peaks at temperatures T ranging from 32 to 60 K. It is generally accepted that the stage at 20–30 K is not related to mobile H centres but arises from secondary reactions initiated by migration of I centres [7, 11, 13, 14]. The H centres trapped by impurities or dimer centres (H_A , H_2 , ...) are not observed at this stage [14].

(v) Finally, above 35–45 K isolated H centres become mobile. The relevant activation energy is in the range $E_a = 0.11$ – 0.13 eV [7, 9, 11].

(vi) Following Ueta [26], the activation energy of free H-centre thermoactivated migration was accepted to be $E_a = 0.09$ eV. Later Zirap [7] suggested a slightly higher estimate of $E_a = 0.11$ – 0.13 eV. This energy corresponds to the F- and H-band annealing stage, at which simultaneously a di-H band arises and the most intensive gas desorption holds; this is caused by mobile H-centre migration to the crystalline surface [7].

In recent years, many theoretical papers have been dedicated to the different aspects of kinetics of correlated defect recombination in solutions [27], glasses and molecular solids [28–30] and crystals [31–36]. The peculiarity of defects in crystals requires taking into account the long-range elastic interaction between even neutral defects. It was Sonder [37] who first focused attention on the elastic interaction of close F and H defects as a key factor determining their survival probability and qualitatively estimated it.

In order to obtain quantitative results and to gain more insight into elementary secondary reactions in which particular F–H and α –I pairs in alkali halides are involved, Monte Carlo computer simulations were undertaken [5, 38, 39]. They estimated an initial F–H distribution function over distances at the moment of H-centre thermalization, considered the mechanism of formation and motion of dynamical crowdions arising when H centres trap an electron and modelled thermoactivated annealing of Frenkel pairs stimulated by their dynamical interactions.

In section 2 of this paper, in line with [32–34] we consider theoretically the kinetics of correlated F–H and α –I annealing in order to suggest the correct formalism necessary for interpreting actual experimental data. In particular, we demonstrate the importance of elastic and Coulomb interactions. From the results of our calculations in section 3 we suggest that caution is required in interpreting many-stage annealing kinetics and, moreover, the effective pseudo-monomolecular activation energies. The

role of tunnelling recombination in the recombination kinetics of correlated F and H centres is discussed briefly in the conclusion.

2. Theoretical details

The energy of an elastic (dipole-dipole) interaction between point lattice defects is [40, 41]

$$\varphi(r) = \alpha(\theta, \phi)/r^3 \quad (2.1)$$

i.e. anisotropic. The actual form of $\alpha(\theta, \phi)$ is defined by the symmetry of defects. If both defects are isotropic (like the F centre) but the crystal is weakly anisotropic, the parameter α is simply proportional to the cubic harmonics [α_4], whereas for anisotropic defects the angular dependence of α cannot be presented analytically [42]. The H centre under study is an example of a strongly anisotropic defect; it is an X_2^- molecule occupying a single anion site [43]. However, since it begins to rotate at a much lower temperature than that required for hopping, we can assume confidently that H centres always strive for an energetically profitable position and thus one could perform upper-bound estimates considering the case of isotropic attraction with a constant α corresponding to the relative orientation with maximum attraction between F and H centres. According to the calculations [44], $\alpha_{\max} \simeq -3 \text{ eV } \text{\AA}^3$ in KBr.

Our model, which is treated in detail below, assumes that the H defect created at a given distance l from its complementary F centre undergoes random walks and annihilates it when approaching to within a certain small distance R_0 (the so-called *clear-cut reaction radius*). By fitting the results of our continuous model to computer simulations of random walks on a discrete lattice [38], we obtained $R_0 \simeq 3 \text{ \AA}$ in KBr, very close to the interionic spacing. Incorporation of the elastic attraction acts to lower the energy barriers for H-centre hopping in the direction of the F centre and thus stimulates their annihilation.

The annealing kinetics of defects in geminate pairs could be described through joint correlation functions of dissimilar defects [32], giving the probability density of finding an F-H pair at a relative distance r . For an arbitrary defect interaction $\varphi(r)$ it is given by [45]

$$\partial Y(r, t)/\partial t = D[\Delta Y + (1/kT) \nabla(Y \nabla \varphi)] \quad (2.2)$$

$$Y(r \leq R_0, t) = 0 \quad Y(r, 0) = \delta(r - l)/4\pi l^2. \quad (2.3)$$

Here D is the diffusion coefficient given by

$$D = D_0 \exp(-E_a/kT) \quad (2.4)$$

E_a is the incoherent hopping activation energy, T is the temperature and D_0 is the pre-exponential factor. The use of the continuous approximation for diffusion is justified for close defects from the results of the study in [31].

The proportion of defects recombined up to time t is given by the flux through the annihilation sphere:

$$S(t) = \frac{C}{C_0} = \int_0^t dt' 4\pi R_0^2 D \frac{\partial Y}{\partial r'} \Big|_{r=R_0}. \quad (2.5)$$

In the particular case of elastic interaction (equation (2.1)), after substitution of $Y = y/r$ we find that equation (2.2) reads

$$\partial y / \partial t = D(y'' - 3gy'/r^4 + 9gy/r^5) \quad (2.6)$$

where $g = \alpha/kT < 0$.

The survival probability $S(t \rightarrow \infty)$ of a geminate pair (i.e. the proportion of H centres which after prolonged walks left their complementary F centres without recombination) was found in [32] to be

$$P_s = 1 - (R_0/l) \{ [\exp(-g/l^3) \phi(1, \frac{4}{3}, g/l^3)] / [\exp(-g/l^3) \phi(1, \frac{4}{3}, g/R_0^3)] \} \quad (2.7)$$

where ϕ is the degenerate hypergeometrical function.

At low temperatures, when $|g| \gg l^3$, R_0^3 we have

$$P_s = [l^2 \exp(-g/l^3) - R_0^2 \exp(-g/R_0^3)] / \Gamma(\frac{1}{3}) g^{2/3} \quad (2.8)$$

where Γ is Euler's gamma function. Finally, if $l \gg R_0$, we arrive at

$$P_s = \exp(-g/l^3) [l^2 / \Gamma(\frac{1}{3}) g^{2/3}]. \quad (2.9)$$

This shows that at low temperatures the F-centre accumulation efficiency, which is proportional to P_s , can be described by an Arrhenius law with an effective energy $E^* = \varphi(l)$ which thus equals the energy of elastic interaction at the moment of a pair creation—exactly as suggested by Sonder [37]. However, at sufficiently high temperatures, as $g \ll l^3$, we arrive at another limiting case [31]:

$$P_s = 1 - R_{\text{eff}}/l \quad (2.10)$$

widely used in the radiation physics of metals.

Equation (2.10) contains the effective radius of an annihilation stimulated by an elastic interaction [31, 32]:

$$R_{\text{eff}} = R_0 \exp(-g/R_0^3) / \phi(1, \frac{4}{3}, g/R_0^3) \\ = \begin{cases} R_0 & g \ll R_0^3 \\ 3g^{1/3} / \Gamma(\frac{1}{3}) \simeq 1.12(\alpha/kT)^{1/3} & g \gg R_0^3 \end{cases} \quad (2.11)$$

It has been shown [31] that, for an arbitrary potential $\varphi = \alpha/r^m$, equation (2.10) holds, as $|g| \ll l^m$. If $|g| < l^3$, equation (2.10) should be slightly modified [32].

Finally, for charged defects (α -I pairs), we arrive at [32]

$$P_s = \exp(-L/2l) \sinh[L(1/2R_0 - 1/2l)] / \sinh(L/2R_0) \\ L = e_1 e_2 / \epsilon kT > 0. \quad (2.12)$$

Here L is the so-called *Onsager radius* [45].

Again, as $L \gg R_0$ and $l \gg R_0$, P_s can be described by the Arrhenius law but with an effective energy equal to that of the Coulomb interaction: $E = e^2/\epsilon l$. Since for alkali halides at $T \simeq 100$ K the Onsager radius typically exceeds 50 Å, only initially well separated α -I pairs have a non-vanishing chance of survival.

The mechanism of α -I production was presented by Lisitsyn and Sigimov [38]. The trapping of an electron by an H centre after its tunnelling transfer from an F centre results in a strong Coulomb repulsion of two close X^- anions. This leads to the creation of a dynamical crowdion which can be separated from a vacancy behind by four to five interionic spacings.

There are two kinds of experiment that can be carried out:

(i) pulse (10^{-9} – 10^{-6} s) studies when the defect absorption (defined by a relative concentration C of short-lived defects) or recombination intensity ($J \propto dC/dt$) is monitored at a given temperature;

(ii) stationary thermoactivated studies in which the sample temperature increases linearly: $T = T_0 + \beta t$, where β is the heating rate, thus stimulating migration of otherwise stable defects (see equation (2.4)), the recombination of which occurs typically on a time scale of minutes.

The idea is widespread that the latter kinetics of correlated recombination could be described in terms of a pseudo-first-order process [9, 46]:

$$dC/dt = -KC \quad (2.13)$$

with the reaction rate $K \propto \exp(-E^*/kT)$. The effective activation energy E^* was expected to coincide either with the diffusion activation energy E_a [46] or with E_a corrected by the energy of elastic interaction [9, 37]. If this were the case, the plot of $\ln(C^{-1} dC/dt)$ versus $1/kT$ should give a straight line with a slope equal to E^* .

On the other hand, it is clear that equation (2.13) is only an approximation since $C(t) \simeq \text{constant}$ at both short and long times (the defects have just started to approach each other to recombine or those which survived have already separated at a large distance).

3. Results and discussion

The theoretically calculated decay of the F–H concentration during the linear heating of KBr is plotted in figure 1 for two different initial distances corresponding to the first-nearest neighbours (1NNS) along the $\langle 110 \rangle$ axis (the direction in which the STE decays into defects [1–3]) and to the second-nearest neighbours (2NNS) along the $\langle 100 \rangle$ axis. The obvious conclusion to be drawn is that the survival probability depends markedly on the initial separation between defects but even more on the strength of the elastic interaction. A plot of these kinetics in the above-discussed coordinates ($\ln(C^{-1} dC/dt)$ versus $1/kT$) yields the effective energies E^* given in table 1. When the elastic interaction is neglected, we have $E^* < E_a$ whereas, if it is incorporated, $E^* > E_a$. This latter fact contradicts the intuitive idea [9] that $E^* \simeq E_a - |\alpha|/l^3$. Note that the incorporation of an elastic interaction greatly reduces the survival probability for third-nearest neighbours (3NNS): from 80% (no interaction) down to 20% (when interaction is taken into account).

Table 1. Estimates of the effective energies E^* of F–H annealing in KBr based on a model of pseudo-first-order kinetics.

Elastic constant α (eV \AA^{-3})	Effective energy E^* (eV) for the following initial separations l		
	$l = 4.2 \text{ \AA}$ (1NNS)	$l = 6.0 \text{ \AA}$ (2NNS)	$l = 8.5 \text{ \AA}$ (3NNS)
0	0.06	0.08	0.09
-3	0.17	0.12	0.10

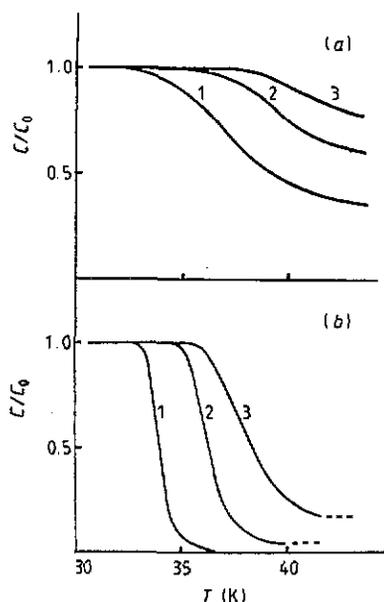


Figure 1. The calculated kinetics of diffusion-controlled correlated annealing of F-H pairs in KBr (a) neglecting and (b) taking into account their elastic interaction for the following initial relative distances between defects: curve 1, 4.2 Å (1NNs); curve 2, 6 Å (2NNs); curve 3, 8.5 Å (3NNs). The survival probability is shown in (b) by a broken line. The parameters used were $\beta = 0.1 \text{ K s}^{-1}$, $E_a = 0.09 \text{ eV}$, $\alpha = -3 \text{ eV Å}^3$, $R_0 = 3 \text{ Å}$ and $D_0 = 1.5 \times 10^{11} \text{ Å}^2 \text{ s}^{-1}$ [34].

In both cases ($\alpha = 0$ and $\alpha < 0$) E^* approaches E_a as l increases. We also simulated the case of weakly correlated defects considering a quasi-uniform distribution within the separation interval $l = 15\text{--}55 \text{ Å}$. Here the value of E^* obtained (0.086 eV) is indeed very close to the given E_a (0.09 eV).

It should be stressed once more that the survival probabilities P_s (shown by the broken lines in figure 1(b)) are directly related to the elastic interaction energy at the initial (geminate) pair separation. For 2NNs ($l \simeq 6 \text{ Å}$), $E^* = 0.015 \text{ eV}$ which according to equation (2.9) yields, at 40 K, $P_s \simeq 0.05$. This survival probability simply defines the proportion of defects that could be accumulated in a crystal after its irradiation. It was found experimentally [47] that for low doses the F-centre accumulation efficiency in KBr could indeed be approximated by the Arrhenius law with $E^* \simeq 0.017 \text{ eV}$ (instead of the generally accepted value of 0.03 eV [43] observed at higher doses), thus arguing for preferential F- and H-centre creation at a 2NN distance (see also [48] and the discussion below).

Since actual distribution functions can involve several kinds of pairs separated by different distances [4, 39] we have simulated this effect in figure 2. (In all the cases considered, the partial defect concentrations at different distances were taken to be the same, thus giving after summation unity (i.e. C_0), irrespective of the number of lattice sites in the relevant positions.)

The step structure for the simultaneous 1NN and 2NN defect annealing (curve 1) is more pronounced than for the simultaneous 2NN and 3NN annealing (curve 2). Note that the smooth curve 2 lies just between two curves corresponding to 2NNs and 3NNs only (i.e. curves 2 and 3 in figure 1). For fourth-nearest neighbours (4NNs) and fifth-nearest neighbours (5NNs) (figure 2(b)) the decay is very smooth and the survival probability high.

Finally the case of simultaneous decay concentrations of *all five* kinds of close defect in equal proportions is presented in figure 2(c). As can be easily seen, the annealing kinetics reveal a weakening structure up to three to four neighbours, but more separated defects could hardly be resolved. This is in agreement with the

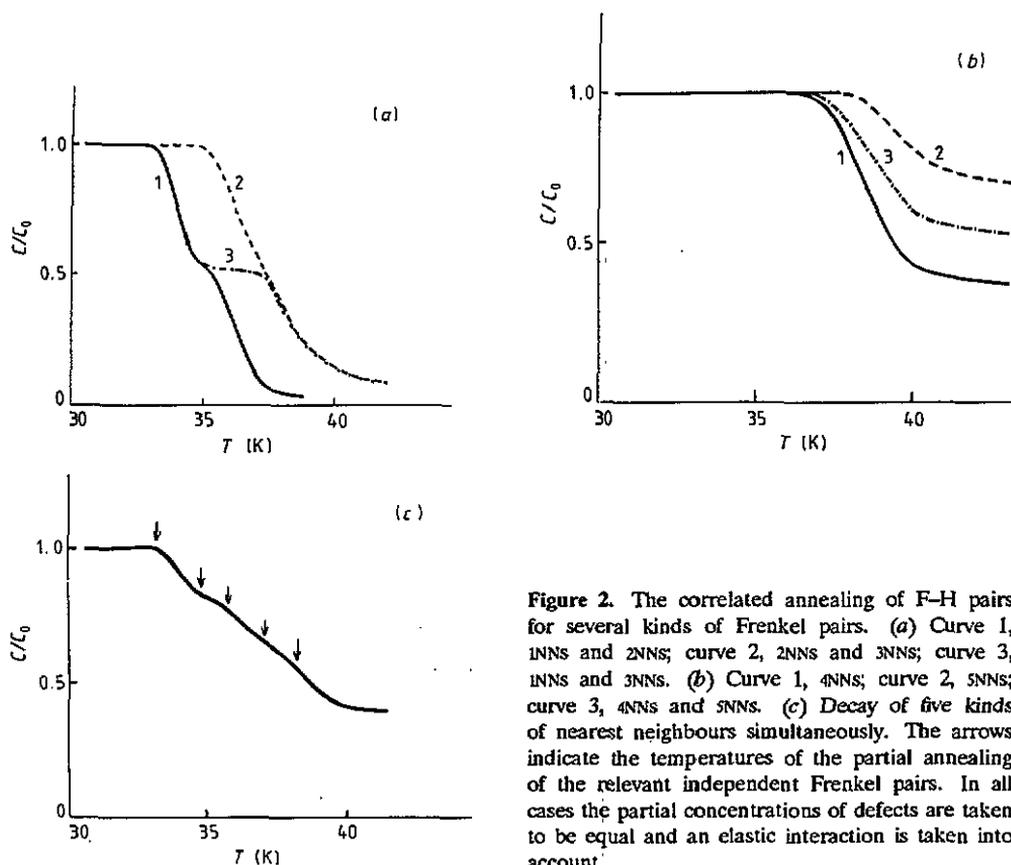


Figure 2. The correlated annealing of F-H pairs for several kinds of Frenkel pairs. (a) Curve 1, 1NNS and 2NNS; curve 2, 2NNS and 3NNS; curve 3, 1NNS and 3NNS. (b) Curve 1, 4NNS; curve 2, 5NNS; curve 3, 4NNS and 5NNS. (c) Decay of five kinds of nearest neighbours simultaneously. The arrows indicate the temperatures of the partial annealing of the relevant independent Frenkel pairs. In all cases the partial concentrations of defects are taken to be equal and an elastic interaction is taken into account.

conclusion in [39], putting the critical distance at (400) neighbours.

The calculated kinetics of isothermal decay of different kinds of F-H pairs show that the decay kinetics cannot be perfectly described in terms of pseudo-first-order kinetics (equation (2.13)), even for a single defect separation l . On the other hand, this means that 'successful' decomposition of the decay kinetics into several exponentials does not give final evidence for the existence of several recombination channels (e.g. several initial defect separations).

Figure 3 gives the results obtained for oppositely charged Frenkel defects: α and I centres.

As follows from equation (2.12), only well separated defects ($r > 30$ Å) have a real chance of survival. Under such conditions a step structure on the annealing curves might be observed only if there existed several groups of I centres which strongly differ in their distances (figure 3(c)). In order to distinguish between the kinetics of monomolecular recombination of geminate pairs and bimolecular recombination of defects belonging to different pairs (e.g. when pairs become mixed owing to diffusion), a plot of $\log(dC/dt)$ versus $\log t$ could be useful [9]. From the point of view of standard bimolecular kinetics [45] we have

$$I \propto dC/dt = -K(t)C^2 \quad (3.1)$$

with the reaction rate $K(t) = 4\pi DR_0(1 + R_0/\sqrt{\pi Dt})$.

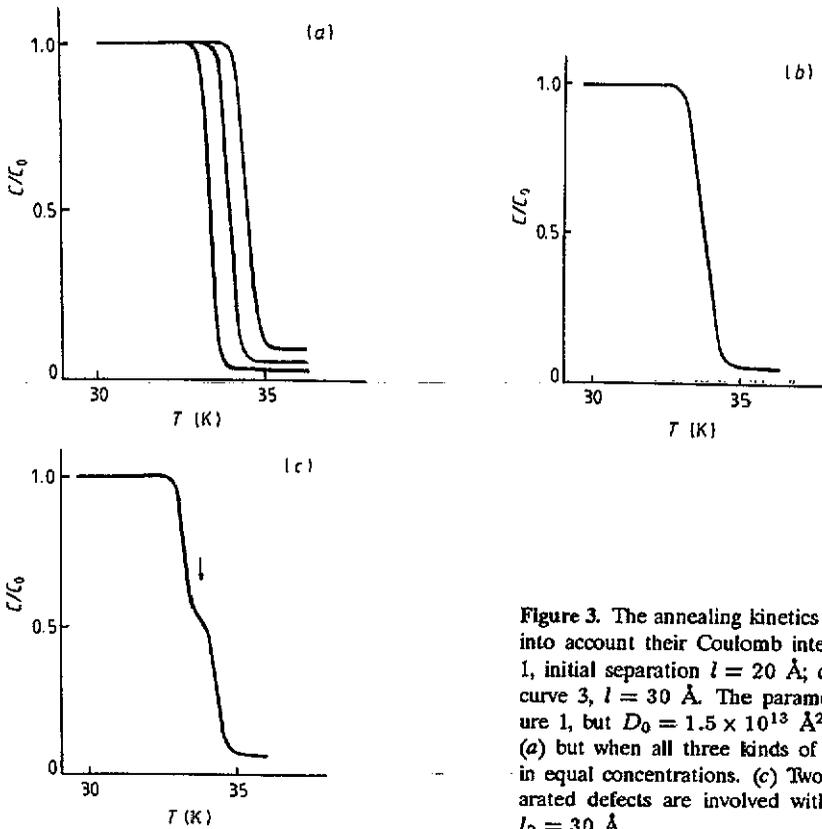


Figure 3. The annealing kinetics of α -I pairs taking into account their Coulomb interaction. (a) Curve 1, initial separation $l = 20$ Å; curve 2, $l = 25$ Å; curve 3, $l = 30$ Å. The parameters are as in figure 1, but $D_0 = 1.5 \times 10^{13}$ Å² s⁻¹. (b) Same as (a) but when all three kinds of defect are present in equal concentrations. (c) Two kinds of well separated defects are involved with $l_1 = 20$ Å and $l_2 = 30$ Å.

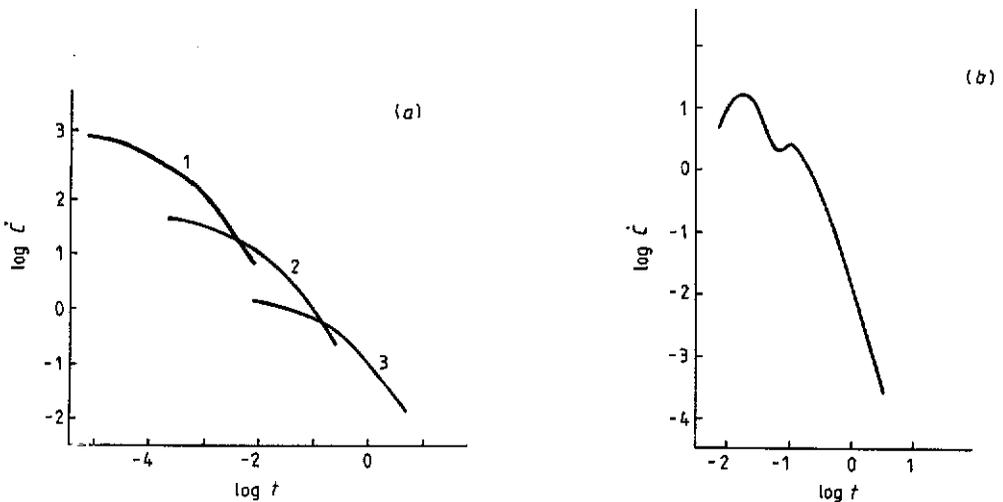


Figure 4. (a) Kinetics of isothermal decay of F-H centres at 60 K (curve 1), 50 K (curve 2) and 43 K (curve 3). The initial distribution consists of 1NNs and 2NNs (in equal concentrations). The elastic interaction is neglected. (b) The same as curve 1 in (a) but taking into account the elastic interaction.

As $t \rightarrow \infty$, equation (3.1) yields $C \propto t^{-1}$ (or $I \propto t^{-2}$). That is the plot in the coordinates of $\log J$ versus $\log t$ yields a straight line. Moreover, this curve has to decay more steeply at short times; this has indeed been observed experimentally more than once in different materials [49, 50]. However, in the case of geminate pairs the kinetic curves reveal the opposite effect, as shown in figure 4(a); the initial decay stage typically is quite flat. The incorporation of the elastic interaction permits one to resolve efficiently different kinds of close defect (if any) (figure 4(b)).

All the above supports the same conclusion: multi-stage annealing kinetics could be observed in alkali halides for very close defects only, typically no more than 4NNS. However, it is well known from pulse experiments [1-4, 51-53] that close F-H pairs in KBr (as well as in other alkali halides) disappear rapidly because of electron tunnelling from the electron to the hole centre.

As this process is temperature independent, at liquid-helium temperature it is the only recombination channel. Typical lifetimes observed for the three kinds of nearest F-H pairs are 0.3 μs , 15 μs and 70 μs [51]. (Shorter and longer lifetimes have also been reported; see, e.g., [52, 53].) This means that defects still existing on the time scale of the steady-state experiments at any rate cannot be close.

Therefore, we suggest that the many-stage kinetics originate from a set of secondary reactions between Frenkel defects rather than from their correlated annealing.

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