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ON THE RELATIONSHIP BETWEEN STORED ENERGY AND COLLOIDAL SODIUM PRODUCTION IN IRRADIATED ROCK SALT

H. Donker, W.J. Soppe and A. García Celma

ABSTRACT

A few parameters of the theoretical models used to describe the formation of radiation damage in rock salt were critically reviewed. It is discussed that the back reaction used in the models should be described as $\gamma = 10^{16} \exp(-0.4/kT)$ and that for the conversion factor between defect concentrations and stored energy a value of 5 eV/F–H pair should be used. With these modified parameters the models were compared with the experimental results of Jenks and Bopp and the experimental results obtained from the GIF A irradiation experiments.

1. INTRODUCTION

The first model describing the formation of radiation damage in alkali halides was developed by Jain and Lidiard [1977]. This model was later modified by Van Opbroek and den Hartog [1985], according to a proposal of Lidiard [1979]. This modification, the inclusion of a back reaction, was introduced in order to be able to explain the experimental results of Jenks and Bopp [1974;1977] and Jenks et al. [1975].

A disadvantage of this model is that it does not describe the nucleation stage of the colloids and dislocation loops. Moreover, the effects of impurities, strain and grain boundaries are not taken into account. Recently two new, slightly different models based on the old Jain–Lidiard model, but with extensions describing the nucleation stage have been developed [Soppe, 1993; Seinen et al.,1992].

In a recent paper [Soppe et al., 1994] the experimental results on radiation damage

formation in NaCl and the theoretical models used to describe this process were reviewed. Since then the availability of new experimental evidence and a thorough study of the literature has convinced us that a few parameters used in the models have to be modified. These modifications will be discussed below. A comparison with computer simulations made with the modified Jain–Lidiard model [Opbroek and den Hartog, 1985] and the model of Soppe [1993] using these modified parameters with the experimental results of Jenks and Bopp [1974; 1977] and Jenks et al. [1975] and our experimental data obtained for heavily irradiated Asse speisesalz samples [Donker and Garcia Celma, 1995] will be made.

2. THE BACK REACTION

In the modified Jain–Lidiard model [van Opbroek and den Hartog, 1985] as well as in the model of Soppe [1993] the probability for the back reaction between the F–centres and the Cl_2 molecules is described by:

$$\gamma = \gamma_0 \exp\left(\frac{-E_\gamma}{kT}\right) \qquad (1)$$

The assumed values for the pre–exponential factor γ_0 used in these models are $3\ 10^{18}$ and 10^{18} cm^{-2} respectively, and for the activation energy E_γ values of 0.6 and 0.55 eV were used.

Reasons can be given that the values used in both models for the pre–exponential factor are two orders of magnitude to high. γ_0 should be of the order of 10^{16} cm^{-2} which is a normal value for reactions between point defects [Seinen, private communication].

The values used for the activation energy E_γ are not known with great accuracy. Soppe obtained 0.55 eV from fitting his model to the experimental data of Jenks and Bopp, while Lidiard based the 0.6 eV on the annealing experiments of these authors, who derived an overall activation energy E_a of 1.7 eV for the back reaction. Seinen [1994], however, has measured the activation energy of the back reaction on doped samples and reported values for this energy of 1.2, 1.5 and 1.7 eV depending on the method. Since the origin of the difference between the various measurements is not yet clear, we will use their average value i.e. 1.5 eV.

In the models, the back reaction is assumed to proceed via evaporation of F–centres from the colloids which then diffuse to and recombine with the molecular Cl_2 centres. This last, recombination step is assumed to be rate determining. This leads to a first order back reaction which can be described by :

List of symbols : page 411
a = Na
f = f-center
(p. 411)

$$\frac{dc_a}{dt} = -c_a A \exp\left(\frac{-E_a}{kT}\right) = -c_a \gamma D_F c_F^{(e)} \qquad (2)$$

If the F–centre diffusion coefficient (D_F) is 0.01 exp(–0.8/kT) and the mole fraction of F–centres in equilibrium with the colloids ($c_F^{(e)}$) is $2\ 10^{-4}$ exp(–0.3/kT), an overall activation energy (E_a) of 1.5 eV implies that E_γ would be 0.4 eV.

In the theoretical models we will assume that the back reaction is first order and that, regarding the discussion above, the probability of the back reaction can be described as $\gamma = 10^{16} \exp(-0.4/kT)$.

3. THE CONVERSION FACTOR BETWEEN DEFECT CONCENTRATIONS AND STORED ENERGY

A problem when comparing the theoretical models with the experimental results is the conversion factor which relates concentrations of defects with stored energy. There is no scientific agreement on the magnitude of this factor. The reported experimental results vary from 4.6 to 12.4 eV per F–H pair corresponding to 75 to 205 J/g per mol% defects. Hughes [1978] gives a theoretical value for this factor of 5.3 eV per F–H pair for free F–centres and 5 eV per F–H pair when the F–centres have agglomerated into colloids. Since there is disagreement among the reported results, we will critically review them and try to understand where the origin of the differences lies. The values given by the various scientists are summarized in Table 1.

Table 1 *Stored energy per F–H pair obtained by various scientists*

Authors	Value (eV/F–H pair)	Experimental techniques	Maximal defect concentration (F–H pairs/cm^3)
Hughes [1978]	5 – 5.3	Theoretical	
Phelps and Pearlstein [1962]	9.2 ± 0.3	L.A., solution calorimetry	$7\ 10^{17}$
Bunch and Pearlstein [1969]	12.4 ± 0.3	L.A., D.T.A.	$2\ 10^{18}$
Jenks et al. [1975]	4.6	drop calorimetry, H_2 and OCl^- measurements	$2\ 10^{20}$
Delgado and Alvarez Rivas [1979; 1980]	10	L.A., D.T.A.	$4\ 10^{18}$
Groote and Weerkamp [1990]	7.3 ± 0.7	L.A., D.S.C.	10^{20}
Groote and Weerkamp [1990]	9.4	D.S.C. (latent heat – stored energy)	10^{21}
Seinen [1994]	6.2 ± 1	L.A., D.S.C.	10^{20}
Seinen [1994]	5.3 ± 0.5	D.S.C. (latent heat – stored energy)	10^{21}

In his derivation of the theoretical value of the conversion factor Hughes neglected any contribution from the line energy of the dislocation loops. As will be shown below, this approximation is only valid for high damage levels i.e. when the loops are relatively large. The elastic energy of a prismatic dislocation loop (E_l) of radius R can be given by [Hirth and Lothe, 1968]:

$$E_l = \frac{Gb^2R}{2(1-\nu)}\left[\ln\left(\frac{32R}{b}\right) - 1\right] \qquad (3)$$

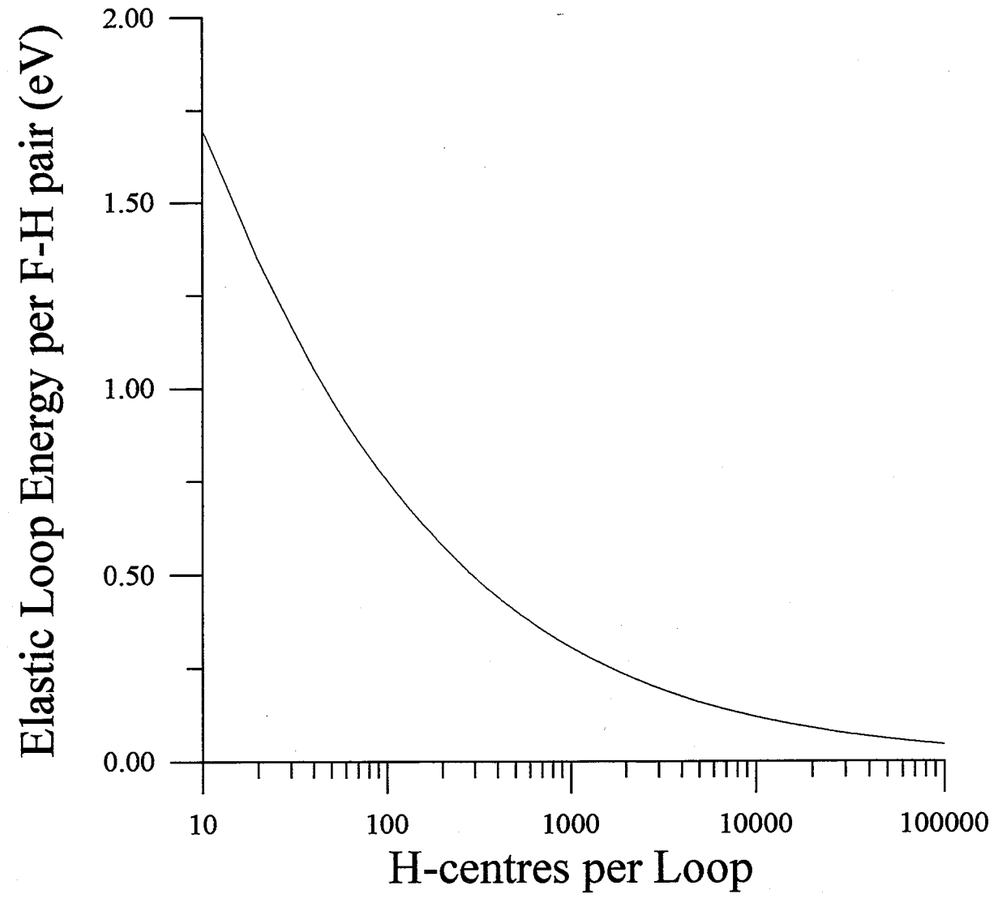

Figure 1: *Contribution of the elastic loop energy to the stored energy per F–H pair (E_l/N) as a function of the number of H–centres per dislocation loop (N).*

where G is the shear modulus (0.15 Mbar [Simmons and Wang, 1971]), ν is Poisson's ratio (0.25 [Simmons and Wang, 1971]) and b the burgers vector of the dislocation ($3.98\ 10^{-8}$ cm). The radius of the loop will depend on the number of H–centres condensed in the loop (N) and can be approximated by:

$$R = \sqrt{\frac{N}{\sqrt{2}\ \pi}}\ b \qquad (4)$$

This means that the contribution of the elastic loop energy to the stored energy per F–H pair (E_l/N) will depend on the number of H–centres in the loop (N) as:

$$\frac{E_l}{N} = \frac{Gb^3\sqrt{\frac{1}{\sqrt{2}\,\pi}}}{2(1-\nu)\sqrt{N}}\left[\ln\left(32\sqrt{\frac{N}{\sqrt{2}\,\pi}}\right) - 1\right] \qquad (5)$$

This dependence is shown in Fig. 1. In this figure it can be seen that the contribution of the elastic energy of the dislocation loops to the stored energy per F–H pair is only negligible when there are more then 1000 H–centres per loop. In the Jain–Lidiard model 10^{15} loops/cm^3 are assumed based on electron microscopy observations of Hobbs. This means that for pure NaCl the contribution of the elastic loop energy is only negligible when the defect concentration is above 10^{18} defects/cm^3. As can be seen from Table 1 this is the case for most of the experiments except those of Phelps and Pearlstein. The experiments of Groote and Weerkamp and Seinen however, have not only been performed on pure but also on doped NaCl samples. According to Hobbs [1973] doping of alkali halides with interstitial trapping impurities leads to an increase in loop density and a decrease of loop size. In the recently published extended versions of the Jain–Lidiard model [Soppe, 1993; Seinen et al., 1992] it is predicted that in doped samples the concentration of dislocation loops can be as high as 10^{17} loops/cm^3 . Therefore, it has to be kept in mind that in the derivation of the conversion factor by Groote and Weerkamp and by Seinen from their light absorption measurements a small contribution of dislocation loop elastic energy might have influenced the resulting conversion factor.

The value of 9.2 eV/F–H pair reported by Phelps and Pearlstein contains a correction term of 3.6 eV/F–H pair based on the assumption that on solution of their samples H_2 would be produced. De las Cuevas et al. [1992] and Garcia Celma et al. [1993] however, have shown that on solution, of irradiated NaCl containing only F centres and no colloids (as is the case in the experiments of Phelps and Pearlstein), no significant amounts of H_2 are produced. The measurements of Phelps and Pearlstein thus lead to a stored energy per defect of 5.6 eV. As stated in the previous paragraph this value also might contain a small contribution from the dislocation loops.

The results obtained by Groote and Weerkamp and those obtained by Seinen are based on the same measurements. The fact that the results of Groote and Weerkamp are higher than those given by Seinen is due to the fact that Groote and Weerkamp made a few errors which have been corrected by Seinen. For instance, a value of 0.0493 eV for the atomic specific heat of melting of sodium was used by Groote and Weerkamp to derive the value of 9.4 eV/F–H pair from their latent heat measurements . The enthalpy of fusion for bulk sodium is 2.598 kJ/mol [Cordfunke and Konings, 1990] leading to a specific atomic heat of 0.027 eV. Using the latter value, Seinen obtained his value of 5.3 eV/F–H pair. On analysing their L.A. measurements Groote and Weerkamp assumed a 50% lorentzian and 50% gaussian line shape for the colloid band. According to Doyle [1958] the colloid band has an approximately lorentzian line shape. Using a lorentzian line shape Seinen obtained a stored energy per F–H pair of 6.2 eV. However, it is unclear in both thesis whether pure or doped samples were used for the L.A. measurements. As stated above if doped samples have been used a small contribution of dislocation loop elastic energy might have influenced the result.

The samples of Phelps and Pearlstein, Bunch and Pearlstein and those of Delgado and Alvarez Rivas were irradiated at room temperature, whereas the samples of the other authors were irradiated at temperatures around 100 °C. In samples irradiated at room temperature only F–centres and no colloids will be present. The difference between the stored energy per F–H pair for free F–centres and F–centres agglomerated into colloids is however, expected to be small (0.3 eV) [Hughes, 1978]

Concluding we can say that all reported values for the conversion factor are approximately equal to the theoretical value of 5 eV/F–H pair except for the values of 12.4 eV/F–H pair reported by Bunch and Pearlstein and 10 eV/F–H pair by Delgado and Alvarez Rivas, which are about twice as large as the other values. Bunch and Pearlstein, however, have measured three stored energy peaks for their irradiated samples. If we only consider the peak at 250 °C, which is ascribed to the anneal of colloids their stored energy per F–H pair amounts to 8.4 eV, which, although still higher, is much closer to the theoretical value of 5 eV/F–H pair. The value reported by Delgado and Alvarez Rivas might be rather inaccurate due to the fact that the F–centre concentration was not determined from the maximum of the F absorption band but calculated from the optical absorption at several wavelengths on the high energy edge of this band. If their reference spectrum contained another absorption at these wavelengths, this would lead to an

overestimation of the result.

The values reported by Bunch and Pearlstein and Delgado and Alvarez Rivas are larger than the energy of lowest exciton state 7.9 eV (and even larger than the bandgap) of NaCl and are therefore inconsistent with the now generally accepted excitonic formation mechanism of the F and H centres [Itoh, 1982]. The high values reported by Bunch and Pearlstein and Delgado and Alvarez Rivas can only be explained by the presence of some other kind of defects than those mentioned in the introduction of this paper. We have not found convincing experimental evidence for the existence of other defects than F and H centres and their related agglomerates. Therefore, it is not clear why Bunch and Pearlstein and Delgado and Alvarez Rivas found such high values for the conversion factor. We, however, conclude that for the conversion of concentration of defects into stored energy a value of about 5 eV/F–H pair should be used.

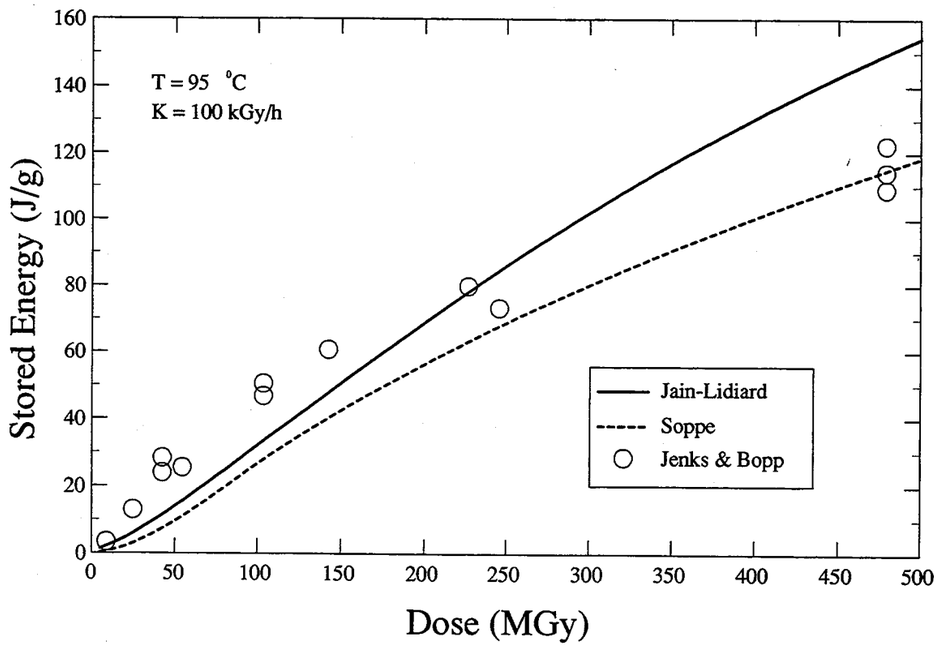

Figure 2: *Comparison of predicted stored energy values according to the Jain–Lidiard model and according to Soppe's model with experimental results obtained by Jenks and Bopp at 95 °C. Dose rate 100 kGy/h.*

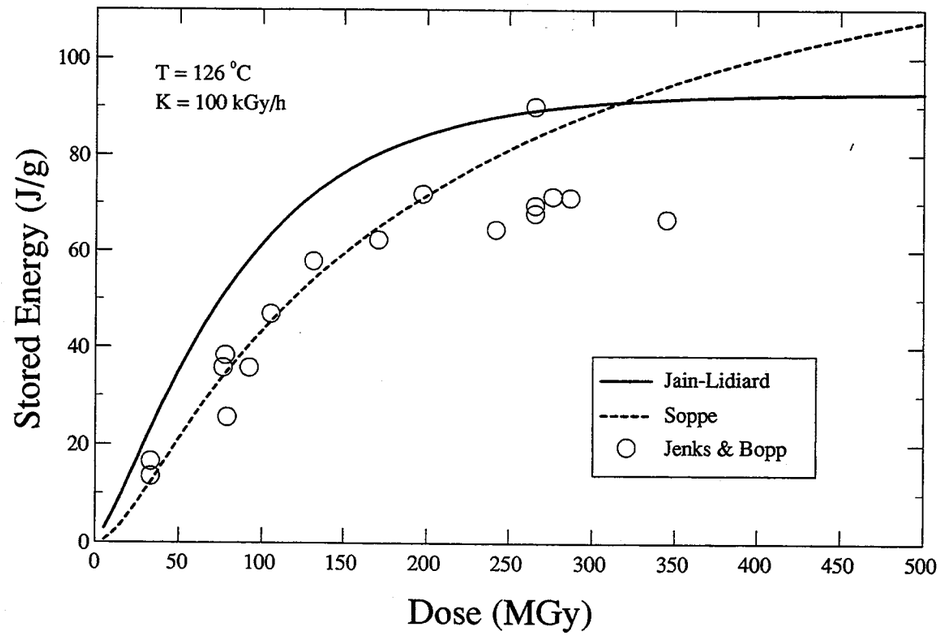

Figure 3: *Comparison of predicted stored energy values according to the Jain–Lidiard model and according to Soppe's model with experimental results obtained by Jenks and Bopp at 126 °C. Dose rate 100 kGy/h.*

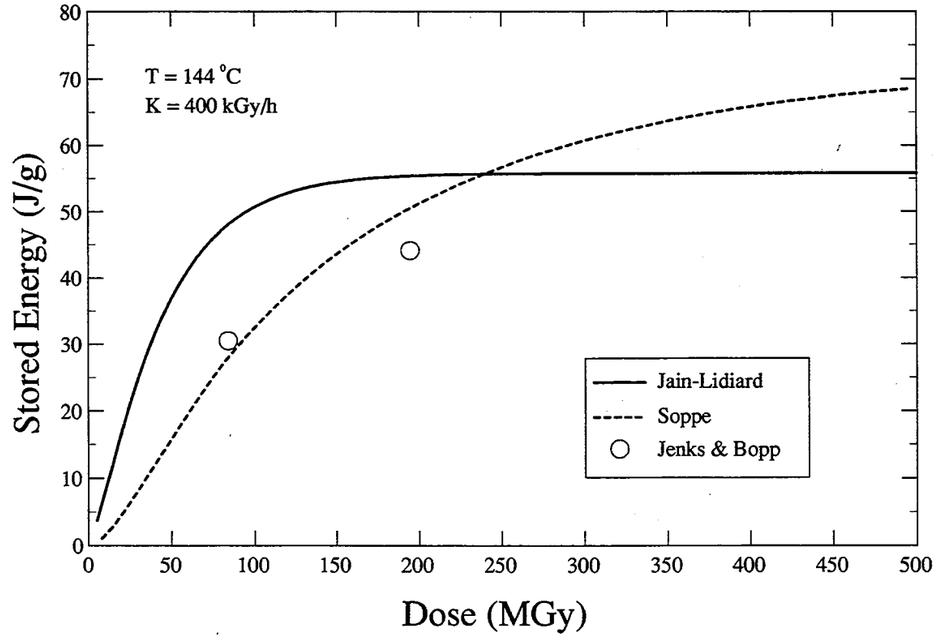

Figure 4: *Comparison of predicted stored energy values according to the Jain–Lidiard model and according to Soppe's model with experimental results obtained by Jenks and Bopp at 144 °C. Dose rate 100 kGy/h.*

4. COMPARISON OF THE MODIFIED MODELS WITH THE EXPERIMENTAL RESULTS OF JENKS AND BOPP

With the modifications discussed in the previous chapters both models were compared with the experimental results obtained by Jenks and Bopp [1974; 1977]. For the Jain–Lidiard model a satisfactory agreement between model calculations and these experiments was obtained as can be seen in Fig. 2 to 4. For the model of Soppe a satisfactory agreement between calculations and experiment could only be obtained if the maximum dislocation density was increased by a factor two i.e. $\rho_m = 2\, C_1^{2/3}$ which is in between the value suggested by Lidiard [1979] $\rho_m = C_1^{2/3}$ and the value used by Seinen et al. [1992; 1994] $\rho_m = (6\pi^2 C_1^2)^{1/3}$. With this increased value for ρ_m there is a reasonable agreement between the model calculations and the experiments as can also be seen in Fig. 2 to 4.

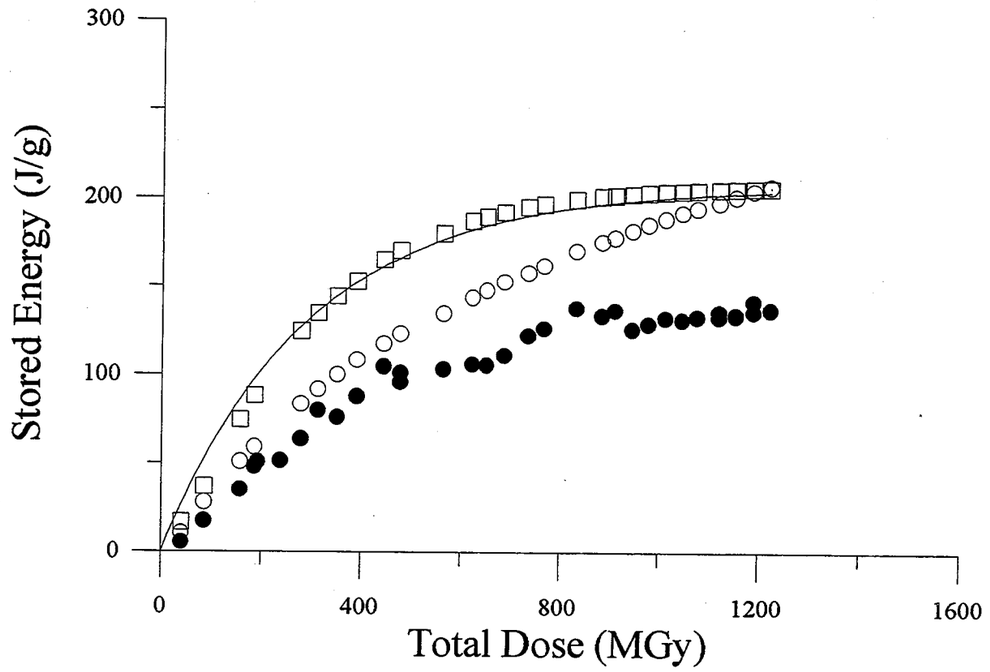

Figure 5: *Comparison of measured stored energy values on irradiated Sp–800 samples (full circles) with model calculations using the modified Jain–Lidiard model (open squares) and with model calculations using the extended Jain–Lidiard model by Soppe (open circles). The drawn line is a prediction of the damage according to Eq. (6) (see text)*

5. COMPARISON OF THE MODIFIED MODELS WITH SP–800 SAMPLES IRRADIATED IN GIF A

In Fig. 5 the stored energy data obtained for the Sp–800 samples irradiated in GIF A [Donker and Garcia Celma, 1995] are compared with predictions obtained from simulating our experiments with the models as mentioned and modified in the previous chapters. Since we do not know the impurity content of our samples we have used the model of Soppe for pure salt. In Fig. 5 it can be observed that the values predicted by the model are higher than those experimentally observed. At low total doses the predictions obtained from the model of Soppe are closer to the experimental data then the Jain–Lidiard model. The Jain–Lidiard model, however, qualitatively agrees with the experiment in respect to the occurrence of a saturation level for the stored energy at high doses. The model of Soppe does not predict a saturation level at the studied total doses.

In Fig. 2 to 4 it can be observed that at least the Jain–Lidiard model and to a less extent also the model of Soppe slightly overestimate the experimental results of Jenks and Bopp at high doses. According to the Jain–Lidiard model the colloid fraction for large irradiation times can be approximated by [Soppe et al., 1994]:

$$c_A(t) = c_A^{(sat)} \left(1 - \exp\left(-\alpha t\right)\right) \qquad (6)$$

where

$$c_A^{(sat)} = \frac{\rho_d^{(max)}}{\gamma}\left[\left(z_H - z_F\right) - z_H \frac{c_F^{(e)}}{c_F^{(sat)}}\right] \qquad (7)$$

and

$$\alpha = \frac{4\pi r_c C_c D_F}{4\pi r_c C_c + z_H \rho_d}\gamma c_F^{(sat)} \qquad (8)$$

For large irradiation times $z_H\rho_d << 4\pi r_c C_c$ and α can be approximated by:

$$\alpha = D_F \gamma c_F^{(sat)} \qquad (9)$$

The solid line in Fig. 5 is a prediction of the stored energy in our Sp–800 samples using Eq. 6 and the average dose rate in our experiments, i.e. 44 kGy/h and all the other parameters the same as in the Jain–Lidiard model. As can be seen this prediction practically coincides with the predictions obtained from simulating the experiments with the full Jain–Lidiard model (squares in Fig. 5).It can be shown that a similar coincidence occurs when simulating the experiments of Jenks and Bopp.

According to Eq. 7 the saturation value for the colloid fraction is mainly determined by $\rho_d^{(max)}$, γ and $(z_H - z_F)$. This will also hold for the full models. All three terms are not known very accurately. It is therefore surprising that the agreement between the models and the experiments is that good. It is however possible that with another choice of parameters, within their physically known accuracy, an equally good or even better correspondence can be obtained. If for instance the activation energy for the back reaction E_γ is decreased from 0.4 to 0.39 eV, $c_A^{(sat)}$ in Eq. 7 for our experiment decreases from 200 to 150 J/g yielding a much better correspondence with our experimental result. We, however, refrain from making this parameter change in the models for several reasons. Firstly, on doing so we would suggest an accuracy that is not real. The experiments of Seinen and our DTA scans show that the back reaction depends heavily on sample composition. At this moment it cannot even be excluded that the mechanism or rate determining step of the back reaction is different for different sample compositions. Secondly, an equally good correspondence between model and experiment could be obtained from modifying $\rho_d^{(max)}$ and/or $(z_H - z_F)$.Thirdly, the differences between model predictions and experiments might very well not be due to the parameterisation of the models but due to the fact that in the samples certain processes occur that are not yet included in the models. Our conclusion about the accuracy and reliability of the models can therefore not go further than that the model predictions are of the same order of magnitude as the experimental results and that they at least do not give an underestimation of the experimental results.

6. CONCLUSIONS

The discussions above shows that not to much can be said about the reliability of the model predictions under repository conditions. At this moment regarding the present results, our feeling is that it is very unlikely that the model predictions for a repository will give an underestimation of the damage. Also because some anneal mechanisms which we have observed in our experiments and of which we expect that they will increase in importance when the dose rate decreases have not yet been included in the theoretical models. To certify our statement however, more or more accurate information about certain parameters in the models and a theoretical inclusion of the mentioned anneal mechanisms is necessary. Also a sensitivity analysis to determine which model parameters are essential for the predictions for a repository is called for. On the experimental side we can say that at least more research into the nature of the back reaction and a determination of the maximum dislocation density is necessary.

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