

Figure 1 *Micrograph of a natural rock salt sample irradiated up to 2.6 MGy at 15 kGy/h and 100 °C. The black structure crossing the micrograph from top-right to bottom-left is a grain boundary void. Observe the colourless rim limited by the intense blue rim. The other colloid decorated structures are cellular patterns and incipient subgrain boundaries. Magnification 338 X.*

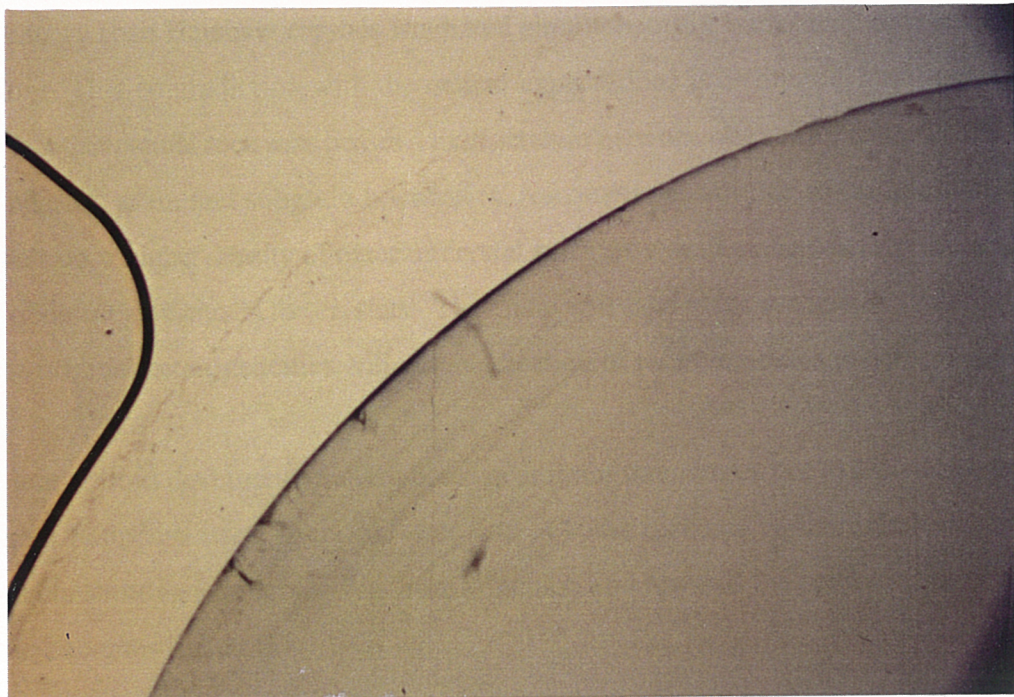


Figure 2 *Micrograph of a pure undeformed single crystal of NaCl irradiated up to 4 MGy at 15 kGy/h and 100 °C. Observe the dark colloid decorated, circular crystal outer surface. The outermost surface is too thin to be observed in this photograph but can be observed in Fig. 3. Magnification 34 X.*

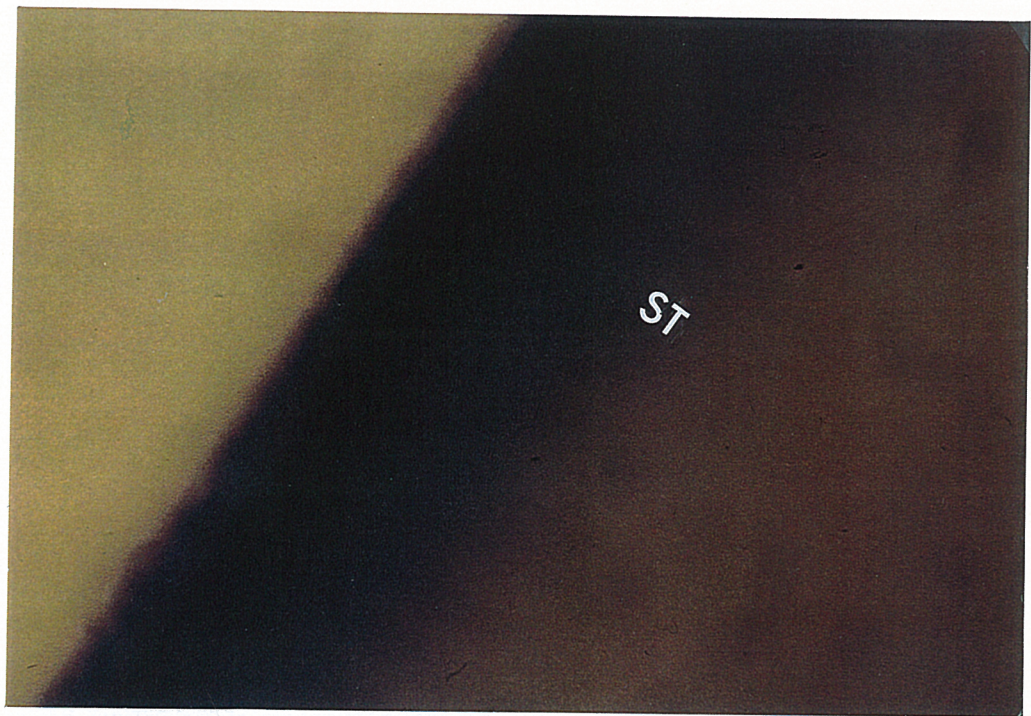


Figure 3 *Blow up of the dark rim of Fig.2. There is a colourless outer rim of the crystal which is seen as a less blue blurred rim at this magnification. The direction of incipient slip traces (ST) is indicated. The blue rim is made up of these incipient slip traces. Magnification 1343 X.*

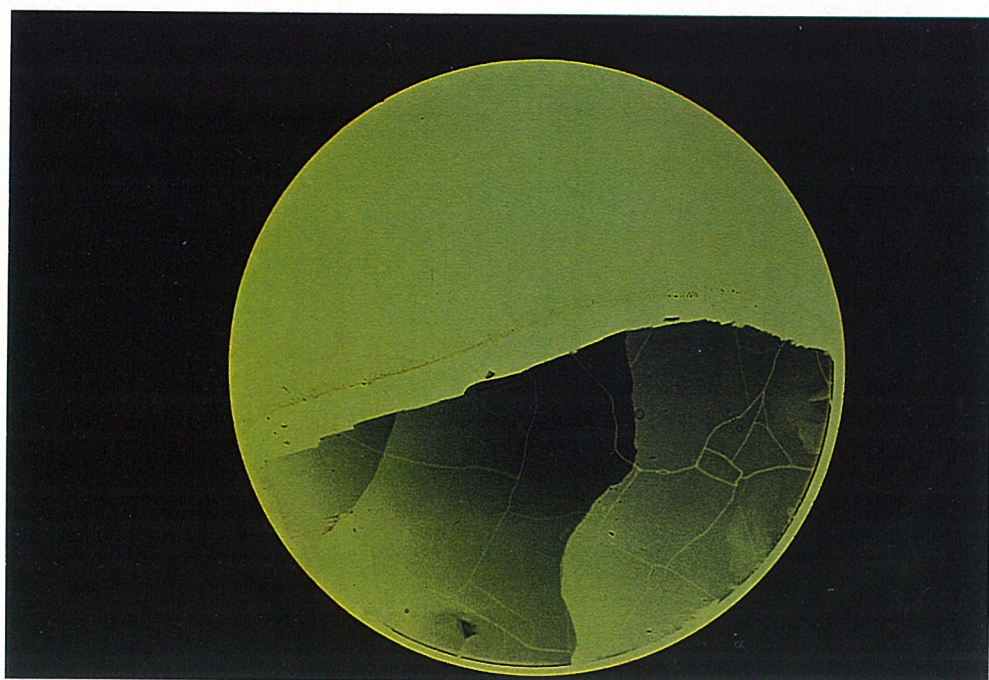


Figure 4 *Micrograph of a pure undeformed NaCl crystal irradiated up to 24 MGy at 15 kGy/h and 100 °C. The different darkness of parts of the crystal are a thin section preparation artifact. The colourless (white) lines network is a subgrain boundary network developed during irradiation. Magnification 4 X.*



Figure 5 *Micrograph of a natural rock salt sample irradiated up to 2.6 MGy at 15 kGy/h and 100 °C. Subgrain boundaries developing by climb of colloid decorated cross slip structures (cellular patterns), while near the polyhalite (P) boundary with the halite some of the subgrain boundaries already constitute a preferred diffusion path (incipient discolouration can be observed). Magnification 338 X.*

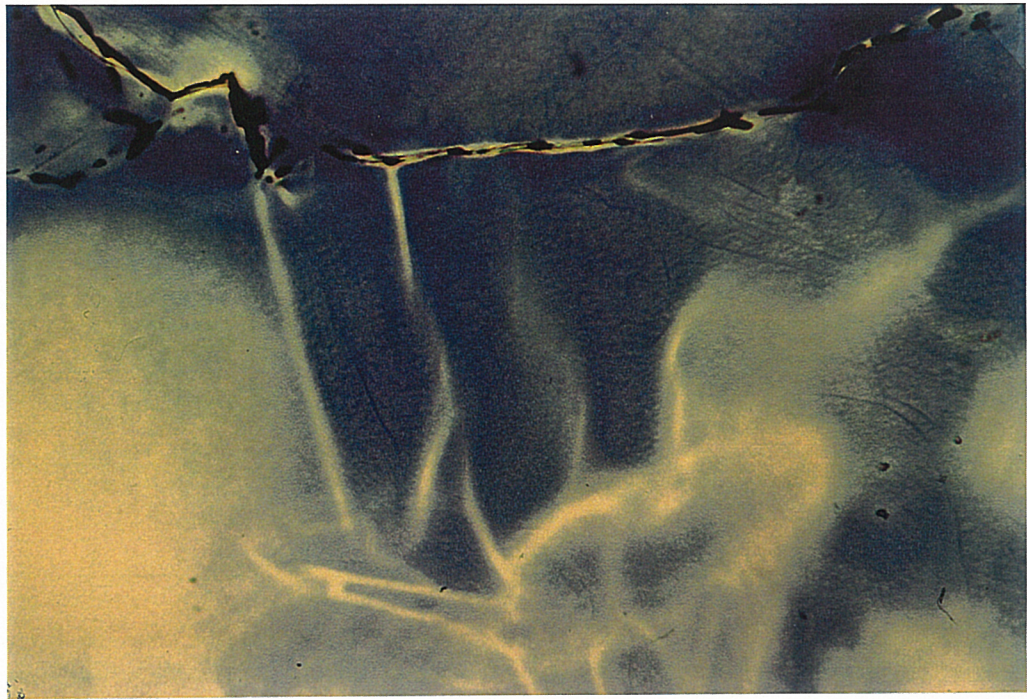


Figure 6 *Micrograph of a natural rock salt sample irradiated up to 4 MGy at 15 kGy/h and 100 °C. Well developed subgrain boundaries ending at a grain boundary, all bleached by preferred diffusion of radiation damage defects towards the grain boundary void. Magnification 338 X.*

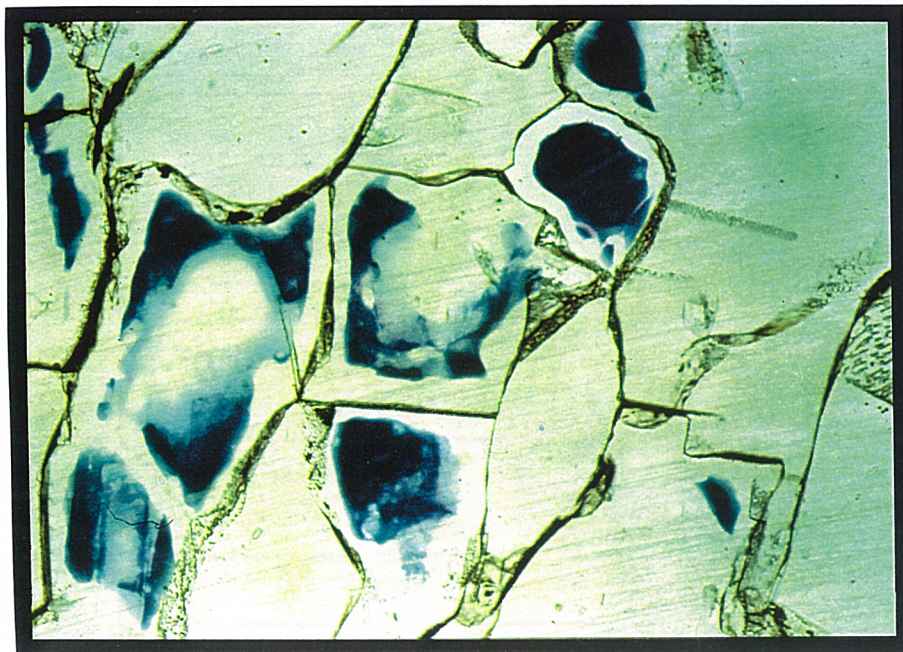


Figure 7 *Micrograph of a natural rock salt sample from the Brine Migration Test (in situ irradiation experiment at the Asse Mine). Observe the very exaggerated white (colourless) rims near the grain boundaries and the blue rims fading towards the grain cores. Magnification 7 X.*

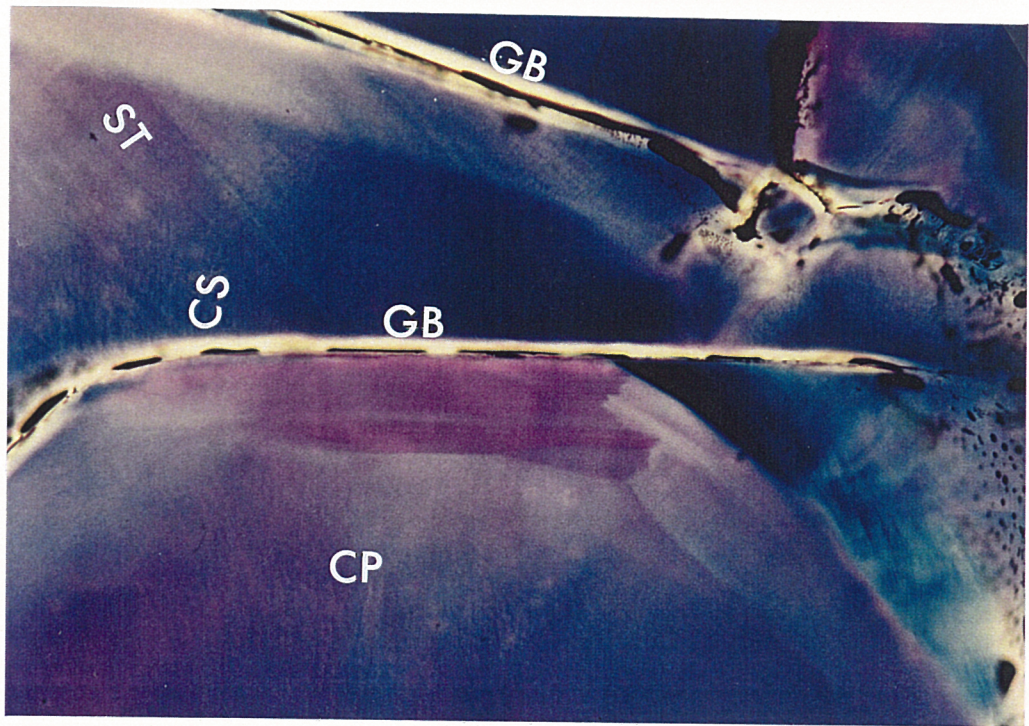


Figure 1 : *Colourless bands at grain boundaries (GB). Slip traces (ST) cross slip (CS) structures and cellular patterns (CP) indicated. See as well Fig.4. Asse Speisesalz sample, 15 Sp-800. Irrad. cond.: 100°C, 4 kGy/h, 4.6 MGy. Mag. 216 X*



Figure 2. *Colourless rims limited by intense blue lines developed at (001) fractures (F) and also at plastic deformation (PD) structures. Slip traces (ST)  $\langle 011 \rangle$  indicated. Asse Speisesalz sample T1 Sp-800. Irrad. cond.: 100°C, 15 kGy/h, 2.6 MGy. Mag. 338 X.*

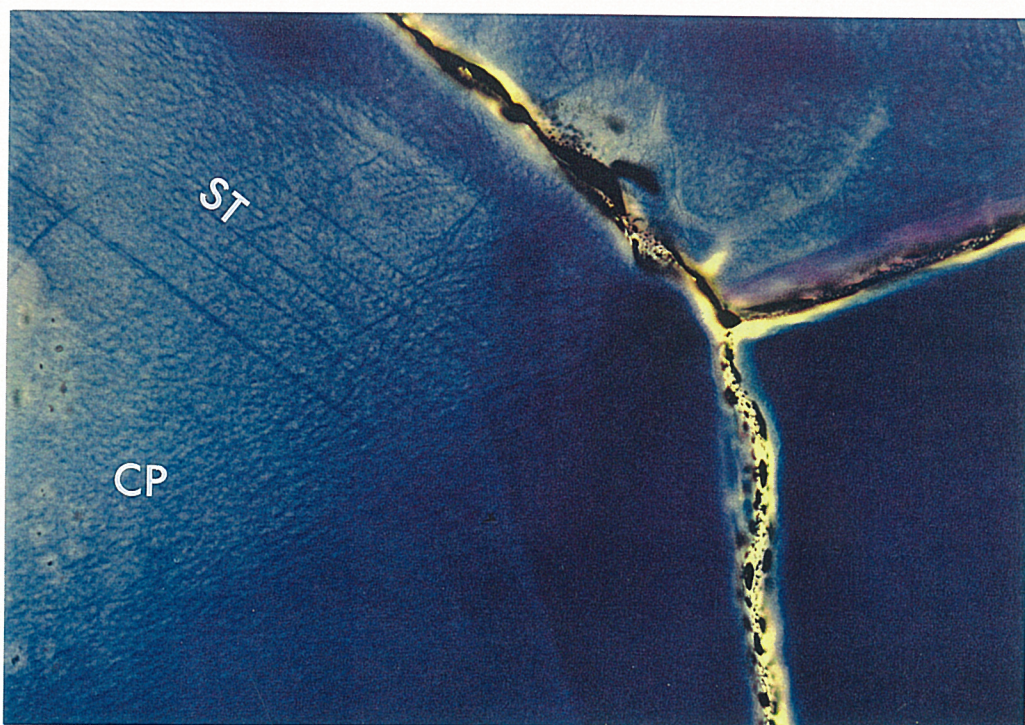


Figure 4. *Slip traces (ST), to cross slip and cellular patterns (see as well Fig.3). Colourless grain boundary with black structures. Asse Speisesalz (15 Sp800). Irrad. cond.: 100°C, 4 kGy/h and 4.6 MGy. Mag. 216 X.*

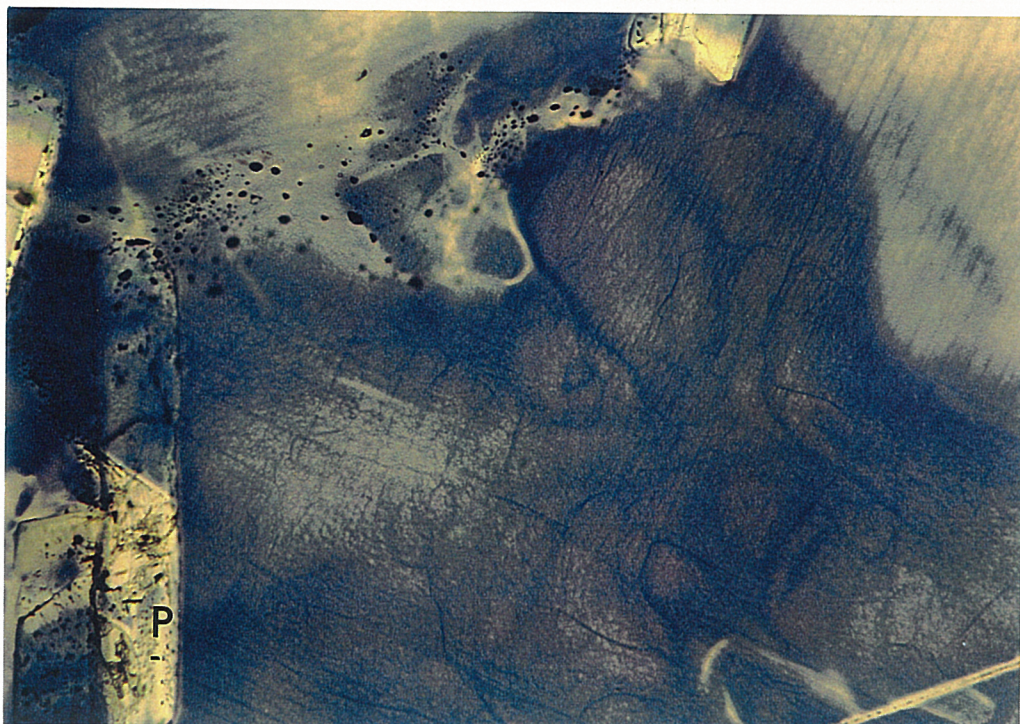


Figure 5. *Subgrain boundaries developing from rearrangement of cellular patterns. The grain boundary between the polyhalite (P) and the halite contained many fluid inclusions, the black structures. Asse Speisesalz (T3 Sp800). Irrad. cond.: 100°C, 15 kGy/h, 4 MGy. Mag. 216 X.*

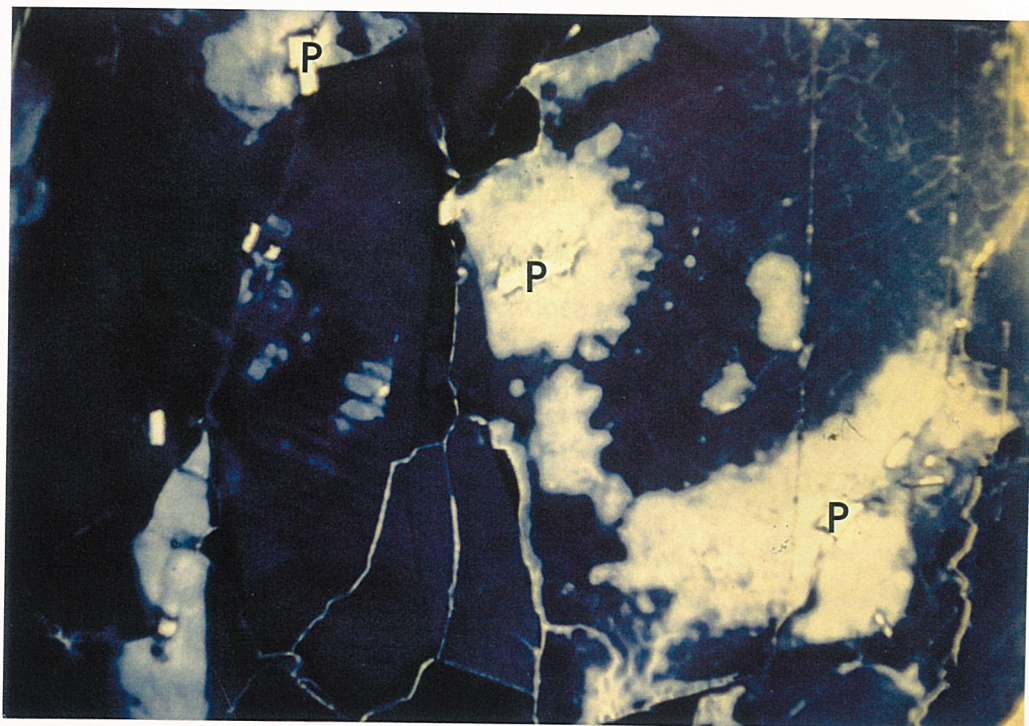


Figure 6 a. *Anneal of colloids produced by dislocation wall migration ("dry" recrystallization). Note however, that these bleached areas can contain polyhalite (P). Asse Speisesalz (T41 Sp-800). Irrad. cond.: 100°C, 15 kGy/h , 15.9 MGy. Mag. 34 X*

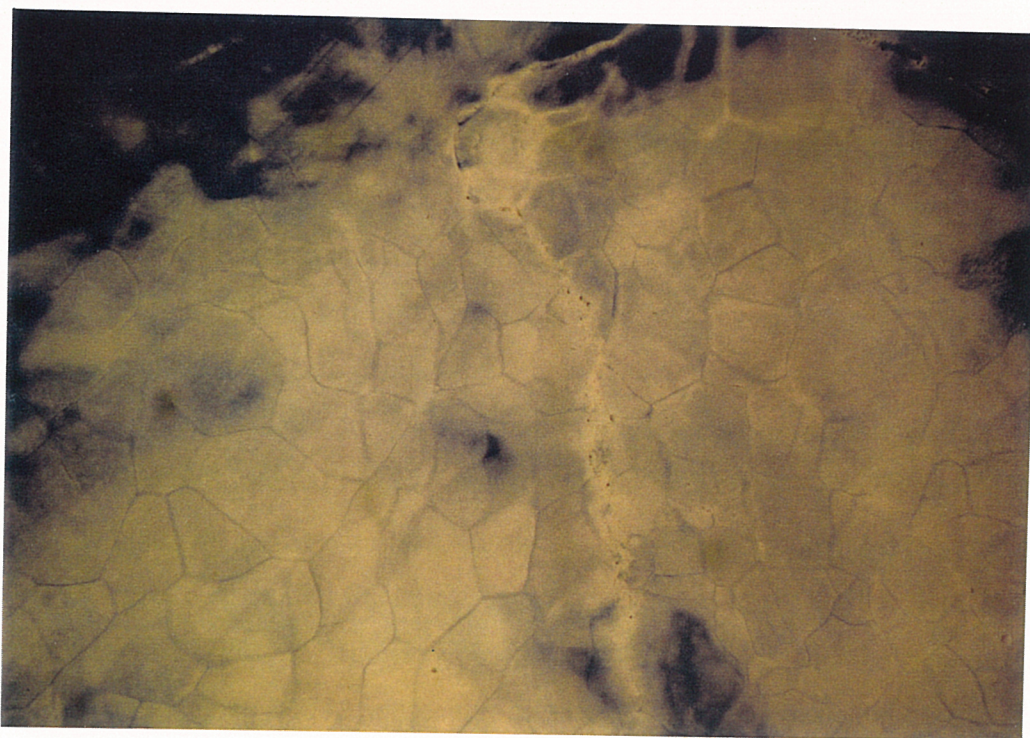
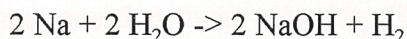


Figure 6 b. *Foam texture developed by dislocation wall migration ("dry" recrystallization) inside the bleached areas observed in Fig. 6 a. Asse Speisesalz (T50 Sp-800). Irrad. cond. : 100 °C, 15 kGy/h , 15.9 MGy. Mag. 134 X.*

damaged NaCl in the brine at the grain boundary voids, transport in solution, and reprecipitation of newly produced NaCl. In this way the wet grain boundary migrates [Urai et al., 1986; Urai, 1983]. Note that this process also eliminates the defects which were present in the old crystal, since it is dissolved in the brine.

García Celma et al., [1988] showed that FAR extensively takes place (after irradiation) in heavily irradiated samples and eliminates radiation damage. In their experiments FAR operation was proven by the existence of fluid inclusions containing H<sub>2</sub> and decorating the growth surfaces of the recrystallized grains. Hydrogen is produced by the reaction:



which takes place during solution of the Na-colloids at the grain boundary void.

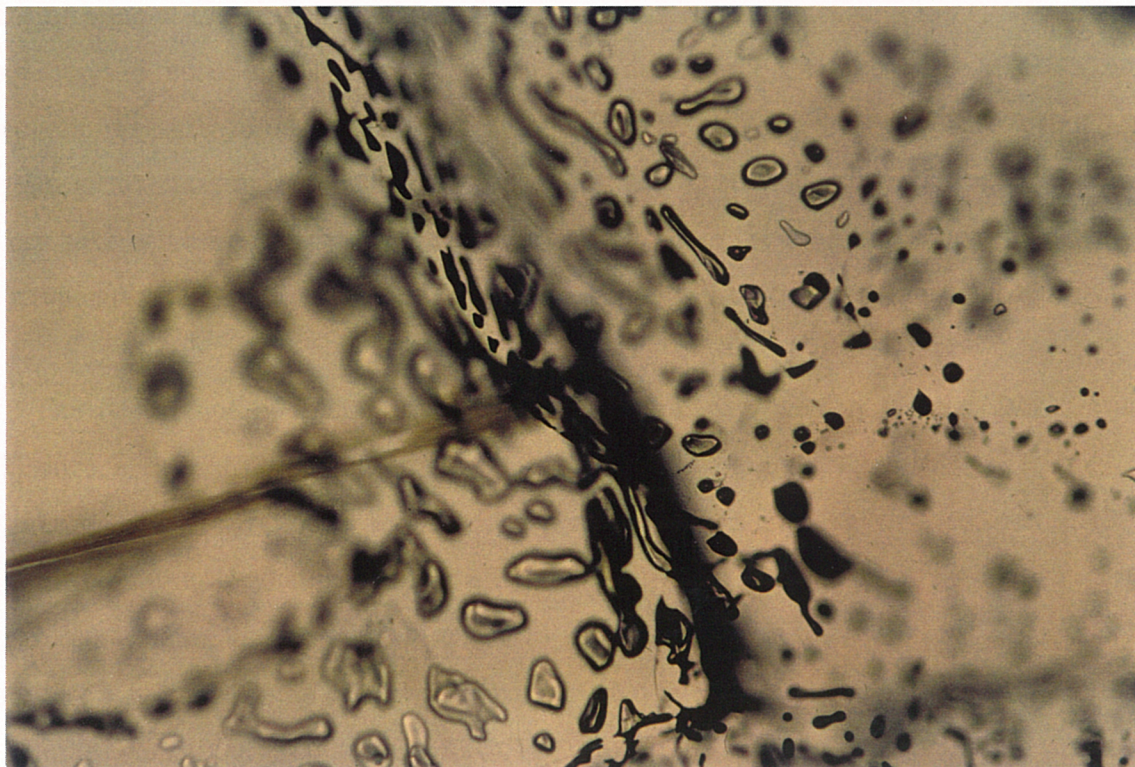


Figure 7: *Wet grain boundary. The voids in the grain boundary surface at various depths contain brine or have lost it during section preparation in which case they are observed as black. Micrograph of a "thick" thin section of an Asse Speisesalz sample (8Sp-800) . Mag. 216 X*



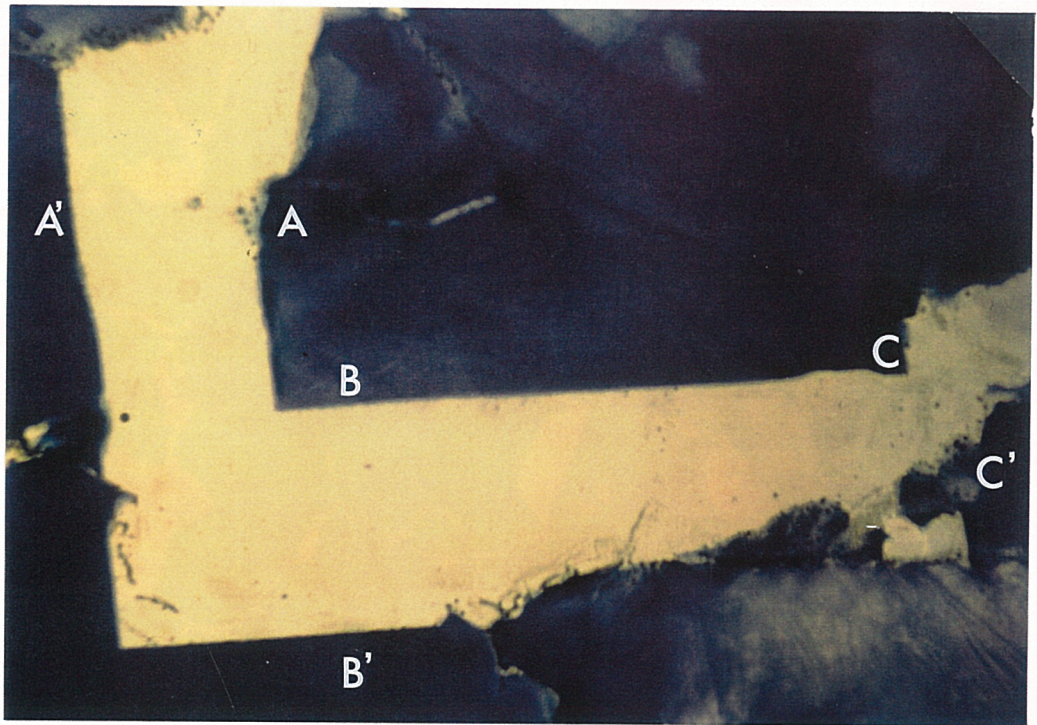


Figure 8 a : *Fluid Assisted Recrystallization. The grain boundary at ABC has migrated to A'B'C' and the colloids have been completely annealed. Asse Speisesalz sample T8 Sp-800. Irrad. cond: 100°C, 15 kGy/h, 7 MGy. Mag. 86 X.*

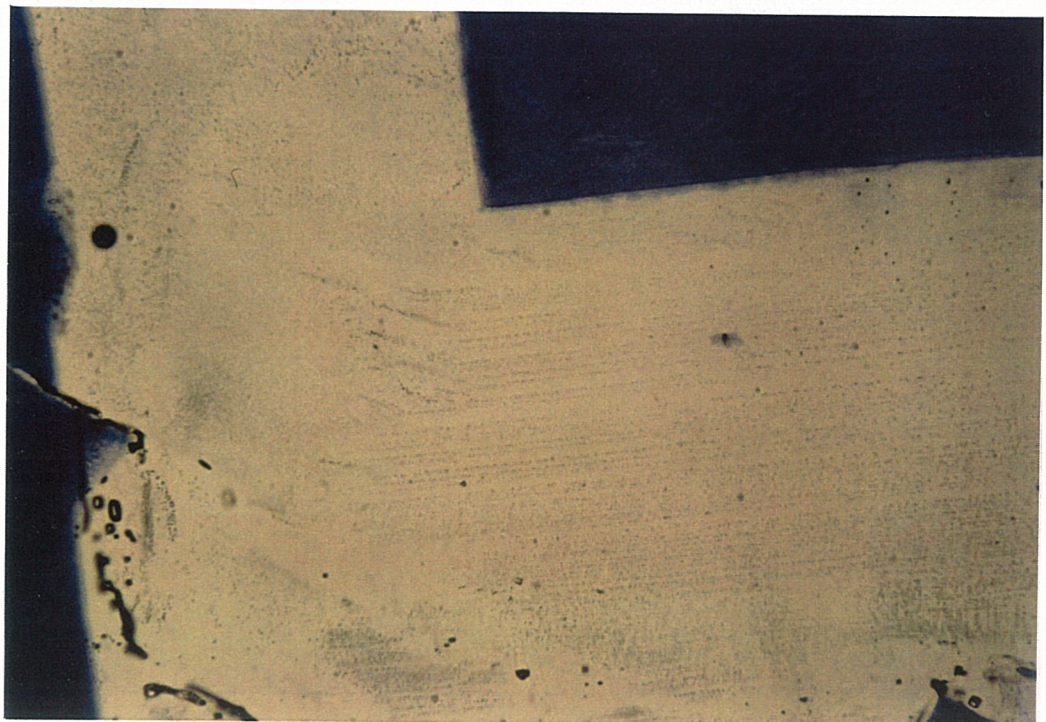


Figure 8 b: *Blow up of Fig. 8 a. Little bubbles arranged in lines consist of fluid inclusions characteristically containing  $H_2$  originating from the reaction of the Na-colloids with brine which is incorporated in the growing surfaces. Mag. 216 X*

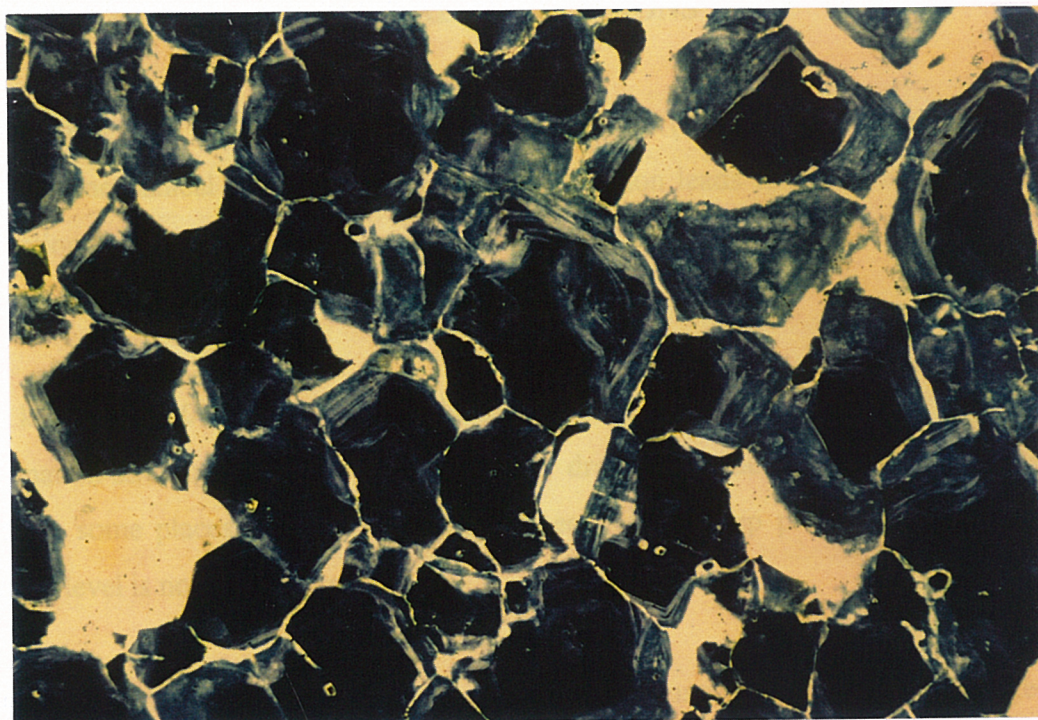


Figure 9: *Characteristic aspect of FAR grown and redecorated grains. Compare the size of the grains with that from Fig.10. NaCl Pressed Powder sample (20PP). Irrad. cond. : 100°C, 15 kGy/h, 44.6 MGy. Mag. 97 X.*

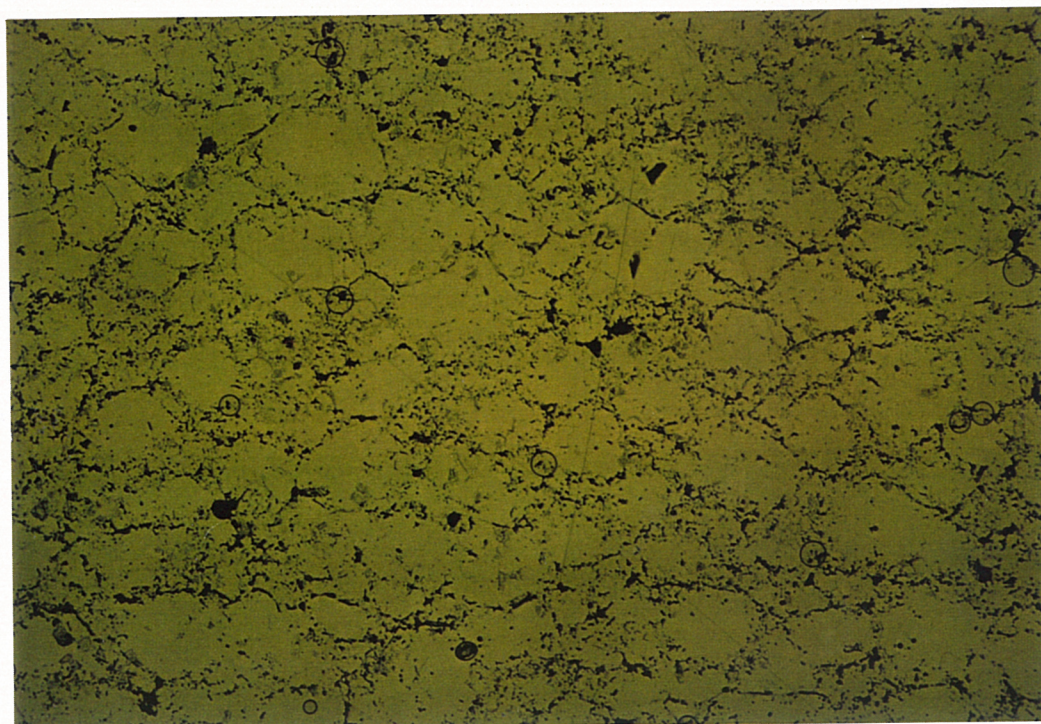


Figure 10 : *Micrograph of the original structure of the NaCl Pressed Powder samples before irradiation. Compare with Fig 9 to see the difference in grain size. Same magnification as Fig.9. Mag. 97 X.*

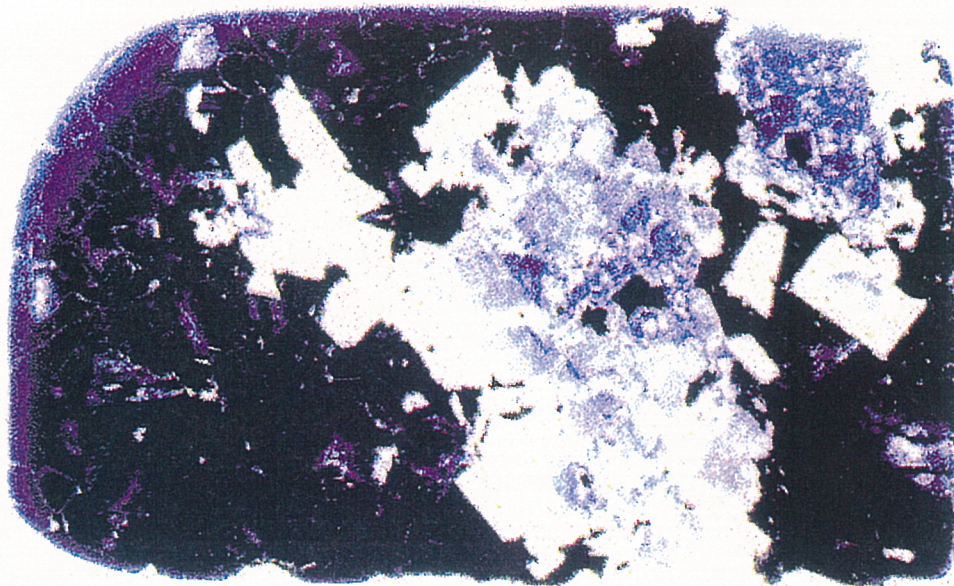


Figure 11: Transmitted light micrograph of the pressurized sample 40PLL irradiated to a dose of 44 MGy (dose rate 4 kGy/h). More than a half of the sample is represented by big crystals of new precipitated halite which tend to develop cubic faces. Long axis of the micrograph is 4 cm.

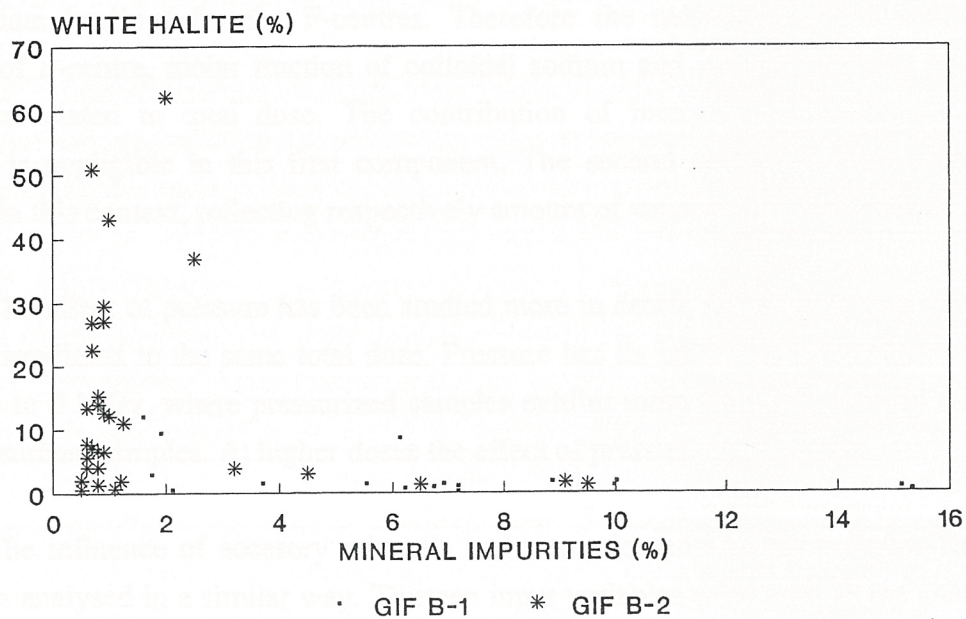
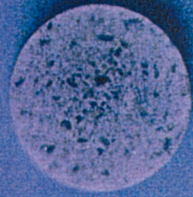


Figure 12: New precipitated white halite versus solid impurity content in irradiated rock salt.

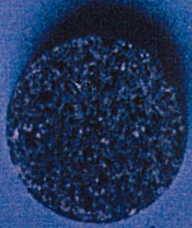
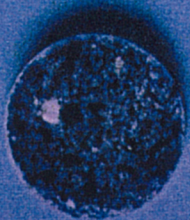
Influence de la dose intégrée d'irradiation



Sel de ASSE  
non irradié

Sel de ASSE irradié  
 $10^3$  Gy -  $10^4$  Gy/h

Sel de ASSE irradié  
 $10^4$  Gy -  $10^4$  Gy/h



Sel de ASSE irradié  
 $10^5$  Gy -  $10^4$  Gy/h

Sel de ASSE irradié  
 $10^6$  Gy -  $10^4$  Gy/h

Sel de ASSE irradié  
 $10^7$  Gy -  $10^4$  Gy/h

Fig 2 : Coloration of salt samples irradiated at different doses

The above compositions are considered to be satisfactorily representative for the individual samples that were filled into the ampoules. In total, 160 ampoules each having 300 g of rock salt were prepared, sealed and irradiated in the HFR at Petten (for results see Jockwer et al. [article nr. 13, this volume] and Mönig et al. [article nr. 16, this volume]). As everytime the entire subsample participates in the production of radiolytical gases

The situation, however, is very different when partial samples are being taken for the calorimetric determination of stored energy. In that case, the single sample to be measured weighs only ~ 0.3 g.

The microscopic picture shows very clearly an intergrowth of the halite and sulfate components too close as to allow for a mechanical separation of the minerals (Fig. 2). Likewise, no method is available to take a single specimen having the representative bulk composition.

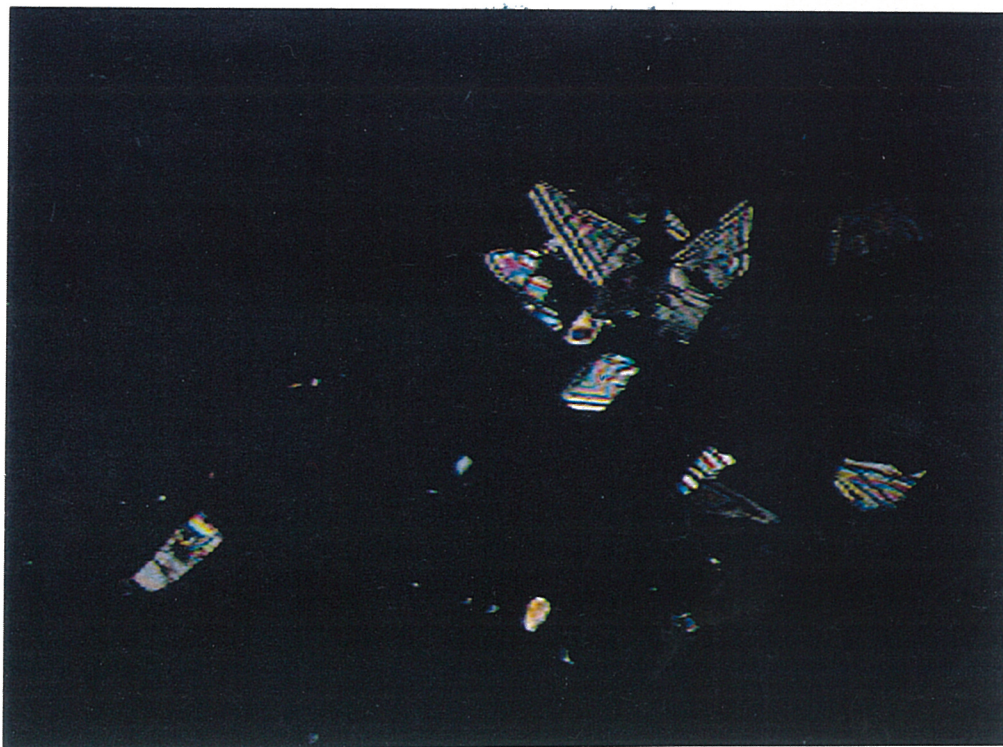


Figure 2a: *Polyhalite and anhydrite crystals in halite (black). (The grain sizes shown here are the coarser ones; a lot of the sulfate portion of the rock salt is represented by very fine grained particles of a few microns). long edge = 1 mm; Nicols †*

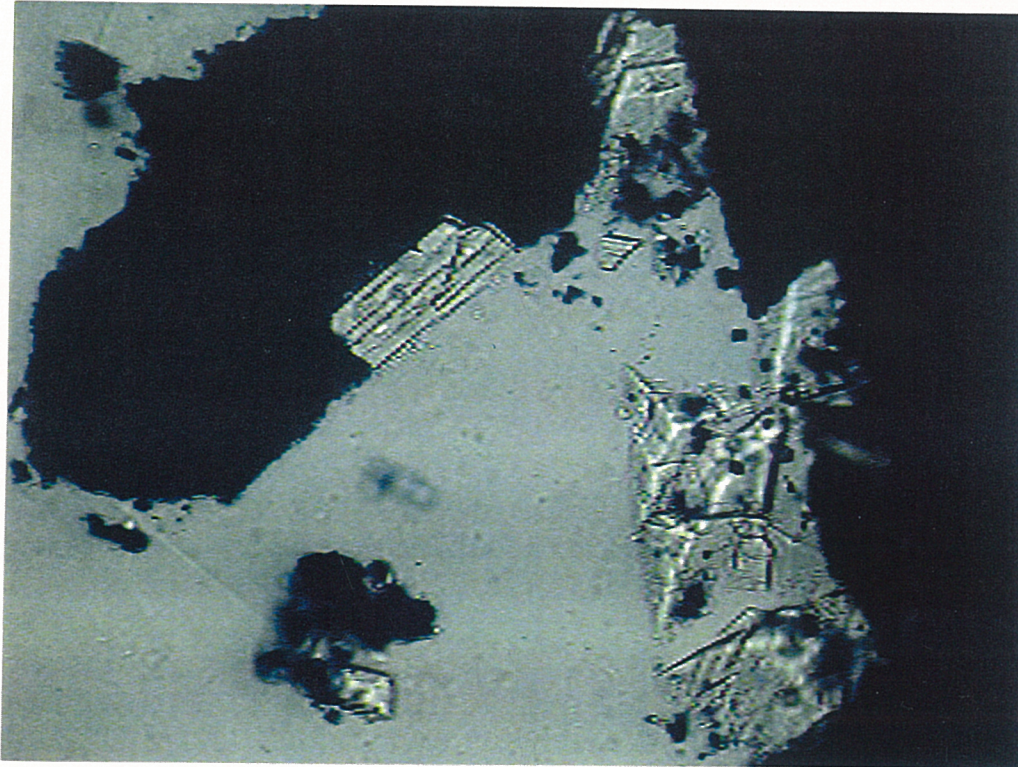


Figure 2b: *Anhydrite and polyhalite crystals in or adjacent to halite turned black (colloidal sodium) by the irradiation. Long edge = 0.2 mm; Nicols //*

The only way to obtain reliable information as to the amount of energy stored in the halite portion of the rock salt seemed to be to perform calorimetric measurements, be it on simple subsamples or on a halite concentrate separated by hand-picking under the microscope. Important in both cases is, that the calorimetric specimens have to be analyzed once more after they were measured, as the originally chosen composition can no longer be expected.

For a general information and evaluation of the potential role of trace elements both in the halite and the sulfate minerals of the rock salt an extensive description was given in the final report on 'Material inventory and petrophysics of the HAW-test field in the Asse' [Gies et al., 1994].