THE RECRYSTALLIZATION APPARATUS

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ABSTRACT

A "recrystallization apparatus" has been designed and constructed. The apparatus was meant to allow observation of static recrystallization. It can subject sample slices up to 3 mm thickness to a temperature of 100°C, and to a lithostatic pressure of 200 bar while allowing microscopic observation of the samples and independent control of the intergranular fluid pressure. We meant to observe and photograph the progression of static recrystallization in irradiated rocksalt samples. As a consequence of the use of salt in contact with metals, repeated corrosion of tubes, connectors and devices constantly caused leaking which had to be solved. The apparatus has now reached a state of development where it can be used without leaking. In this article the apparatus design and materials as they are now are described.

1. INTRODUCTION

Within the framework of the studies on radiation damage in rock salt it was important to observe the progression of fluid assisted recrystallization of irradiated rock salt not subject to differential stress. Fluid assisted recrystallization consists of the solution into the grain boundary fluid of species from the grain boundary, and transport and re-precipitation of the dissolved species in another place [Urai, 1983; García Celma et al., 1988]. A driving force for this process is present provided some parts of crystals in contact with the fluid are damaged while others are not. The non-damaged crystal areas then grow to the expense of the damaged. In our case the damage is produced by gamma irradiation prior to the experiment, and the non-damaged rims present in some crystals are the result of diffusion processes [García Celma and Donker, 1995, art. nr. 20 in this volume]. The fluid at the grain boundary void is brine, which is present in natural rock salts in different amounts.

Experiments where irradiated salt samples were set in pressure vessels under enhanced pressure and temperature had previously shown that fluid assisted recrystallization of rock salt takes place [García Celma et al., 1988]. To directly observe the process while it takes place was the reason for building the apparatus.

J. Urai , and W. Means, constructed apparatus where dynamic recrystallization produced by differential stress could be observed. In their apparatus, thin sample slices are observed by transparency under the microscope, while being deformed by the relative displacement of the sample boundaries. In these apparatus recrystallization is made evident by the change of optical orientation of material portions which results in colour and illumination differences when observed between the crossed polarizers of a petrological microscope. The existing apparatus, however, were not adequate to study the effect of lithostatic pressure and of temperature in (static) recrystallization processes driven by the energy stored in lattice defects of the mineral grains.

The exigences to be met by our apparatus were :

a) The temperature of all parts of the apparatus had to be homogeneous (spatially) to hinder transport of species in solution from hot to cold places with reprecipitation in the cold places.

b) The temperature had to be constant or at least not subject to sudden changes to hinder heterogeneous nucleation.

c) The apparatus had to allow for the pressure on the sample to be constant and controlled but vary in different experiments from 0 to 200 bar.

d) The apparatus had to allow for changes, and control of the pressure of the brine in between 0 and 200 bar.

e) The apparatus had to possess good light sources and thus distributed that the samples could be observed and photographed by transmission and by reflection.

Our apparatus did not need polarized light for recrystallization observation because the irradiated salt is blue or black and the recrystallized salt is not irradiated and therefore colourless. Moreover salt is optically isotropic. Nonetheless, if wished, polarized light can be added to the system.

2. THE DESIGN

2.1. The first design

The first design for the apparatus was partly due to Dr. J. Urai and consisted of a nearly cubic block of brass with a vertical cylindric hole in its centre (Fig.1). A glass plate was placed on the bottom of the hole (which is a cylinder with shorter radius). On top of the glass plate a salt sample slice covered by a second glass plate was placed. The whole arrangement is pressed together by a lid which covers the cylindric hole and can be screwed to the hole walls. The lid also has a round hole of a shorter radius. The light can go through the glass plates and the sample thus allowing vertical microscopic observation.

The volume between the glass plates is further closed by two O-rings each surrounding one glass plate. The O-rings contact the metal walls. An horizontal duct drilled in the metal wall brings the space between the glass plates in contact with the exterior. Brine could be added or subtracted through this duct which was in contact with an "oversized brine pump" acting as well as brine reservoir. The very first version of this pump was a clinical syringe. The idea behind this was that the brine would be pumped into the space between the two glass plates and set the sample under pressure. A manometer was placed in the tube which connects the brine pump with the duct in the metal block to measure the brine pressure.

Heating took place by means of two heating elements fitted into two holes made in the metal block. Another horizontal duct was drilled to introduce a thermocouple. The thermocouple was read by a proportional band temperature regulator which regulated the power sent to the heating elements. The hole system was fixed on top of a copper plate and placed within a "pertinax" box for temperature isolation.

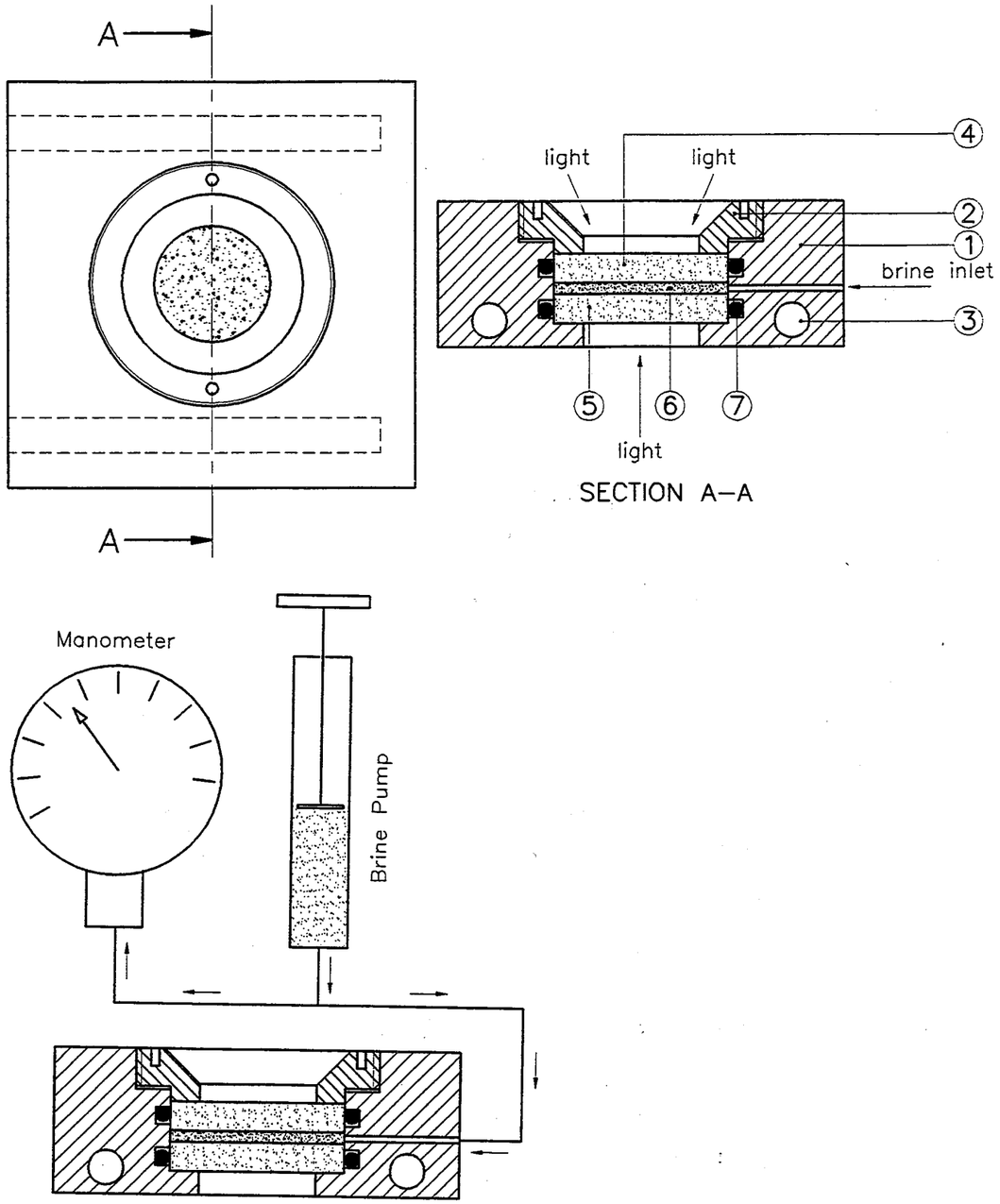

Figure 1. *Schematic views of the old design of the recrystallization apparatus. 1) casing, 2) lid, 3) holes for heating elements, 4) top glass window, 5) bottom glass window, 6) sample slice, 7) O-ring.*

Through progressive mending of all what turned out to disfunction the new design gradually emerged. The main problems of this old design were:

a) the lack of real control on the pressure imposed on the sample which depended mainly on the tightness with which the lid was screwed,
b) the poor contact of the glass plates with the O-rings,
c) the easy corrosion of the material in spite of the electrolytic gold plating,
d) the high temperature gradient in the system, and
e) various leaking points.

These problems did not stop the samples from dissolving as expected, but caused the reprecipitation, which ought to take place in the sample producing the recrystallized material, to take place at the leaking points, outside the apparatus, which, moreover, were colder than the sample.

Nonetheless some observations could be made with this apparatus.

2.2. The new design

Very important in this new design is the choice of materials as discussed in 3. The basis of the new design is a cylindric metallic block which (Fig. 2) contains three glass windows placed between two metallic covers which are attached to each other by studs and nuts.

The casing consists of the cylindric metallic block. The glass windows are circular plates of boronsilicate glass with thin metal rings glued to them. These metallic rings improve the contact of the glass with the O-ring which is otherwise rather poor. The glass windows are on top of each other inside the casing which they divide into two spaces: the sample chamber and the gas chamber.

The space between the two lower glass plates, *the sample chamber*, meant to contain the sample which recrystallization is to be studied, can hold samples of thicknesses up to 3 mm. To introduce brine in the sample chamber two horizontal ducts have been drilled in the metallic walls.

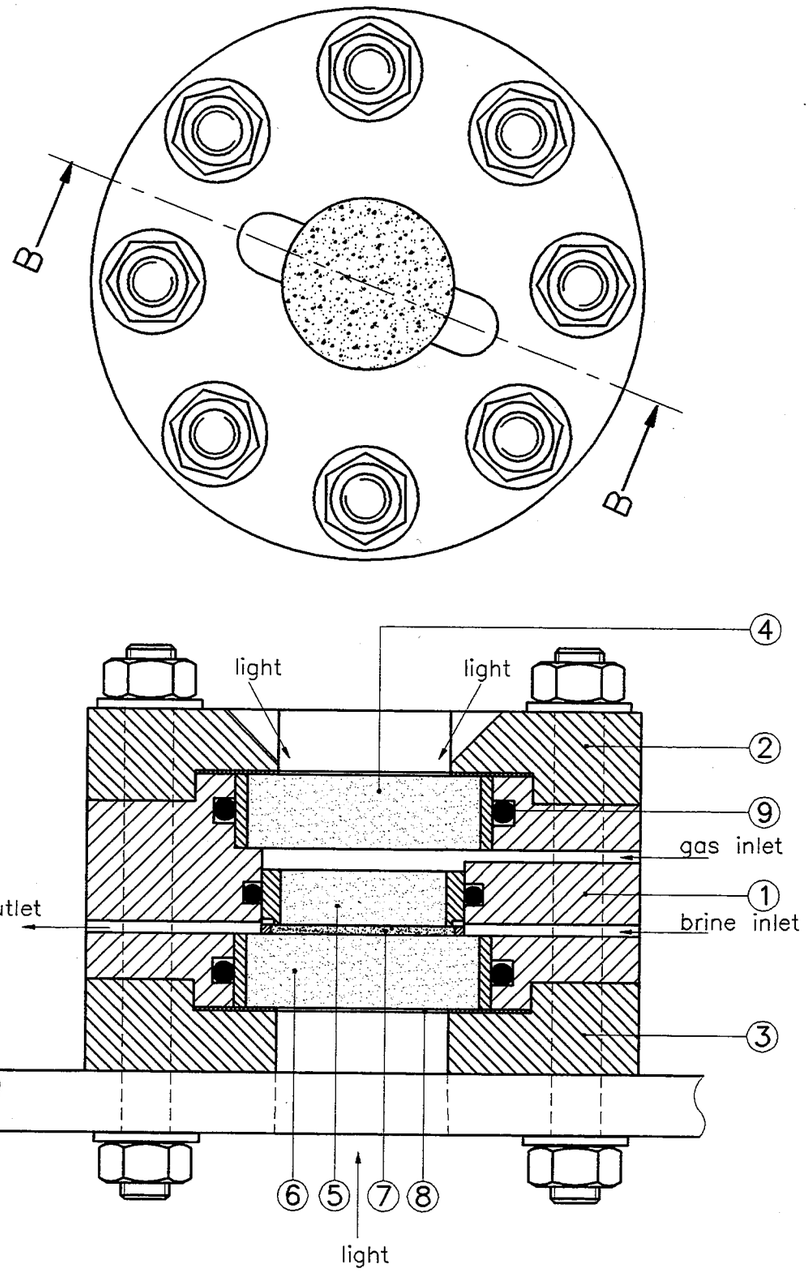

Figure 2. *Two sections of the new design of the recrystallization apparatus. 1) casing, 2) top cover, 3) bottom cover, 4) top glass window, 5) mid glass window, 6) bottom glass window, 7) sample slice, 8) Arlon rings, 9) O-ring.*

The samples can be mechanically pressurized by filling the space between the middle and top glass plates, *the gas chamber*, with gas at high pressure. The gas is introduced into the gas chamber through an horizontal duct drilled in the metallic wall (Figs. 2 and 3).

The metallic *covers* which hold together the sample housing and its contents are very stiff to hinder their bending under pressure. Bending of the covers could produce differential stress on the glass windows and break them. Two arlon rings are placed between the glass and the covers. The arlon rings smooth away eventual imperfection in the material flatness which could also produce differential stress and fracturing. The covers are hold together by 8 studs and 16 nuts.

Two circular holes have been cut out from the covers. The holes have a diameter of 28 mm, as planned for the samples which can therefore be observed (except for a rim of 1.5 mm) during the experiment. A lamp shines through the lower cover circular hole. In the top cover two additional slots have been made on the circular hole wall where two additional lamps can be placed and fixed on a good illumination angle. The samples can thus be observed by transparency and by reflexion and photographed through the upper cover circular hole.

There are two independent pressure systems feeding the experimental pile, one for the gas, and one for the brine. The pressure of the brine can be regulated and read independently of that of the gas (Fig. 3). This opens up the possibility of performing another sort of experiment i.e. on the relationship between intergranular fluid pressure and lithostatic load.

The gas pressure system is made up of a tap, a regulable overpressure safety valve, a manometer, connectors and tubes. This system ends on the one side in the gas chamber, and on the other side is attached to the pressure producing installation i.e. a gas bottle.

The brine system is made up of the same elements as the gas pressure system and some other additional devices. At one side of the sample chamber an additional tap for vacuumimg is present, while at the other side a "brine pump" constitutes the equivalent of the gas producing installation. This "brine pump" is a device to supply brine and brine pressure consisting of a reservoir filled with brine which can be pumped by a piston driven by a spindle.

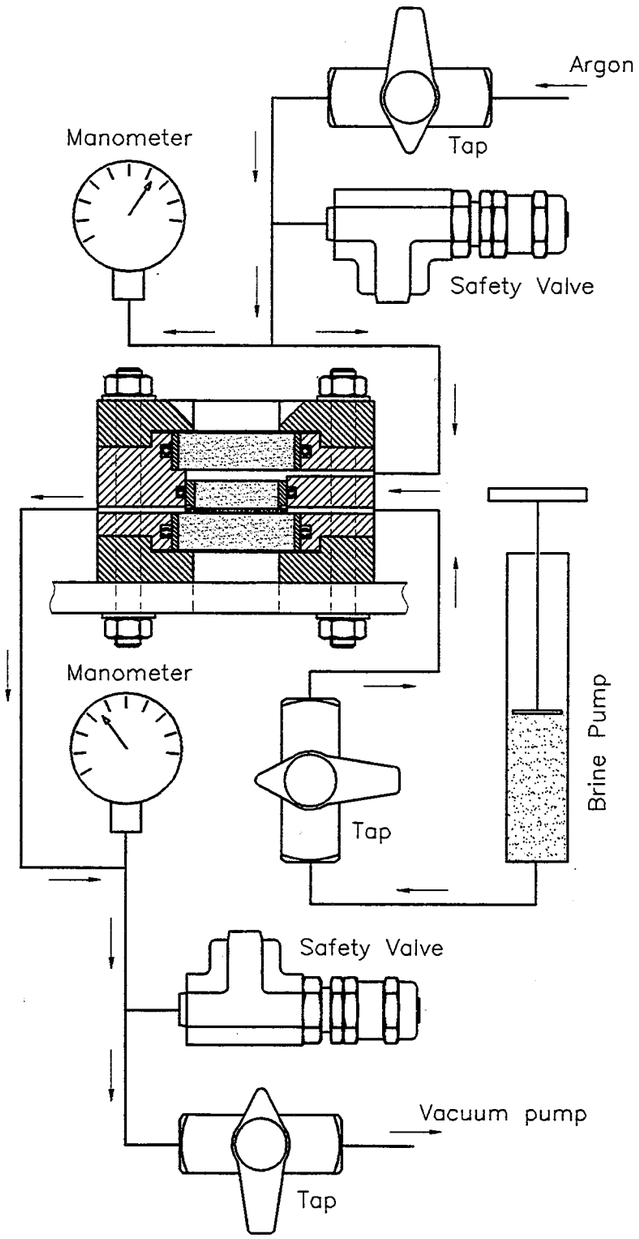

Figure 3. *Schematic representation of the complete recrystallization apparatus including the gas and brine system.*

All connections between system components are made through metal tubes with a diameter of 6 mm and a wall thickness of 1 mm. To link tubes and devices connectors with Swage-Lok tube fittings are used.

The total system as constituted by casing, covers and studs and the two pressure systems is fixed on an aluminium plate measuring 260 x 215 x 10 mm. All the devices and tubes have been fixed as near to each other as possible so as not to loose any heat and not to create heat gradients. To achieve this, parts of the apparatus have been fixed on the down side of the plate. The tubes cross therefore the aluminium plate through a couple of holes. See Fig. 4. The casing and the covers are tightened to the aluminium plate through the same studs and nuts which hold them together, and the other components are fixed using braces and screws.

The sizes and materials have been thus chosen that the hole set up amply accomplishes the safety regulations to work at 200 bar of pressure. This allows for use of the apparatus outside special high pressure laboratories. It has also been repeatedly tested for safety, see 4.

Heat is supplied by four heating elements of 100 W each (at 220 V) which sheaths have been fastened to the aluminium plate (two above and two below) and distributed so as to minimize temperature gradients. Heat power is regulated by a proportional band temperature regulator; the temperature is read from a thermocouple placed near the sample in the casing.

Thermal Isolation is reached by providing the aluminum plate with three legs and placing it all inside an isolation box. The box is 350 x 310 x 210 mm made of stainless steel plate 1 mm thick, covered with a lid, and filled with a 50 mm thick rockwool blanket. The lid on top of the box has three possible openings, one to photograph the sample and/or illuminate it, and two to read the manometers without opening the box. Each of the three holes can be closed with a lid. All exterior lids have a layer of Pertinax in their inner side. There is also a plate of pertinax in the bottom of the box. On the side of the box holes have been perforated for the 220 V electricity cable and its protection, the two jacks for the light plugs and the thermocouple to go through.

Illumination is made through a lamp fixed in the bottom of the box in a position convenient to illuminate the bottom of the sample. On the top lid of the box a holder is positioned to fix the two lamps which have to illuminate the sample from above. All three lamps can be step-

less dimmed.

3. CHOICE OF MATERIALS

The materials for the casing and pressure systems had to be metals due to the thermal conductivity and mechanical properties required. However, sodium chloride in the presence of water easily corrodes and oxidates metals, and therefore the choice of metals was limited by their resistance to corrosion. Most resistant to corrosion by salt are Ni alloys, like Inconel.

The casing is made of Inconel 625 and the rings glued to the glass-windows are made of Inconel 600. Inconel 625 is the most resistant to corrosion by salt. The tubes and the conic parts of the swagelock fittings which join the casing and the brine system are made of Inconel 600. The tubes have been welded to the conic fitting pieces using the Tungsten Inert Gas (TIG) method.

The covers which hold together the casing and glass windows are not in contact with either brine nor salt and therefore could be made of Remanit 4122 which is not resistant to corrosion by salt but can be (and was) hardened up to 48 HRc.

The rings between the glass windows and the metal covers are made of a synthetic material named arlon which can stand the required temperatures remaining elastic. Arlon can deform under high pressure adapting its shape to the eventual small roughnesses in the contacts between glass and metal but does not flow at 100°C and 200 bar.

The tubes and fittings of the gas system are made of stainless steel (316 L).

All fittings, taps and valves of both pressure systems are made of the same type of stainless steel (316 L). In the case of the brine system this material choice is only due to the ease with which fittings, taps and valves could be purchased, but the definitive version of the apparatus was meant to have them made of Inconel 600.

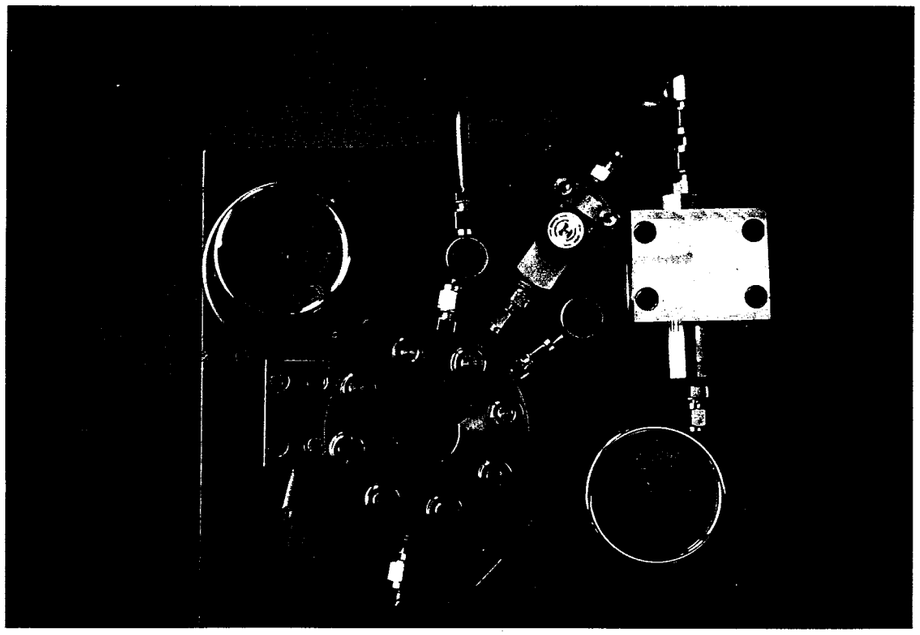

Figure 4a: *Top view of the recrystallization apparatus*

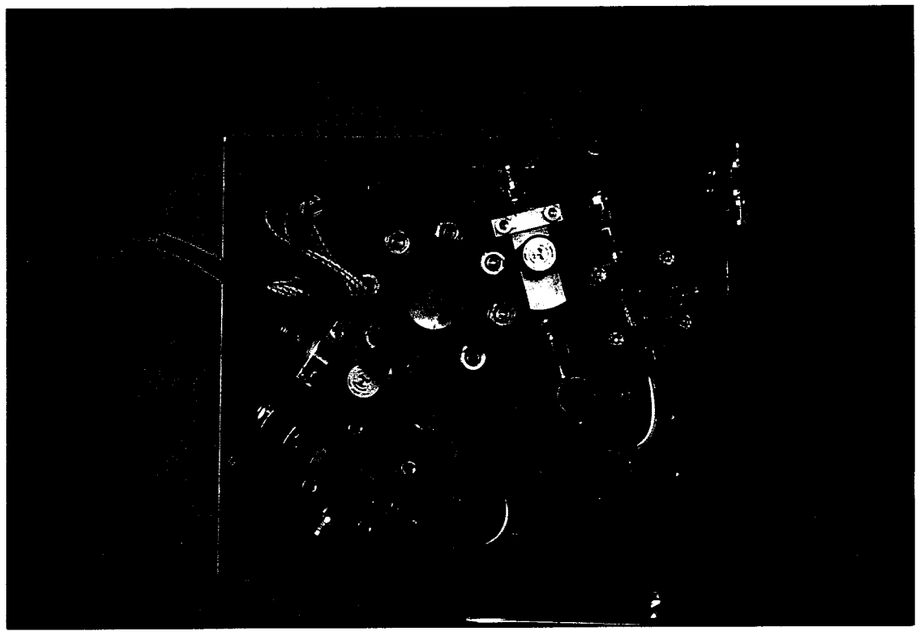

Figure 4b: *View from below of the recrystallization apparatus.*

The three taps of the system are of the type Plug Valves produced by NUPRO, they work by means of a stop with an O-ring which can be twisted into the outlet hole closing it. This taps are chosen because they are simply built and easy to clean.

The two safety valves are of the type R3A from NUPRO, they can be step-less adapted to a chosen pressure. This type of valve has been chosen to allow for a precision release of pressure and increase the safety of the apparatus during the experiments.

The connectors used for the tubes are of the type Swage-Lock for 6 mm diameter. This type of connectors works by means of a conic tube piece which, when a nut is screwed, it is attached to a 6 mm diameter tube constituting a gastight connection. The tube with the conic piece can be connected to another piece with a conic hole and hold together by screwing yet another nut. The contact between the two conic elements is also gastight at the applied temperatures.

We were unable to purchase manometers with inner bourdon of any material more resistant to corrosion than stainless steel (316).

Teflon tape was used at the conic fittings of the Swage-Lock links and at the taps of both gas and brine system and silicon fat to impregnate all O-rings which are found in the casing, the taps, the pressure valves and the fittings of the manometers. The O-rings themselves are made of Viton with a hardness of 70 Shore. This material is nor easily corroded, adequate for vacuum and can be used at temperatures of between -20 and 200°C.

Braces from aluminium and inner hexagonal screw of stainless steel (316) fix all parts of both systems to the aluminium plate.

Glass windows from boronsilicate of 27 mm diameter and 9 mm thickness, and 38 mm diameter and 12 mm thickness, with the following specifications: parallelism deviation less than 0.05 mm, flatness deviation less than 0.01 mm and, between plane and cylindric surfaces a 0.1 mm rounded corner was polished off. For each experiment one small and two big glass -windows are needed.

The glue which attaches the metal rings to the glass windows is one -component epoxy-glue of the Permapox 2032 AB type, which hardens at a temperature of between 90 and 120°C. The glue is gastight and resistant to chemical agents and can be used up to a temperature of 160°C.

Leak fluid not aggressive for the used materials is also used to check on the gas tightness of the system.

4. EXPERIMENTAL METHOD

4.1. Sample preparation

To observe a sample by transparency, a slice of it as thin as possible is needed.

Since it will be impossible to saw and polish a very thin slice of salt without it falling apart, the sample is glued to a support, in this case a metallic ring. It is proceeded as follows: a cylinder of the sample to recrystallize is machined out of a bigger piece in a cylinder adapting to a tube of stainless steel 316 with a precision of 0.4 mm. The sample is introduced in the tube and glued to it using the same glue as for the metal-glass contacts in the apparatus. After hardening of the glue one of the surfaces of the sample is machined flat and polished with a 600 Grit polishing paper. Now, a slot is made in the stainless steel and up to the glue with a width of 0.9 mm, and at a distance of 1 mm from the first flat polished surface. The width of the slot is chosen in relation with the thickness of the diamond saw blade (0.85 mm) with which the salt sample will subsequently be cut, and the distance of 1 mm is the thinnest we could go without the sample falling apart during sawing.

The complete cylinder in which the slot has been cut is now fixed in a precision saw and further cut with the diamond saw into a slice. The slot made in the stainless steel guides now the saw blade very precisely by hindering blade bending. In this way a reasonably flat and plan-parallel slice is obtained.

The obtained slice is fixed in a specially developed holder and further polished with a 600 grit polishing paper into fat shiny surfaces (precision of 0.01 mm) and parallel to each other (within an accuracy of 0.02 mm). The roughnesses and filaments of the stainless steel are polished away with a file, and four small slots are made in the top of the ring to ease brine transport.

4.2. Experimental set up

To prepare an experiment first the sample and the brine have to be prepared, then the safety valves have to be adjusted to the wished pressure taking into account the temperature rises. Assemblage of the apparatus with the sample then takes place.

The order of assemblage of the whole system begins by screwing the 3 legs under the aluminium plate. Then the two tubes connecting the top and down of both systems are made to cross the aluminium plate. Now, in an inverted apparatus, the following pieces and in the following order have to be set within the lower chamber of the sample housing: an O-ring greased with silicon fat, a glass window, an arlon ring and the bottom cover. Now the three connectors with their correspondingly greased O-rings must be screwed into the side wall of the sample housing.

The apparatus is now turned back to a normal upside position, and the whole assemblage is fixed with the 8 bolts which traverse the bottom cover and the aluminium plate and which are fixed at the down side of the aluminium plate by the 8 nuts.

Two of the three connectors are now attached to the connecting tubes which had been made previously to cross the plate. The third connector of the casing is coupled to a tube which on the other side is communicated with a tap which is attached to the plate.

In the top side of the plate the two manometers are coupled to their respective tubes and fixed on top of the aluminium plate, the four heating elements are fastened on the top and down side of the plate and the two remaining taps and security valves are coupled to their corresponding pressure system and further fixed to the plate.

To start an experiment a sample is placed inside the sample housing on top of he lowest glass window, then the mid glass window is placed on top of the sample. This mid window constitutes with the lowest window the lower chamber, and is closed using an O-ring which is greased with silicone fat. On top of this an equally greased O-ring and the top glass window are placed. The complete assemblage is then closed by the synthetic ring (of arlon) and the metallic top cover. Then the nuts are tightened.

In the only tap for the gas system an argon bottle is now placed and the system brought up to the wished pressure. After this, both the bottle of argon and the pressure system are closed and uncoupled. It has to be taken into account that when the temperature of the system rises, the gas pressure will rise as well, and therefore a pressure lower than that desired has to be given to the system.

The brine container with pump has to be coupled now to the upper tap of the brine system and the reservoir has to be filled by about 3/4 of its capacity. The tap has to be closed during this procedure. At the lower side of the plate, the brine tap has to be coupled to a tube which in its turn is coupled through a threeway valve and a Dresler bottle to a vacuum pump.

With the lower tap opened it is now proceeded to vacuum pump the brine system during about 2 minutes, after what the upper tap with the brine reservoir is shortly opened (and closed). As a consequence a little brine appears in the Dresler bottle showing that if any air was present it has been taken out of all the volume between the reservoir and the valve. The brine reservoir can again be filled in up to 3/4 of its capacity.

Now, after 5 minutes more of vacuuming, the top tap is opened and the brine enters the system. When the brine reaches the Dresler bottle the lower tap has to be closed. The system is now full of brine (in spite of the gas pressure) and the vacuum pump with accessories can be taken away.

The complete apparatus, including the brine reservoir is now placed in the stainless steel box, the thermocouple set in the sample casing, the electrical connections for temperature control are made and the apparatus isolated with the rockwool.

The temperature regulator is now set to reach 100°C at the maximum heating speed. When the temperature reaches 60°C the potence of the apparatus is lowered by 40 %, and at 80°C by an additional 20%. These last changes are performed by hand what makes it possible to reach the wished temperature in about 30 min without producing much overshoot. When the temperature reaches 100°C the system can be pressurized using the spindle. The brine reservoir is then taken away and the tip of the tap is cleaned with water. To be able to perform the last handlings, the apparatus has to be taken out of the box. After replacing it in the box and coupling again the electricity connections the two upper lamps are placed and the isolation as well. After 45 min the apparatus has reached the wished temperature.

During the experiment the pressure of the brine and gas systems can be read in the manometers and the state of the sample can be photographed through the holes in the box.

5. TESTS AND MODIFICATIONS

The *temperature gradient* of the apparatus has been measured during a simulation of operation and it has been found that there may be differences of 3°C between different devices. The gradient in the air inside the box near the apparatus is higher. No variation in the gradient could be measured as a consequence of opening the lid of the holes for illumination and reading of the pressure, nor as a consequence of heat produced by the lamps.

Also the *maximum pressure* which can safely be used has been determined with tests. The experiments consisted of filling the system with gas and observing at which pressure the glass windows break. The lower glass window breaks for a pressure on the sample of 195 bar which is reached at a gas pressure of 150 bar and the upper glass window breaks at a gas pressure of 240 bar. The difference in gas pressure which can be withstanded by the glass plates is due to the size and form of the mid plate: the mid plate presses the sample in a surface smaller than its upper surface by a factor 1.3 (see Fig. 2).

The fractures in the glass windows are evidently produced by bending. They pass through the centres of the windows, no pieces are blown apart and the glass remains gastight.

Leakings were repeatedly found when testing the system and they were localized and helped in some different ways. For instance, the *safety valves* for both pressure systems are completely made of stainless steel (316). The closure pin of the valve which is in contact with brine corroded during the tests and leaking appeared at the O-ring of the valve. To avoid this the pin was changed by a self made Arlon pin. The swage-lock fittings also had to be modified.

To be certain that the system is gastight the whole was, before a sample was set together, filled with argon at a pressure of 100 bar through one of the taps of the system while all the other were smeared with leak fluid.

Both systems could be tested together if connected to each other by means of suppressing the mid glass window and O-ring.

We performed many additional tests to know whether the gas from the gas chamber leaks to the sample chamber, this was necessary because there is a drop in the pressure of the gas chamber of about 5 bar within three months.

There were two weak places where leaking could take place, either between the O-rings and the housing or between the glass and the glued metal rings. The last was not the reason, because substituting the glass by a metal plate the "leaking" went on. The only possibility would thus be the O-ring leaking. This was controlled by performing a test on the housing independently with only the upper and mid glass windows. Between these two windows helium was set instead of the usual argon and then an helium leaking test was performed. The measured leaking was very small (2×10^{-9} l/min) en took place after 30 min waiting between each measurement, and yet this leaking, given the size of the sample and gas chamber can justify the pressure drop in the gas system. Therefore experiments with silicon oil instead of with argon were performed since it was expected that less leaking would take place for these bigger molecules but, the changes in pressure remained the same.

The leaking problem has remained unsolved. As a last attempt to solve the problem, an accumulator was made (see red cylinder in the photograph) which was coupled to the communication tube of the brine system. These accumulator is in principle made of a rubber balloon which is coupled to the brine system and will be filled in when the system is filled in with

brine. If the brine pressure in the inner system grows the balloon will increase in volume reducing the pressure in the system. The balloon is surrounded by a stainless steel cylinder where the air is set under pressure by the balloon and which can be let out if needed. It is also possible to increase the pressure of the brine by increasing that of the air in the cylinder. The accumulator did not change anything in the pressure equilibrium, although it certainly increased the system safety.

6. DISCUSSION

The apparently unnecessary complications in the system of pressurizing and heating are due to the wish of avoiding sudden temperature changes when the temperature of the experiments is set near 100°C. The brine has to be introduced in a previously heated apparatus and at the temperature of the apparatus as well. This is necessary to avoid precipitation of the salt dissolved in the brine in the cold apparatus, what would not only disturb the saturation degree at which the experiment ought to take place, but probably also obstruct the ducts with salt crystals. On the other hand, if the brine is introduced at 100°C, in a system which has been vacuum pumped, it will certainly boil producing gas bubbles which disturb the experiment. Notice that if the system has not been vacuum pumped the brine will not penetrate the apparatus due to the pressure of 200 bar exerted by the gas chamber on the sample chamber.

Although the leaking measured is not really relevant for some experiments (a drop of 5 bar in three months is rather good), there is always the possibility of the gas bubbles influencing the recrystallization by poisoning the grain boundaries : It is known that depending on the size of gas bubbles included in the brine the grain boundaries can advance easily or not. This is why we insisted as much in solving the leaking problem.

7. RESULTS AND FURTHER SUGGESTIONS

Five experiments have been carried out and photographs of the samples were taken, however, although little could be observed since in each experiment one or another problem would appear, we were able to control the importance of surface tension in driving grain boundary migration. Mostly leaking problems forced the experiments to stop. Now we think that it would

be worth trying again and accepting the leak between the gas system and the brine system. Anyway, if we would continue this research we would introduce two more changes. First we would make all the pressure systems and casing from Inconel what also means that the sizes of the tubes would have to be changed to a diameter of 1/4 " (6.35 mm) because in Inconel these are easier to purchase than tubes (as the stainless steel tubes used now) with a diameter of 6 mm. And second, we would also have to heat the air instead of the aluminium plate to achieve a yet smaller temperature gradient.

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METHODOLOGY OF IRRADIATION EXPERIMENTS WITH GROUND NATURAL ROCK SALT SAMPLES PERFORMED AT GSF-IFT

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ABSTRACT

The formation of radiation damage in natural rock salt samples from the Asse was investigated with special regard to the chlorine development and its correlation with both sodium colloid development and stored energy deposition in NaCl. In addition, the radiation-induced formation of gases and the release of gases from the rock salt was studied. The experimental set-up for performing these irradiations and the analytical methodologies used are presented.

1. INTRODUCTION

The emplacement of canisters containing vitrified reprocessed high level radioactive waste in boreholes in rock salt will result in an exposure of the host rock to temperatures up to 200 °C and gamma dose rates of 1000 Gy/h. The total dose in a repository is estimated to be in the order of 10^9 Gy. Apart from the formation of radiation damage in rock salt, gamma radiation leads to the formation and release of gases from the rock salt.

We determined the formation of radiation damage in natural rock salt samples from the Asse with special regard to the chlorine development and its correlation with both sodium colloid development and stored energy deposition in NaCl. Since these irradiations were carried out in sealed glass ampoules it seemed worthwhile, also to measure the radiation-induced gas formation in and gas release from these samples in order to allow comparison with previous data. The experimental set-up for performing the irradiations and the analytical methodologies used are described here in some detail. The results of these investigations are presented by Jockwer et al. (1995) [article 13 in this volume] and by Mönig et al. (1995) [article 16 in this volume], respectively.