# Fibre Textures in Extruded Salt

W. Skrotzki* and P. Welch
*Institut für Metallphysik, Universität Göttingen and*
*Institut für Metallkunde, Technische Universität Clausthal,*
*Federal Republic of Germany*
* *At present: Department of Materials Science and Engineering, Cornell University*
*Ithaca, NY, USA*

## ABSTRACT

*The preferred orientation developed in extruded salt is studied as a function of extrusion temperature. In 'pure' salt a ⟨100⟩⟨111⟩ double fibre texture is found for all investigated temperatures. In rock salt the ⟨100⟩ fibre is replaced by ⟨115⟩ around room temperature. The temperature dependence of the pole intensities together with microstructural investigations suggest the ⟨100⟩ and ⟨115⟩ components to be primarily due to dynamic recrystallization. The ⟨111⟩ deformation texture agrees with model calculations based on slip on {110}⟨110⟩ and {100}⟨110⟩ systems, generally observed as primary and secondary slip systems, respectively. Connections with the diapirism of salt domes are discussed.*

## INTRODUCTION

Differences in density of evaporites and covering rocks lead to a halokinetics, i.e., to a salt impetus. During this process the superposed covering layers are torn open and salt intrudes into the clefts and widens them gradually. In this way salt domes are assumed to be generated (Jaritz, 1980). Halokinetics leads to a deformation of rock salt, which in the stage of diapirism reaches strain rates of the order of $10^{-11}$ $sec^{-1}$ (Heard, 1972; Albrecht and Hunsche, 1980). The deformation may also lead to the development of a specific rock texture. Because direct texture analyses of natural rock salt are rather extensive due to the large grain size, it was the purpose of the present work to study the texture of extruded small-grained salt. Such investigations may give an indication of the texture to be expected in salt domes, because the formation of salt diapirs strongly resembles an extrusion process. The knowledge of the texture in salt domes is not only of geological interest but may also have practical relevance, e.g., for an estimation of the thermomechanical processes taking place around a deposit of high-active nuclear waste. Besides these aspects there also was an interest in checking the texture predictions of Chin and Mammel (1973) for axisymmetrically deformed NaCl-type ionic crystals. Their calculations based on {110}⟨110⟩ and {100}⟨110⟩ slip required for polycrystalline plasticity (Skrotzki et al., 1981) show that for axisymmetric tension assumed to be prevalent in extrusion (independent of the value of the plastic anisotropy, i.e., the ratio of the critical resolved shear stresses for slip on {110} and {100} planes), the axis rotates toward [111], yielding a [111] fibre texture.

## EXPERIMENTAL

'Pure' NaCl polycrystals were prepared at room temperature from reagent grade powder by compression to cylindrical blocks (density > 98%). These blocks as well as natural rock salt from the Asse mine (FRG) have been extruded at different temperatures $T_{ex}$ with an extrusion ratio 14/1 and a strain rate in the order of 1 $sec^{-1}$. $T_{ex}$ is the temperature of the samples before extrusion. The high-speed extrusion causes a noticeable temperature increase in the rods. All hot extruded samples were cooled to room temperature in air. The orientation distributions of the grains of the extruded rods were determined by neutron diffraction. The quantitative inverse pole figures were calculated using the 'Orientation Distribution Function' method (Bunge, 1981).

## RESULTS

Extrusion of NaCl polycrystals above room temperature leads to a ⟨100⟩⟨111⟩ double fibre texture, the fibre intensities changing with temperature. Figure 1 shows typical inverse pole figures of synthetic and natural rock salt at different temperatures. A similar behaviour is also

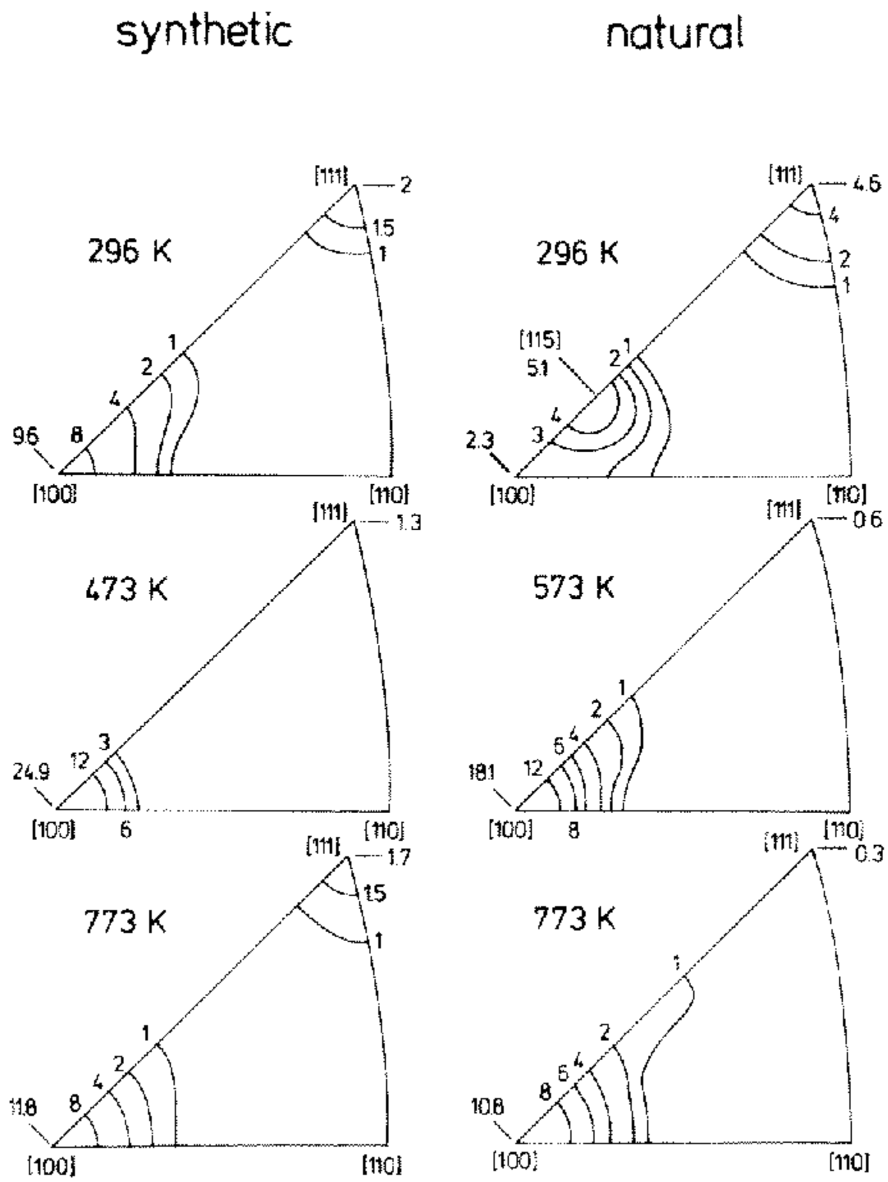


**Figure 1.** Inverse pole figures of 'pure' synthetic and natural rock salt extruded at different temperatures. Each contour is the locus of equal normalized fibre axis density; a value of unity corresponds to a random distribution.

found for other ionic crystals (Skrotzki and Welch, 1983). The temperature dependence of the intensities of the [100], [111] and [115] poles is given in Figure 2. The [100] fibre intensity runs through a maximum at a certain temperature $T_0$ where the [111] fibre has a minimum. There is qualitative agreement between the textures of 'pure' and natural NaCl except at the lowest temperatures investigated. Around room temperature the ⟨100⟩ fibre is replaced by the ⟨115⟩ component. Furthermore, $T_0$ is shifted to a higher temperature relative to pure NaCl; also the maximum intensity of the ⟨100⟩ fibre is lower and the ⟨111⟩ intensity exceeds that of 'pure' salt at low temperatures. Figures 3 and 4 show the intensity of poles lying on the border of the standard stereographic triangle between [100] and [111]. It can be seen, especially for 'pure' salt (Figure 3), that the double fibre texture is sharpest at $T_0$.

Figures 5 and 6 show the influence of temperature on the microstructure of extruded 'pure' and natural salt, respectively. For 'pure' NaCl the grain size depends on the extrusion temperature, being a minimum at $T_0$. This indicates that the structure has recrystallized. With increasing temperature the recrystallized fraction of the deformed structure increases. Above $T_0$ grain growth sets in. For natural rock salt the microstructural development is different. At low temperatures (up to ~400 K) the de-

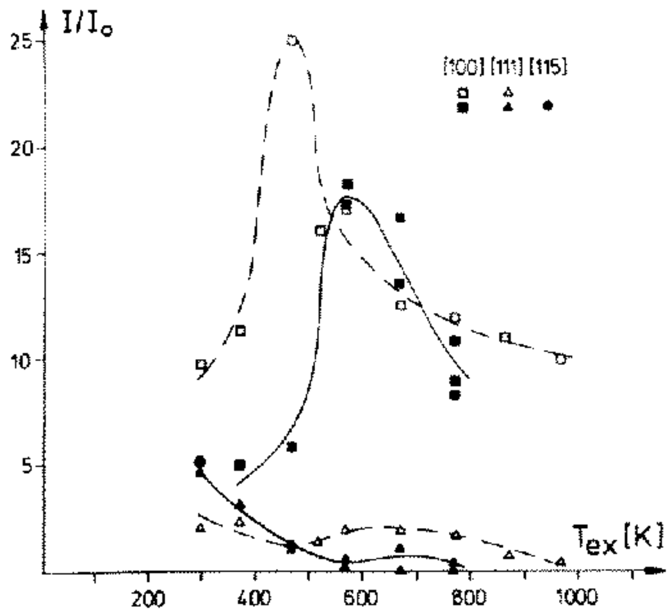


**Figure 2.** Temperature dependence of the intensities of the preferred fibres in extruded 'pure' (open) and natural salt (filled symbols). Intensities are given in units of a random orientation distribution $I_0$.

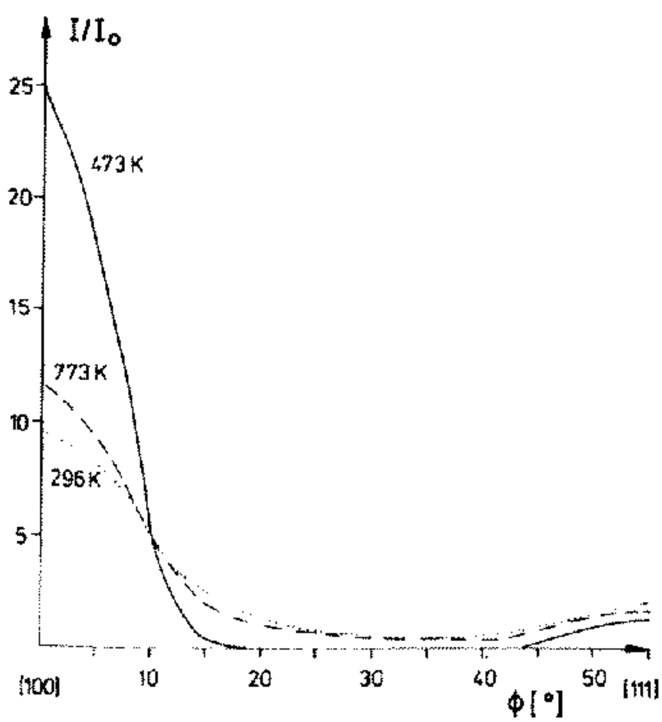


**Figure 3.** Intensity of the poles lying on the border of the standard stereographic triangle between [100] and [111] for 'pure' NaCl (in units of a random orientation distribution $I_0$).

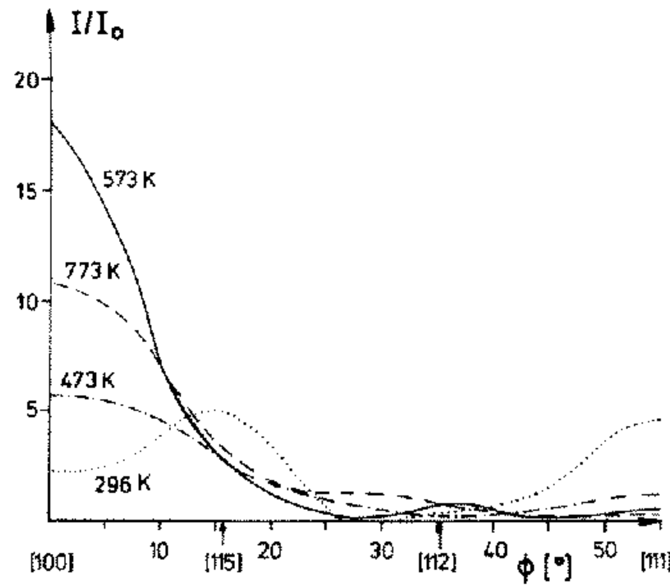


**Figure 4.** Same as Figure 3 for natural rock salt.

formed structure is partly retained. The grain boundaries of the elongated large original grains can be seen clearly. In some grains, however, recrystallization has taken place. The newly formed grains first have a cubic shape and are oriented nearly parallel to the extrusion axis. This shape is maintained during growth into the deformed matrix but is changed as other grains are met. Above ~500 K the deformed matrix is totally recrystallized. In addition, the following effect is observed. Impurities precipitated along the grain boundaries, probably as an anhydrite phase, as have been found by electron microprobe analysis, prevent the newly formed grains from lateral growth (Figure 6: 573 K). The grain growth occurs only within the elongated original grains. In this way an equiaxial recrystallized microstructure is obtained, the long axis coinciding with the extrusion direction. At higher temperatures the impurities no longer act as obstacles for grain boundary migration (Figure 6: 773K). At this point, the microstructure becomes isotropic.

## DISCUSSION

Extrusion of salt at the temperatures used is connected with a texture that is drastically changed by recrystallization. At low temperatures the deformation texture is found to be a ⟨111⟩ fibre texture. With increasing temperature a ⟨100⟩ fibre develops by recrystallization. The intensity of this fibre can exceed that of ⟨111⟩ by an order of magnitude. When recrystallization is suppressed by impurities, as in the case of natural rock salt, the deformation texture is more pronounced and the development of the ⟨100⟩ recrystallization fibre starts only at higher temperatures. The ⟨115⟩ component replacing

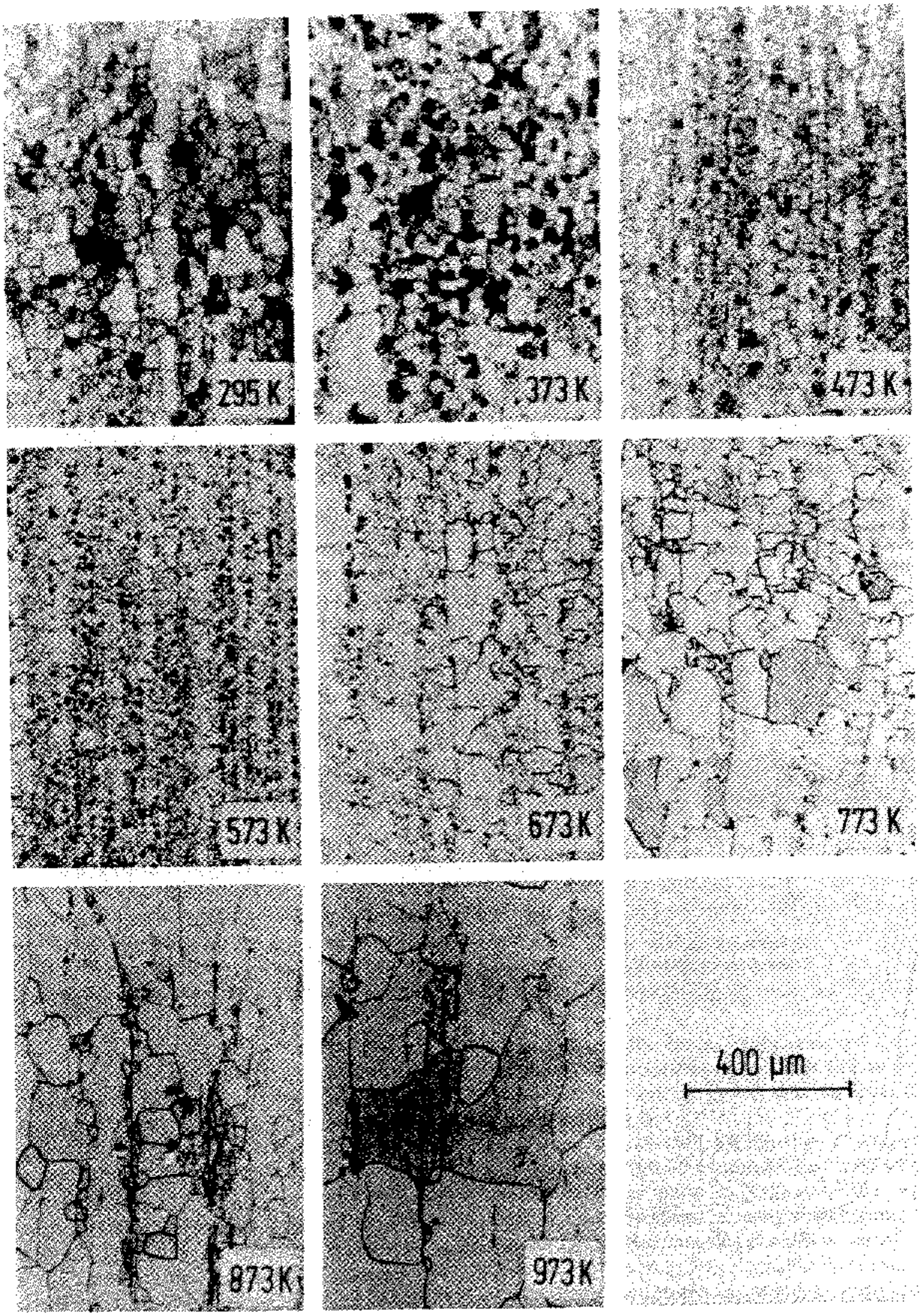


**Figure 5.** Influence of temperature on the microstructure of extruded 'pure' NaCl parallel to the extrusion axis.

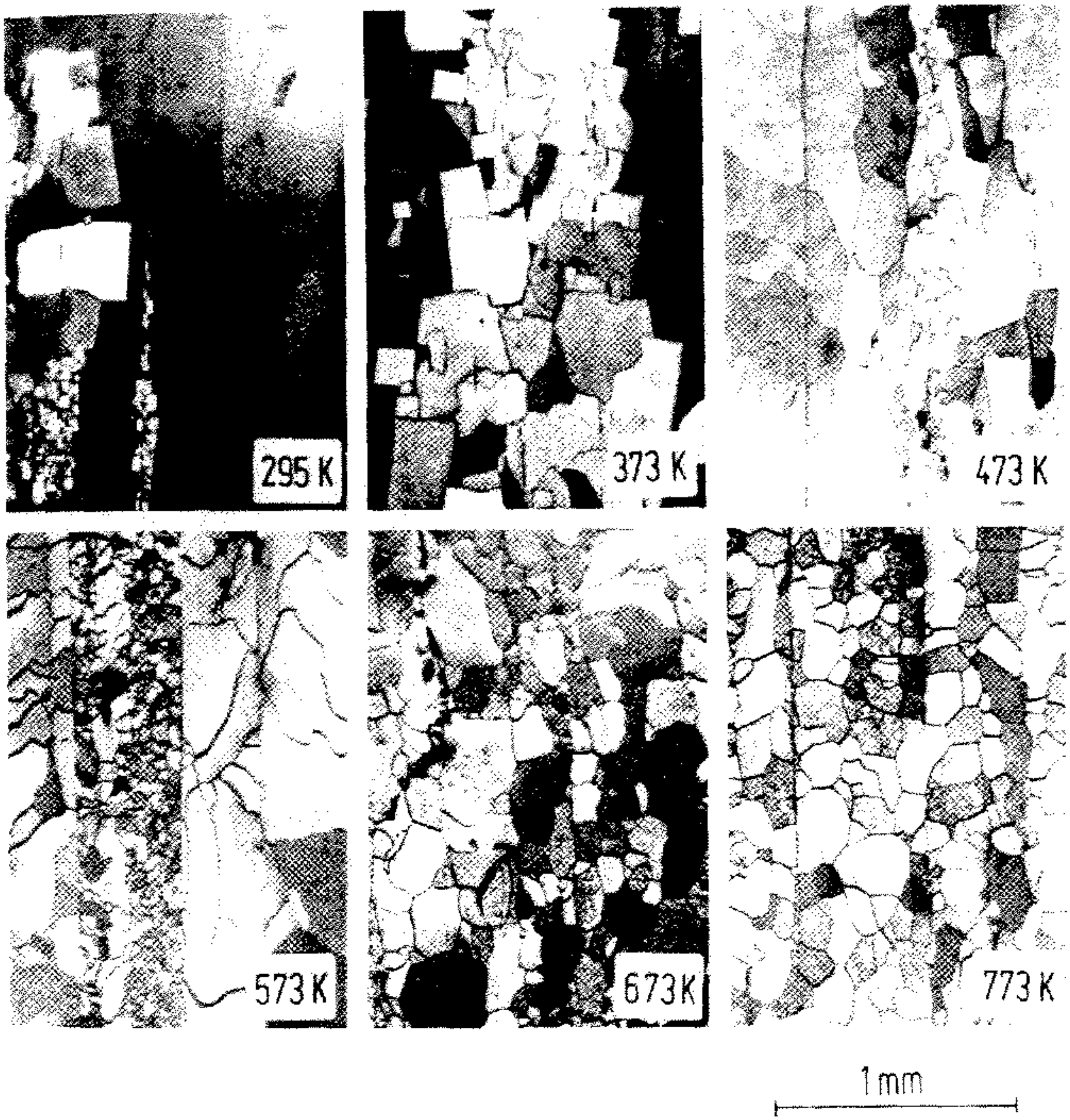


**Figure 6.** Same as Figure 5 for natural rock salt.

⟨100⟩ at low temperatures in rock salt is attributed to isomorphic grain growth.

These results show that the previous discussion of the double fibre texture obtained for 'pure' ionic crystals at $T/T_m \approx 0.5$ ($T_m$ = melting temperature) and assumed to be the deformation texture (Frommeyer et al., 1981) no longer holds. According to our new extended investigations the prediction of a ⟨111⟩ fibre texture on the basis of {100}⟨110⟩ and {100}⟨110⟩ slip by Chin and Mammel (1973) (Figure 7) is supported. Considering collinear slip, i.e., arranging the twelve {110}⟨110⟩ and {100}⟨110⟩ slip systems into six pairs, each pair sharing a ⟨110⟩ slip direction, an unambiguous lattice rotation toward ⟨111⟩ is obtained (Figure 8). Collinear slip, which may be considered as net {111}⟨110⟩ slip, may also explain the parallel texture development in extruded fcc metals (McHargue, 1959).

As has been mentioned at the beginning, diapirism

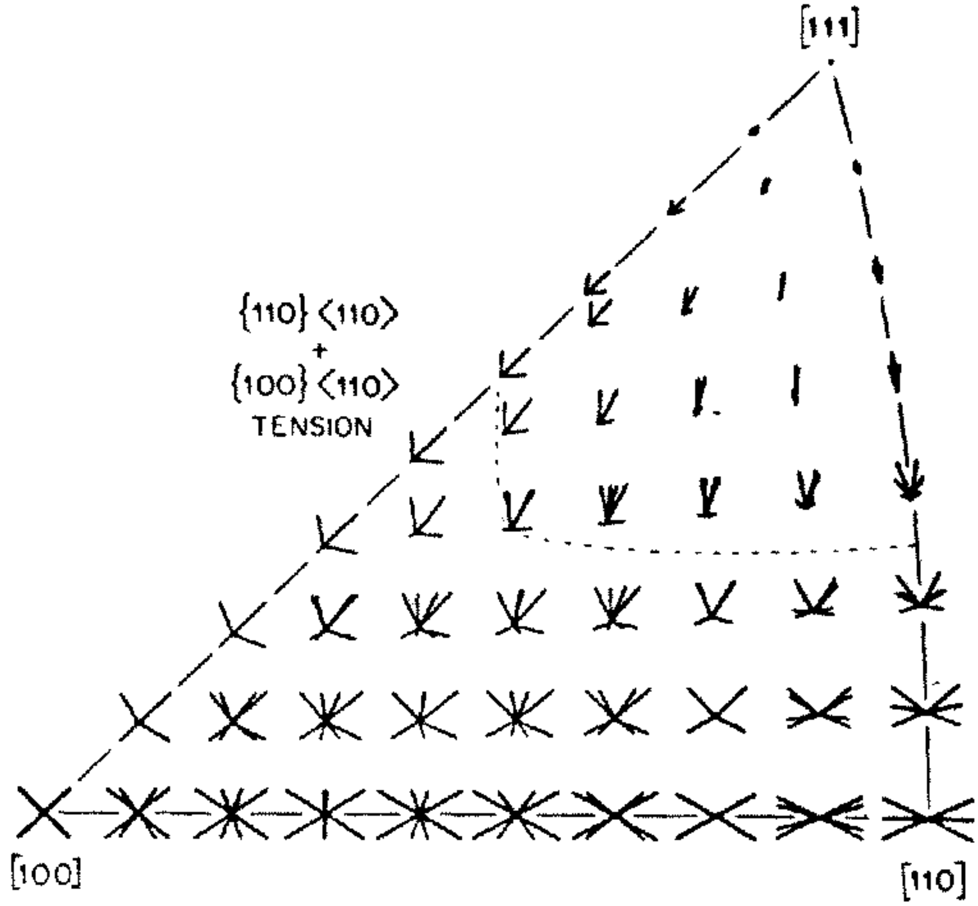


**Figure 7.** Lattice rotation in axisymmetric tension for {110}⟨110⟩ + {100}⟨110⟩ slip. Axial orientations in region near [111] rotate toward [111]; those in remaining region can rotate to any of the three corners, though preference is for [111]. (After Chin and Mammel, 1973).

strongly resembles an extrusion process. Thus, our results suggest a double fibre texture near ⟨100⟩ and ⟨111⟩ in salt domes in the direction of flow, i.e., in vertical direction. Because of the low strain rates and temperatures of about 350 K during diapirism the recrystallization components may be dominant. Indeed, Schwerdtner (1966) and Goeman and Schumann (1977) report a preferred orientation of ⟨100⟩ found in halite rocks of Gulf Coast domes (U.S.A.) and of the Niedersachsen-Riedel pit near Wathlingen (FRG), respectively.

Finally, it should be noted that a texture in salt domes will lead to an anisotropic behaviour when strained thermomechanically. Assuming a predominant [100] fibre in vertical direction, this direction will be the weakest. This prediction should be checked on selected domal salt and necessarily taken into account in estimations concerning the safety of disposals for nuclear waste.

## ACKNOWLEDGMENTS

We would like to thank Prof. P. Haasen and Dr. G. Frommeyer for their current interest in this work. Thanks are also due to the Gesellschaft für Strahlen und Umweltforschung mbH München for support (W. S.). The neutron diffraction measurements were performed at the Hahn-Meitner-Institut in Berlin (West).

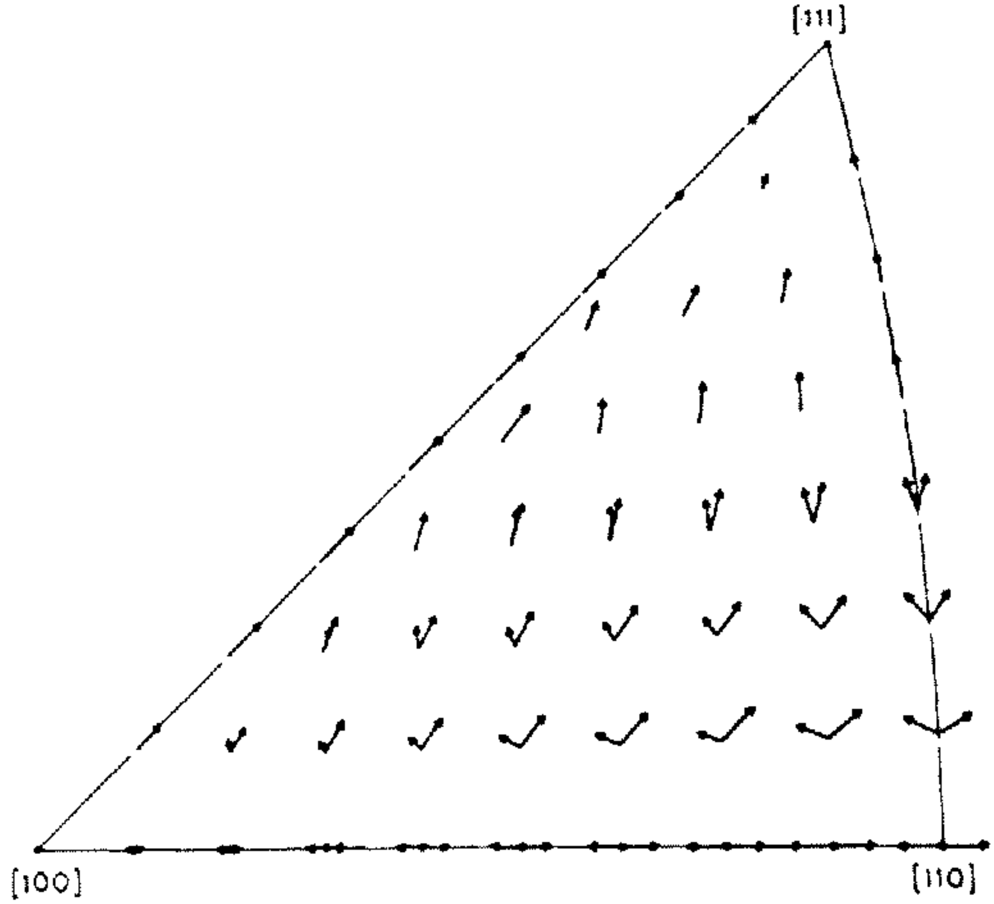


**Figure 8.** Same as Figure 11, except that the activity of collinear slip systems has been maximized. Unambiguous rotation toward [111]. (After Chin and Mammel, 1973).

## REFERENCES

Albrecht, H. and U. Hunsche. 1980. Gebirgsmechanische Aspekte bei der Endlagerung radioaktiver Abfälle unter besonderer Berücksichtigung des Fließverhaltens von Steinsalz. Fortschr. Miner., v. 58:212-247.

Bunge, H. 1982. Texture Analysis In Materials Science: Mathematical Methods, 2nd Edition [Peter Morris, Translator] Butterworths, London.

Chin, G. Y. and W. L. Mammel. 1973. A theoretical examination of the plastic deformation of ionic crystals: II. Analysis of uniaxial deformation and axisymmetric flow for slip on {100}⟨110⟩ and {100}⟨110⟩ systems. Met. Trans. v. 4:335-340.

Frommeyer, G., P. Welch and W. Skrotzki. 1981. Extended plasticity and fibre textures in axisymmetrically deformed ionic polycrystals with the NaCl-structure; Proc. of the 6th Internat. Conf. on Textures of Materials, Tokyo, pp. 1032-1037.

Goemann, U. and H. Schumann. 1977. Röntgenographische Gefügeuntersuchung an einer orientiert entnommenen Probe von grobkörnigem Steinsalz; Tschermaks Min. Petr. Mitt., v. 24:179-190.

Heard, H. C. 1972. Steady-state flow in polycrystalline halite at pressure of 2 kilobars. In: Flow and Fracture of Rocks, American Geophysical Union, Washington, D.C., pp. 191-208.

Jaritz, W. 1980. Einige Aspekte der Entwicklungsgeschichte der norddeutschen Salzstöcke; Z. dt. geol. Ges., v. 131:387-408.

McHargue, C. J., L. K. Jetter and J. C. Ogle, J.C. 1959. Preferred orientation in extruded aluminium rod; Trans MS AIME, v. 215:831-837.

Schwerdtner, W. M. 1966. Intragranular gliding in domal salt; Tectonophysics, v. 5:353-380.

Skrotzki, W., G. Frommeyer and P. Haasen. 1981. Plasticity of polycrystalline ionic solids; Phys. Stat. Sol. (a), v. 66:219-228.

Skrotzki, W. and P. Welch. 1983. Development of texture and microstructure in extruded ionic polycrystalline aggregates; Tectonophysics, v. 99:47-61.