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TEXTURE DEVELOPMENT IN EXPERIMENTALLY DEFORMED CALCITE ROCKS

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INTRODUCTION

Limestones and marbles are frequently found in deformed regions of the Earth's crust. An understanding of their rheological properties which are linked with particular deformation mechanisms would contribute significantly to the elucidation of the kinematic and dynamic aspects of orogenic events. The application of laboratory data on the rheological behaviour of a rock to geological problems requires an extrapolation over many orders of magnitude in strain rate frequently coupled with an extrapolation in temperature and/or stress. It has been shown by previously published work on the rheology of calcite rocks that different deformation mechanisms and consequently different flow laws exist for (i) different calcite rocks, whereby the grain size plays a major role in determining the deformation mechanism and (ii) for the same rock under different conditions of stress, strain rate and temperature (Heard and Raleigh /1/, Rutter /2/, Schmid et al. /3/). The textures developing during experimental rock deformation as well as in natural rock deformation, together with the microstructures, are useful in elucidating the deformation mechanisms in both these environments. Thus, the main purpose of this paper is to relate the textures developed in two calcite rocks of different grain size to their rheological and microstructural characteristics.

MATERIALS

The materials chosen for the study were Solnhofen limestone and Carrara marble. Solnhofen limestone is a fine grained ,5µm, limestone with only minor amounts of quartz and clay (Wenk et al. /4/, Schmid et al /3/). Siemes /5/ and Wenk et al. /4/ showed that the initial texture is axially symmetric about the pole to bedding. This enables the initial texture to be represented as an inverse pole figure for the pole to bedding. The initial textures for the Solnhofen limestones used in this study are shown in fig. 4a, 4b.

Carrara marble is a calcite marble of coarser grain size, 200µm, than Solnhofen limestone. It has no significant initial texture (Owens and Rutter /6/, Ramez and Murrel /7/).

APPARATUS AND TECHNIQUE OF EXPERIMENTAL DEFORMATION

The deformation experiments at temperatures up to 500°C were carried out at Imperial College using apparatus similar to that of Heard /8/ and at higher temperatures using apparatus designed by Paterson /9/ and /10/, at the Australian National University, Canberra. Full descriptions of the techniques are to be found in Rutter /11/ and Schmid et al. /3/. Briefly, the equipment at Imperial College comprised a set of externally heated, liquid (silicone oil) confining medium machines whilst the A.N.U. equipment was internally heated with Argon gas as the confining medium. Fluid confining medium equipment was used (i) to avoid the possibilities of severe damage to the specimen which can occur during off-loading and cooling of weak material, such as calcite rocks after deformation in solid confining medium apparatus and (ii) to enable accurate strength determination down to a few tens of bars.

All the deformation experiments were carried out at constant displacement rate corresponding to constant compressional strain rates in the range 10^{-2}/sec. to 10^{-6}/sec. Confining pressure conditions were either 1.5Kb or 3.0Kb. For Solnhofen limestone the specimens were usually cored with the axis of the cylindrical specimen perpendicular to the bedding planes and thus parallel to the symmetry axis of the depositional texture. The Carrara marble specimens were all cored in a constant direction from a block which showed no discernible planar or linear features.

RHEOLOGY AND DEFORMATION MECHANISMS

Up to 300°C Solnhofen limestone has a very weak strain rate dependence of its strength and deformation twinning on <u>e</u> planes is the dominant microstructure. From 400°C onwards twinning is almost completely absent (Rutter /12/, Schmid et al. /3/). Rheologically three flow regimes, Fig. 1, can be established (Schmid et al. /3/):

--regime 1: At stresses above 1900 bars at 400°C to 600°C an exponential relationship between stress and strain rate is observed.

--regime 2: A power law with a stress exponent of 4.7 holds in an intermediate stress region.

--regime 3: Below a temperature dependent transition stress (300 bars at 900°C and 1000 bars at 600°C) the stress exponent drops to around 1.7.

In regimes 1 and 2 the deformation is essentially achieved by intracrystalline slip on as yet unknown slip systems with no twinning activity. Microstructurally no obvious differences between regimes 1 and 2 were found. Regime 3 has all the characteristics of superplastic flow and grain boundary sliding is the dominant deformation mechanism. Intracrystalline slip occurs simultaneously as a subordinate process and amounts to about one third of the strain according to microstructural investigations (Schmid et al. /3/). From SEM (Rutter /11/) it can be shown that twinning contributes to the deformation of this rock at low (300°C) temperature.

Carrara marble exhibits a very weak strain rate sensitivity of strength at very low temperatures and an exponential relationship between stress and strain rate is observed at 400°C to 500°C. Although a similar flow law to regime 1 in Solnhofen limestone is observed, twinning in Carrara marble persists as a dominating deformation mechanism up to at least 500°C, probably in combination with dislocation slip (Rutter /12/). There is a tendency for twin lamellae to become broader and fewer at higher temperatures and for irrational twin boundary orientation to develop near grain boundaries.

Thus the three different deformation mechanisms such as (i) intracrystalline deformation with a large contribution from <u>e</u> twinning (ii) intra-

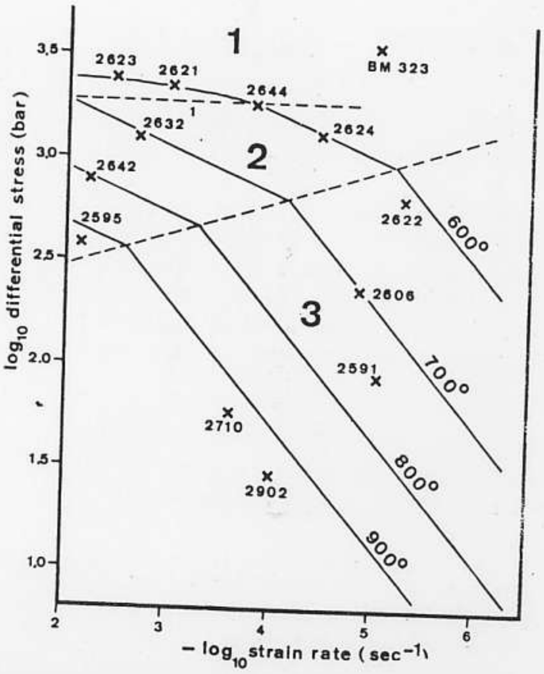


Fig. 1 Log-log plot of differential stress at 10% strain vs. strain rate for Solnhofen limestone. Taken from Schmid et al. /3/.

crystalline by dislocation motion alone and (iii) superplastic behaviour can be expected to produce distinct textures.

X-RAY TEXTURE ANALYSIS

X-ray texture measurements were carried out in transmission mode on thin, 70μm or 50μm, specimens cut parallel to the compression axis. The equipment used was either the McLean texture goniometer at Leeds (Frost and Siddans /13/) or the Philips texture goniometer at Canberra. For the Solnhofen limestone specimens analysed at Leeds a single scan was made at zero tilt, step scanning the specimen in its own plane through 180° in intervals of 5° or 6°. Data were collected by accumulating counts for a preset time at each data point. The counting times were chosen so that there was a difference of at least 1000 counts between measurements taken on the diffraction peak and on the background close to the peak. Since the specimen was not tilted out of the Bragg angle the diffracting volume of the specimen and the path length of the X-ray beam within the specimen remained constant. Thus no absorption correction was necessary. The variation of the background with specimen rotation was measured, using a data interval three times that of the peak data.

Provided that the grain size exceeds 30μm, standard universal stage optical methods of texture determination can be applied to calcite rocks, Turner and Weiss /14/. For finer grained rocks X-ray methods are essential and were used for this study. Whilst X-ray texture goniometry proved ideal for the fine grained Solnhofen limestone, the large grain size of the Carrara marble caused sharp maxima in the scans. In an attempt to smooth out these spikes measurements of peak and background variation through 360° were produced on a chart recorder. The charts were digitized ready for computer processing using a D-mac 1b digitizing table interfaced to a Digico M16V mini-computer.

The method of X-ray analysis used for the Solnhofen limestone specimens at A.N.U. is described by Schmid et al. /3/.

Data were collected for between 6 and 8 diffraction peaks per specimen. For a given specimen the number of peaks measured and the data collection interval, where appropriate, were chosen with regard to the anticipated sharpness of the texture. For sharper textures more peaks were measured at smaller data intervals. The peaks used are shown in Table 1 which was taken from Wenk et al. /4/. A stereogram of the calcite peaks used in this study is shown in Fig. 2.

The data were processed on the ICL 1906A computer at the University of Leeds. The techniques are described by Bunge /15/, Baker et al. /16/ and Casey and Whalley /17/ and are summarised below.

1) Calculate background below peak.
2) Subtract background data from peak data.
3) Fit a cubic spline function to the unnormalised data.
4) Perform step-wise analytical integration of the product of the data, represented by the spline function, and Legendre polynomials in Fourier series form.
5) Normalise the data and Legendre polynomial coefficients.
6) Calculate the orientation distribution function.
7) Calculate the inverse pole figure.

In calculating the orientation distribution function it was found that a maximum order of expansion of 12 was appropriate to the accuracy of determination of the coefficients of the Legendre polynomials.

RESULTS

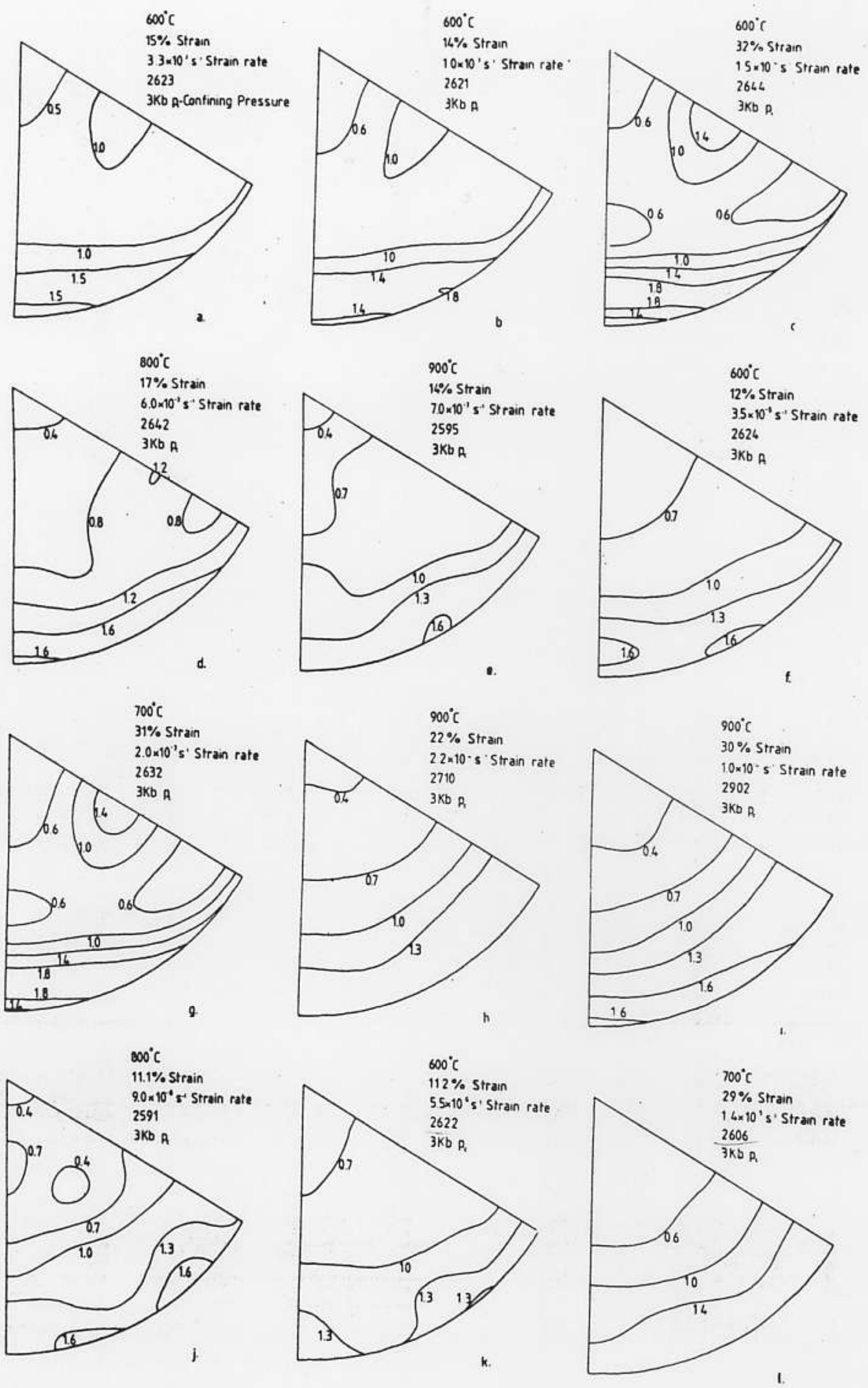
Inverse pole figures for the textures are shown in Figs. 3 and 4. All figures are upper hemisphere equal area stereographic projections of the asymmetric sector for calcite.

a) Solnhofen limestone

The condition of deformation for the Solnhofen limestone specimens are shown on a stress strain rate plot in Fig. 1.

The Solnhofen limestones deformed in flow regimes 1 and 2 develop a characteristic pattern, Fig. 3a-g. There is an equidimensional maximum in the region of the e plane normal and a ridge running from the a to h plane normals. Directions close to the f plane normal have a very low concentration of compression axes. Directions close to the r plane normal also show a reduction in concentration. There is no discernible difference between textures developed in flow regimes 1 or 2.

The texture developed in Solnhofen limestone specimens deformed in flow regime 3, Fig. 3 h-l, is relatively weak at comparable strains and does not differ in symmetry from that of the undeformed material. There is no tendency to develop a maximum near e or a minimum near r. In one specimen, 2902 Fig. 3 i, a ridge from a to h has developed.



Regime 1 & 2

Regime 3

Fig. 3 Inverse pole figures for specimens deformed at A.N.U.

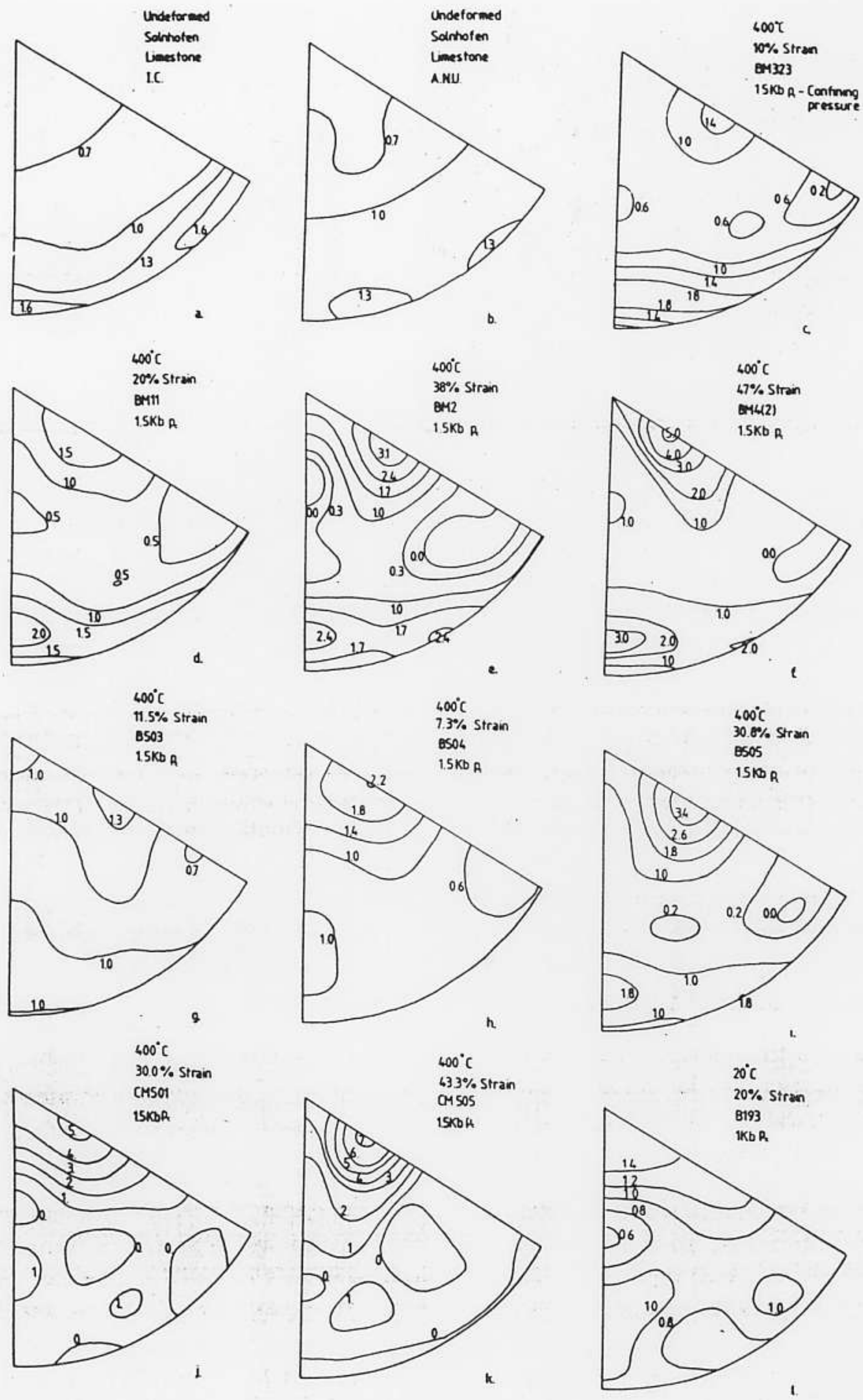


Fig. 4 Inverse pole figures for specimens deformed at I.C.

Table 1

Diffraction peak number	Diffraction angle $2\theta(^{o})$	Bravais Indices	Spherical angles $\theta(^{o})\phi(^{o})$	Intensity contri-bution
1.	26.80	$\{02\bar{2}1\}$,f	63.1 60.0	1.0
2.	34.28	$\{10\bar{1}1\}$,r	44.6 0.0	1.0
3.	36.60	$\{0001\}$,c	0.0 0.0	1.0
4.	42.02	$\{11\bar{2}0\}$,a	90.0 30.0	1.0
5.	46.08	$\{44\bar{8}3\}$	66.3 30.0	1.0
6.	50.54	$\{40\bar{4}1\}$,h	75.8 0.0	1.0
7.	55.32	$\{02\bar{2}1\}$,f	63.1 60.0	0.24
8.		$\{01\bar{1}2\}$,e	26.3 60.0	0.76
9.	56.98	$\{22\bar{4}3\}$	48.8 30.0	1.0

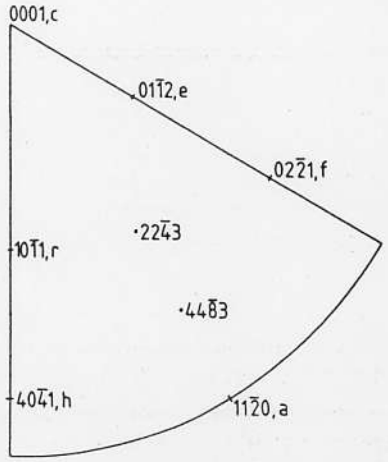


Fig. 2 Stereogram of diffraction peaks used

Specimens 2622 and 2606 were deformed in flow regime 3 close to the boundary with flow regime 2 but show no features of the pattern characteristic of flow regimes 1 and 2. This indicates that the change of textural pattern and dominant deformation mechanism occurs very rapidly with changing conditions of deformation.

The deformation mechanism of flow regime 3 is dominantly diffusion accommodated grain boundary sliding, Schmid et al /3/, a mechanism which is not expected to give rise to any crystallographic preferred orientation. The relatively weak textures of flow regime 3 can be attributed to intracrystalline slip which is thought to contribute to the total strain as a subordinated deformation mechanism.

In fig. 4c-f a sequence of Solnhofen limestone textures from specimens deformed under identical conditions to different finite strains is shown. The deformation condtions, 400^{o}C, strain rate 10^{-5}/sec, place the experiment in flow regime 1. The pattern which develops is identical to that described previously for Solnhofen limestones deformed in flow regimes 1 and 2. The sequence shows a steady intensification of the pattern with increasing finite strain. At high finite strains the concentration close to the e plane normal becomes more intense than the concentrations around a and h.

Fig. 4g-i shows the textures developed in specimens which were cored parallel to the bedding and deformed under identical conditions to different strains. The conditions were such (400^{o}C, strain rate 10^{-5}/sec) that the deformation occurred in flow regime 1. The lower strained specimen has an anomalous texture. This was probably caused by the non-coaxial superimposition of the experimentally induced texture on the depositional texture, resulting in a texture which is not axially symmetric. The texture in the two higher strained specimens has the pattern typical of flow regimes 1 and 2.

Thus, the new textural pattern has been established once a strain of only 11.5% has been attained.

b) Carrara marble

The texture developed in two Carrara marble specimens are shown in fig. 4j, k. The large grain size of these specimens has made the determation of the textures difficult and the results less accurate than those for Solnhofen limestone. However, the textures are identical in type and intensity to those determined by both optical and magnetic methods using the same or similar specimens and described by Owens and Rutter /6/. The specimens were deformed under conditions (400°C, 10^{-5}/sec strain rate) giving a flow regime analogous to flow regime 1 of Solnhofen limestone. The characteristic pattern has a strong concentration close to the e plane normal and a weak concentration near the h plane normal. The concentration develops extremely rapidly at quite small strain, Owens and Rutter /6/. Areas around the f and r plane normals show a reduction in concentration.

DISCUSSION

The Carrara marble texture differs from the Solnhofen limestone textures in that there is a single maximum at or near e which consequently is much stronger than the concentration near e as observed for the more complex Solnhofen limestone textures for regimes 1 and 2. Since we know that twinning is practically absent in the case of Solnhofen limestone and abundant in Carrara marble it is tempting to explain the Carrara marble texture as primarily caused by the twinning activity. This explanation is supported by the result shown in fig. 4,l, obtained from a Solnhofen limestone specimen deformed at 20°C where twinning is a major deformation mechanism in this rock as well. Indeed, the texture produced is remarkably similar to the Carrara marble textures. Furthermore, C.J. Spiers /19/ has shown that grains favourably oriented for twinning are rapidly moved towards the region around c, both by external rotation caused by e-twinning and re-orientation of the twinned volume itself.

The textures observed in Solnhofen limestone are more difficult to explain at this stage. From earlier work, e.g. Goetze and Kohlstedt /18/, we know that glide on r and f occurs in calcite rocks. Wenk et al. /14/ have argued that slip on pairs of r planes would contribute significantly to the development of an e maximum in the inverse pole figures. This could mean that the e-maximum observed in Solnhofen limestone deformed in regimes 1 and 2 could be due to r-glide since no twinning is observed.

The ridge of high concentrations running from the a to the h plane normal is difficult to interpret without performing sophisticated texture simulating work. G. Lister /21/, using the Taylor-Bishop-Hill analysis, finds that a similar texture is produced when r-glide is combined with f-glide and a minimum activity by e-glide is assumed. However, it is not clear yet to what extent the assumptions underlying this simulation method are justified in high temperature creep.

The texture transition into regime 3 is to be expected because grain boundary sliding dramatically alters the constraints imposed on a grain by its neighbours. An interpretation of the type of texture observed in regime 3 cannot be given at this stage.

Wenk et al. /4/ reported X-ray texture data obtained from samples of Solnhofen limestone deformed using a solid confining medium testing machine. The patterns of preferred orientation reported appear more complex and less consistent than those presented here. The differences are probably attributable to a variable degree of damage introduced into the specimens of Wenk et al. during cooling and offloading of the solid medium machine. The following factors, relating to their technique, could also explain the differences observed: (i) the introduction of water into the specimen above the dehydration temperature of talc used as a confining medium (ii) the unknown state of stress of a weak limestone specimen in a stronger confining medium, and (iii) the higher confining pressure used in most of their runs. Our textures for regimes 1 and 2 are compatible, however, with the Solnhofen limestone textures found by Kern /22/.

CONCLUSIONS

The conclusions which may be drawn from this work are as follows:

1) Solnhofen limestone deformed under conditions favouring dislocation creep develops a distinctive texture pattern.
2) Solnhofen limestone deformed under conditions favouring grain boundary sliding develops a weaker and different pattern which is related to the initial texture.
3) The change from one texture pattern to another occurs over a narrow range of temperature and strain rate.
4) The new texture pattern of flow regimes 1 and 2 replaces the pre-existing pattern after about 10% strain.
5) Carrara marble deformed under conditions favouring twinning has a distinct texture which is different from the textures observed in Solnhofen limestone deformed at the same temperature. Grain size, therefore, influences the textural pattern through the inhibition of twinning in fine grained Solnhofen limestone.

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REFERENCES

1. Heard, H.C. and Raleigh, C.B. Geol. Soc. Am. Bull., 83: 935-956, 1972
2. Rutter, E.H. Tectonophysics, 22: 311-334, 1974
3. Schmid, S.M. et al. Tectonophysics, 43: 257-291, 1977
4. Wenk, H.R. et al. Contr. Mineral. and Petrol. 38: 81-114, 1973
5. Siemes, H. Proc. of First Congress of the Intern. Soc. of Rock Mechanics (Lisbon, Portugal), 205-215, 1966
6. Owens, W.H. and Rutter, E.H. Phys. Earth Planet. Int.,in press, 1978
7. Ramez, M.R.H. and Murrell, S.A.F. Int. J. Rock Mech. Min. Sci., 1: 217-229, 1964
8. Heard, H.C. J. Geol., 73: 162-195, 1963
9. Paterson, M.S. Int. J. Rock. Mech. Min. Sci., 7: 517-526, 1970
10. Paterson, M.S. 2nd Int. Conf. on High Pressure Engineering, Brighton, to be published, 1976
11. Rutter, E.H. Tectonophysics, 14: 13-33, 1972
12. Rutter, E.H. Unpubl. Ph.D. thesis, Univ. London, 1970
13. Frost, M.T. and Siddans, A.W.B. to be pubished in J. Appl. Crystallog. 1978
14. Turner, F.J. and Weiss, L.E. Structural Analysis of Metamorphic Tectonites, 545pp. McGraw-Hill, New York 1963
15. Bunge, H.J. Mathematische Methoden der Texturanalyse 330pp. Akademieverlag, Berlin 1969
16. Baker, D.W. et al. J. Geol., 77: 144-172, 1969
17. Casey, M. and Whalley, J.S. in preparation
18. Goetze, C. and Kohlstedt, D.L. Contrib. Mineral. & Petrol., 59: 293-306, 1977
19. Spiers, C.J. To be submitted for publication in Contrib. Mineral. & Petrol.
20. Barber, D.D. and Wenk, H.R. J. Mater. Sci., 8: 500-508, 1973
21. Lister, G.S. this volume
22. Kern, H. Contrib. Mineral. & Petrol. 31: 39-66, 1971