

**THE GEOLOGICAL SIGNIFICANCE OF MICROSTRUCTURAL ANALYSES BY
TRANSMISSION ELECTRON MICROSCOPY**

Die geologische Bedeutung von Mikrostrukturanalysen mittels
Transmissionselektronenmikroskopie

Habilitationsschrift

vorgelegt von

Werner Skrotzki

aus

Hann.-Münden

Institut für Geologie und Dynamik der Lithosphäre
Georg-August-Universität zu Göttingen

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SUMMARY

The present work gives a review on transmission electron microscopic methods for analysing deformation-induced microstructures in minerals and rocks in order to unravel their thermo-mechanical history.

First of all the main microstructural features, like dislocations, stacking faults and grain boundaries are characterized. Then different TEM techniques are described for analysing these defect structures. Examples for the application of the TEM techniques are given. Emphasis is put on the characterization of the grain boundary structure, in particular on the periodicity and width of grain boundaries. As a model material for minerals with a high degree of covalent bonding germanium is used.

Depending on stress, temperature, strain rate, etc., different deformation mechanisms dominate, like dislocation glide, twinning, power-law and diffusional creep. These mechanisms are briefly characterized and the main microstructures they produce are described. The influence of metamorphic reactions on the deformation behaviour is discussed.

The central part of the work is the derivation of the thermo-mechanical history of rocks from their microstructure. Possibilities are shown of estimating deviatoric stresses from dislocation densities, subgrain sizes, dynamically recrystallized grain sizes and mechanical twinning. Since these parameters react differently on changes of stress and temperature, conclusions on the deformation history may be drawn from differences in the stress level. In certain cases several deformation episodes and recovery stages may be distinguished. Analyses of grain shapes and deformation-induced phase transformations allow a crude estimate of strain. Temperature estimates are possible from predominant slip systems and a change of the deformation mechanism.

Finally a model analysis is presented of the microstructure and texture in peridotites of the Balmuccia-Massif (Ivrea-zone, NW-Italy). Two deformation events may be recognized in the microstructure. The first deformation led to a coarse-grained dynamic recrystallization. The second deformation produced the subgrain and dislocation structure as well as a fine-grained dynamically recrystallized rim around the matrix grains in the western part of the massif. The subgrain boundaries are (100) and occasionally (001) tilt boundaries with variable tilt axis. The free dislocations are mainly screw dislocations with $[001]$ Burgers vector. An analysis of the dislocations bound in subgrain boundaries and the free dislocations yields $\{0kl\}[100]$ and $\{hk0\}[001]$ as main activated slip systems. The orthopyroxenes are not recrystallized and show deformation-induced clinoenstatite lamellae. The texture of olivine is characterized by $[010]$ perpendicular to the foliation and $[100]$ parallel to the lineation. In the

orthopyroxene [100] is normal to the foliation and [001] normal to the lineation.

Stress and temperature estimates based on the dislocation density, subgrain size, dynamically recrystallized grain sizes and the ortho-clinoenstatite transformation yield ≈ 10 MPa and $\approx 1000^\circ\text{C}$ for deformation event I and ≈ 300 MPa and $\approx 650^\circ\text{C}$ for deformation event II. The first and second deformation event is interpreted as intrusion of mantle material into the lower crust and the tilting of the Ivrea-zone, respectively. According to recent dating of tectono-metamorphic events in the Ivrea-zone the deformations may have taken place at caledonian (≈ 480 Ma) and hercynian times (≈ 300 Ma). From the correlation of the texture and microstructure it is concluded that the texture in the olivine goes back to the first deformation event. The texture of the relatively hard and therefore only weakly deformed orthopyroxene may be explained by external rotation in the soft olivine matrix.

The conclusions consider future TEM work on rocks and analogous materials necessary to corroborate statements in structural geology based on microstructures.

ZUSAMMENFASSUNG

Die vorliegende Arbeit gibt eine Übersicht über transmissionselektronische Methoden zur Analyse von verformungsinduzierten Mikrostrukturen in Mineralen und Gesteinen mit dem Ziel, ihre thermo-mechanische Vorgeschichte zu ergründen.

Zunächst werden die wesentlichen Mikrostrukturen, wie Versetzungen, Stapelfehler und Korngrenzen, charakterisiert. Danach werden unterschiedliche TEM-Techniken zur Analyse dieser Defektstrukturen beschrieben. Beispiele für die Anwendung der TEM-Techniken werden gegeben. Die Betonung wird auf die Charakterisierung der Korngrenzenstruktur gelegt, insbesondere die Periodizität und Breite von Korngrenzen. Als Modellschubstanz für Minerale mit hohem kovalenten Bindungsanteil wird Germanium benutzt.

In Abhängigkeit von Spannung, Temperatur, Verformungsgeschwindigkeit, etc., dominieren unterschiedliche Verformungsmechanismen, wie Versetzungsgleiten, Vezwilligung, "Power-law"- und Diffusionskriechen. Diese Mechanismen werden kurz charakterisiert; wesentliche Mikrostrukturen, die sie erzeugen, werden beschrieben. Der Einfluß metamorpher Reaktionen auf das Verformungsverhalten wird diskutiert.

Im Zentrum der Arbeit steht die Ableitung der thermo-mechanischen Geschichte von Gesteinen aus ihrer Mikrostruktur. Es werden Möglichkeiten aufgezeigt, Spannungen anhand von Versetzungsdichten, Subkorngößen, dynamisch rekristallisierten Korngrößen und mechanischer Zwillingsbildung abzuschätzen. Da die einzelnen Parameter

unterschiedlich auf Spannungs- und Temperaturänderungen reagieren, lassen Spannungsvergleiche Rückschlüsse auf die Verformungsgeschichte zu. In bestimmten Fällen können mehrere Deformationsereignisse und Erholungsphasen unterschieden werden. Analysen von Kornformen und verformungsinduzierten Phasenumwandlungen lassen eine grobe Abschätzung von Verformungsbeträgen zu. Temperaturabschätzungen sind anhand von dominanten Gleitsystemen und dem Wechsel des Verformungsmechanismus möglich.

Schließlich wird eine Modellanalyse der Mikrostruktur und Textur in Peridotiten des Balmuccia-Massivs (Ivrea-Zone, NW-Italien) vorgenommen. Es lassen sich zwei Verformungsphasen in der Mikrostruktur erkennen. Die erste Verformung hat zu einer grobkörnigen dynamischen Rekristallisation geführt. Die zweite Verformung hat die Subkorn- und Versetzungsstruktur bestimmt und im westlichen Teil des Massivs einen feinkörnig rekristallisierten Saum um die Altkörner erzeugt. Die Subkorn Grenzen sind (100) und vereinzelt (001) Kippgrenzen mit variabler Kippachse. Die freien Versetzungen sind im wesentlichen Schraubenversetzungen mit [001] Burgersvektor. Eine Analyse der in Subkorn Grenzen gebundenen und der freien Versetzungen ergibt {0kl}[100] und {hk0}[001] als hauptsächlich aktivierte Gleitsysteme. Die Orthopyroxene sind nicht rekristallisiert und zeigen verformungsinduzierte Klinoenstatitlamellen. Die Textur des Olivins ist charakterisiert durch [010] senkrecht zur Foliation und [100] parallel zur Lineation. Im Orthopyroxen ist [100] senkrecht zur Foliation und [001] normal zum Lineation.

Spannungs- und Temperaturabschätzungen anhand von Versetzungsdichte, Subkorngröße, dynamisch rekristallisierten Korngrößen und der Ortho-Klinoenstatitumwandlung ergeben ≈ 10 MPa und $\approx 1000^\circ\text{C}$ für Verformungsphase I und ≈ 300 MPa und $\approx 650^\circ\text{C}$ für Verformungsphase II. Die erste Verformungsphase wird als Intrusion von Mantelgestein in die untere Kruste, die zweite als die Steilstellung der Ivrea-Zone interpretiert. Nach neusten Altersbestimmungen der tektono-metamorphen Ereignisse in der Ivrea-Zone könnten die Verformungen zu kaledonischer (≈ 480 Ma) und zu herzynischer Zeit (≈ 300 Ma) abgelaufen sein. Die Korrelation von Textur und Mikrostruktur legt den Schluß nahe, daß die Textur des Olivins noch auf die erste Verformungsphase zurückgeht. Die Textur des relativ harten und daher nur schwach verformten Orthopyroxens läßt sich durch externe Rotation in der weichen Olivinmatrix deuten.

In den Schlußbemerkungen werden zukünftige TEM-Arbeiten an Gesteinen und Analogsubstanzen angesprochen, die notwendig erscheinen, um strukturgeologische Aussagen anhand von Mikrostrukturen weiter zu erhärten.

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I INTRODUCTION

Tectonic processes within the earth's lithosphere as globally described by the theory of plate tectonics manifest themselves in characteristic deformation structures ranging from macro- to micro-scale (Tab. 1). To decipher these structures, that is to establish the thermo-mechanical history the rocks have experienced, is the ultimate aim of structural geology. While field work and optical microscopy are essential methods of working toward the overall goal other methods like transmission electron microscopy (TEM) have been proven recently to be more and more important. The power of TEM in analysing microscopic features of minerals has been clearly demonstrated by the comprehensive works edited by Wenk (1976, 1985). What is missing, however, is the direct relation to problems to be solved in structural geology. It is the purpose of the present thesis to put emphasis onto this aspect.

Tab. 1: Typical deformation-induced structures and methods to analyse them.

Structure (scale)	Typical ductile deformation features	Essential analytical methods
Macro-structure (> km - cm)	folds shear zones	field work (outcrop)
Meso-structure (cm - μm)	foliation lineation	optical microscopy (thin section)
Micro-structure (μm - Å)	grain boundaries subgrain boundaries stacking faults dislocations	transmission electron microscopy (TEM-foil)

The microstructure of a crystal consists of lattice defects which are not in thermodynamical equilibrium characterized by type, density, distribution, size and shape (Haasen, 1974). According to this definition the microstructure considered consists of dislocations, stacking faults and grain boundaries. Interphase boundaries are excluded and therefore will be considered only marginally. The defects investigated may be either grown-in or deformation-induced. Modern TEM allows their characterization down to the atomic level. Informations on the thermo-mechanical history of rocks may be obtained by comparing their microstructure with that in the same or similar material deformed in the laboratory under well-defined conditions. Another way of getting

informations is a comparison of the microstructural results with those expected from theory. However, such comparisons are rather complicated:

- Rocks have an enormous range of chemical and mineralogical compositions.
- Deformation of rocks occurs in a large range of physical and chemical environments.
- Rocks may have experienced several deformations. Therefore the question arises whether the deformation events may be separated in the microstructure and how many.
- Syn- or posttectonic metamorphism may have changed the microstructure. The same may be due to the passage of fluids through rocks.
- Natural deformation occurs at low strain rates while experiments are run orders of magnitude faster. Thus, extrapolation from laboratory to natural conditions always raises the question of a change in deformation mechanism.
- Microstructural investigations are done on very small volumes. Thus, the homogeneity of the microstructure in bulk rocks will be always a matter of question.

These problems will be discussed throughout the paper.

The work is divided into a basic and an applied part. The basic part (sections II to IV) describes the microstructural features, TEM techniques to characterize them and mechanisms of microstructural development. Because of the lack of suitable specimens to clearly demonstrate grain boundary structures on rocks advantage is taken of well-defined germanium bicrystals. Germanium may be regarded as a model substance for rocks with a high degree of covalent bonding, for example quartz.

The applied part (sections V and VI) demonstrates the geological significance of microstructural analyses of rocks. After presenting different relations between microstructure and thermo-mechanical history their application to problems in structural geology is shown by detailed studies of the microstructure in peridotites of the Balmuccia massif, an ultrabasic body located within the Ivrea zone (NW-Italy). The nomenclature of the ultrabasic rocks used is given in the appendix. Peridotites have been selected for the following reasons:

- Olivines and pyroxenes, the main constituents of these rocks, are insensitive to electron radiation.
- Numerous studies have shown that these minerals are suitable for conventional and high resolution TEM thus allowing a detailed defect characterization.
- For ultrabasic rocks there exist numerous analyses of experimentally induced microstructures. Thus, a direct comparison of natural microstructures with those from experiment is possible.
- Standard universal stage measurements give a complete determination of the crystallographic preferred orientation (here referred to as texture). Thus, microstruc-

ture and texture may be correlated to each other. Based on the microstructural results mechanisms of textural development can be discussed.

- There exist various macrostructural, petrological and geochronological data of rocks of the Balmuccia massif and the Ivrea zone. Thus, microstructural studies provide additional informations for a discussion of models on the tectonic evolution of the massif.

So far microstructural analyses have been mainly applied in materials science. Therefore the main methods of materials science as well as the most important models and recent experimental results in this field will be reviewed. Because of the interdisciplinary character of the work the materials scientific aspects will be emphasized.

II MICROSTRUCTURAL FEATURES

This section gives a short description of the characteristics of extended crystal defects in so far as it seems appropriate for the understanding of this work. For further information the reader is referred to standard text books in this field, e.g. Hirth and Lothe (1968) and Bollmann (1970, 1982).

II.1 Dislocations

Dislocations are the main carriers of crystal plasticity. They are line defects characterized by their translation vector (Burgers vector) $\mathbf{b}$ and their line direction $\mathbf{l}$ (Fig. 1). Depending on the orientation of $\mathbf{b}$ with respect to $\mathbf{l}$ two extreme cases are distinguished: $\mathbf{b}$ normal to $\mathbf{l}$ defines an edge dislocation, $\mathbf{b}$ parallel to $\mathbf{l}$ a screw dislocation. The vector product $\mathbf{b} \times \mathbf{l}$ defines the slip plane, the dislocation motion on which produces plastic deformation. Screw dislocations do not have any slip plane unless they are dissociated (see below). Edge dislocations may leave its slip plane by climb, dissociated screw dislocations by cross slip. Climb occurs by absorption or emission of vacancies, cross slip by the constriction of the stacking fault separating the partial dislocations. Both processes are thermally activated and therefore become important only at higher temperatures.

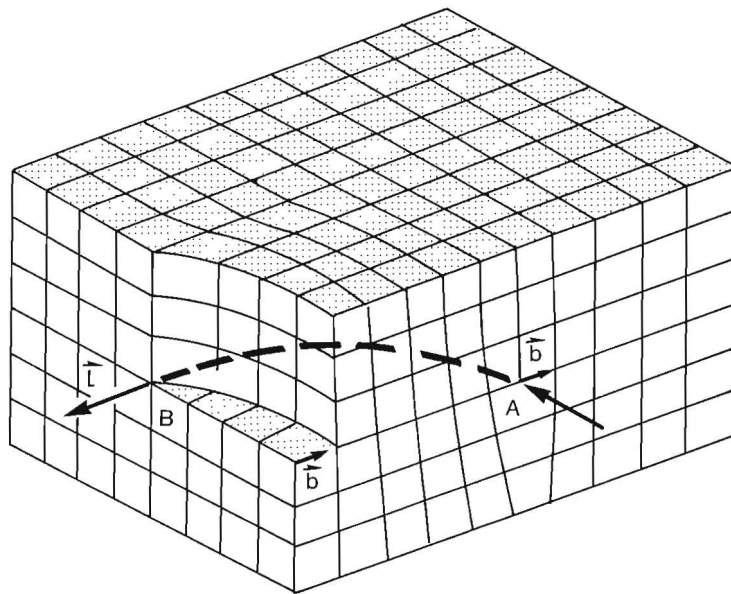


Fig. 1: Change of a dislocation from the edge- (A) to the screw-orientation. Burgers vector $\mathbf{b}$ is drawn in the FS/RH (Finish-Start/Right Hand) convention according to a Burgers circuit in the undistorted reference lattice.

II.2 Stacking faults and antiphase boundaries

Stacking faults and antiphase boundaries are faults which play an important role in dislocation dissociation and therefore affect significantly the dislocation behaviour. The self-energy of a dislocation is proportional to the square of its Burgers vector. Thus any dislocation with a Burgers vector $\mathbf{b}$ is energetically favoured to dissociate into two partial dislocations $\mathbf{b}_{p1}$ and $\mathbf{b}_{p2}$, provided that b^2 is greater than $b_{p1}^2 + b_{p2}^2$. Since the partial dislocations are not lattice vectors they produce a stacking fault by moving apart (Fig. 2). The cost of stacking fault energy prevents the partials to separate too far by elastic repulsion. The equilibrium width of the stacking fault is given by the force balance. Stacking faults are characterized by the displacement vector $\mathbf{R}$ ($= \mathbf{b}_p$) and the fault normal $\mathbf{n}$. Not only can stacking faults be produced by glide, but they also occur as accidents of growth and as a result of a plate-like condensation of vacancies and interstitials.

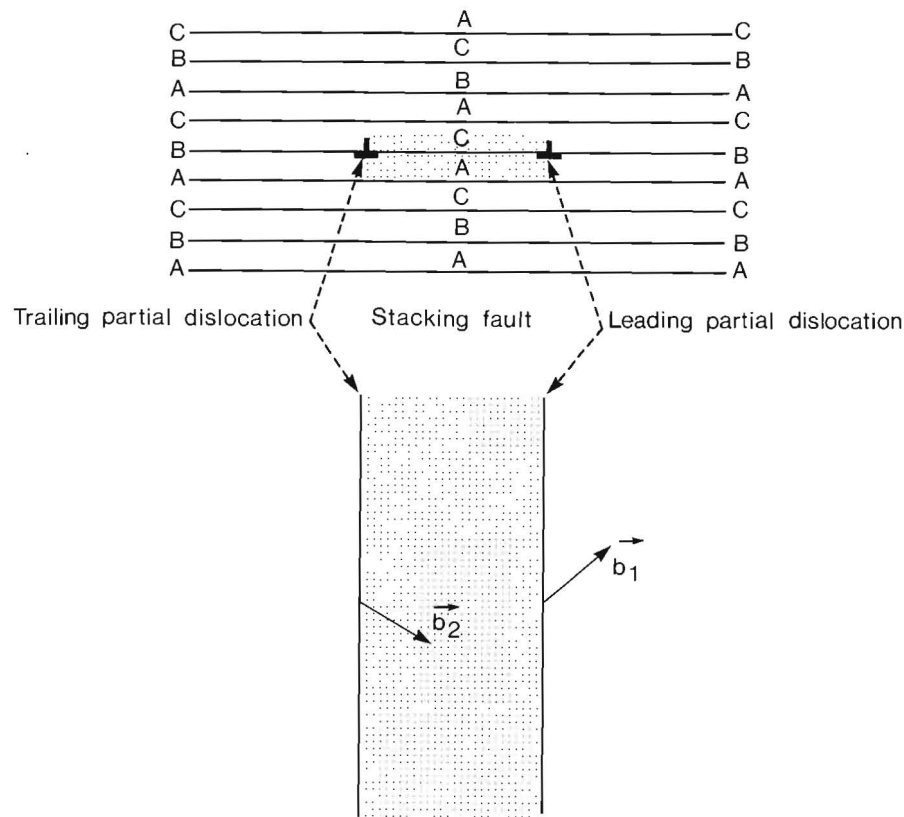


Fig. 2: Stacking fault in the face-centred cubic lattice between two partial dislocations. Top section is perpendicular to the (111) planes stacked in ABC... sequence outside the ribbon, in BCACAB... sequence inside the ribbon of fault. Bottom section shows the dissociated dislocation with the fault in a (111) plane. (After Nicolas and Poirier, 1976)

Antiphase boundaries can be defined similarly as irregularities in the stacking when taking into account that two or more types of atoms are present in the lattice of ordered crystals. Antiphase boundaries are introduced by dislocations with Burgers vectors different from translation vectors of the ordered structure. They are also generated during disorder-order transformations by the intergrowth of ordered domains.

II.3 Grain boundaries

Grain boundaries are the interfaces between crystals of the same nature. They are characterized by the misorientation angle Θ around an axis $\mathbf{u}$ and by the boundary plane normal $\mathbf{n}$ (Fig. 3). Depending on the orientation of $\mathbf{u}$ with respect to $\mathbf{n}$ two extreme cases are distinguished: $\mathbf{u}$ normal to $\mathbf{n}$ defines a tilt, $\mathbf{u}$ parallel to $\mathbf{n}$ defines a twist grain boundary.

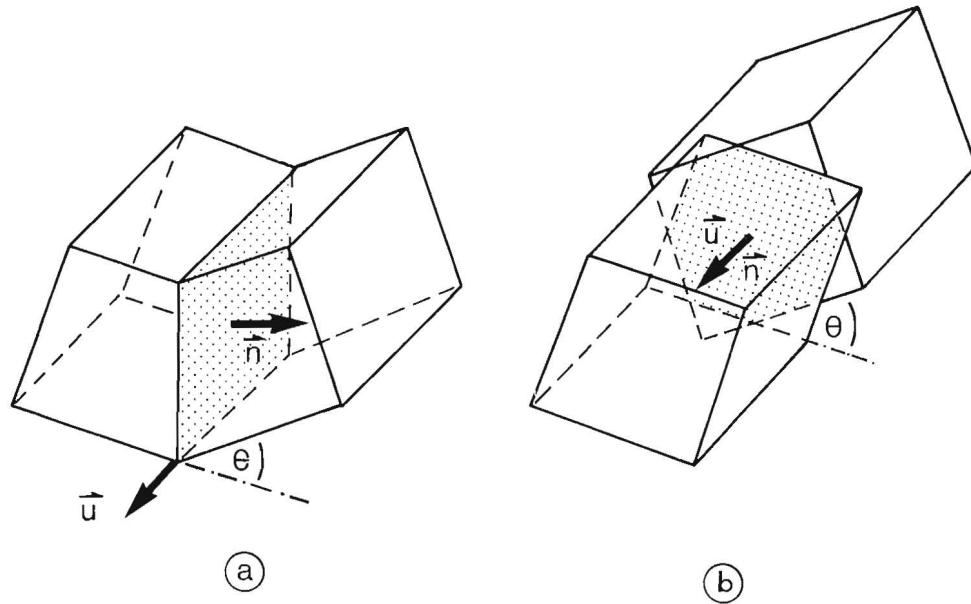


Fig. 3: Grain boundary between two crystals of the same nature produced by a misorientation Θ . If the grain boundary normal is perpendicular and parallel to the rotation axis the boundary is called tilt (a) and twist boundary (b), respectively.

Grain boundaries play an important role in controlling many physical and mechanical properties. For example the ductility of polycrystals strongly depends on the ability of the material to dynamically recrystallize while the strength may be determined by

intercrystalline cracking. Diffusion of water which may be a catalyst in metamorphic reactions (Rubie, 1986) can be enhanced along grain boundaries. In both cases not only the coarse features of the boundary like dislocations or ledges but also some aspects of the detailed atomic structure of the boundary may be important. Therefore in the following a review is given on current theories of the structure of grain boundaries.

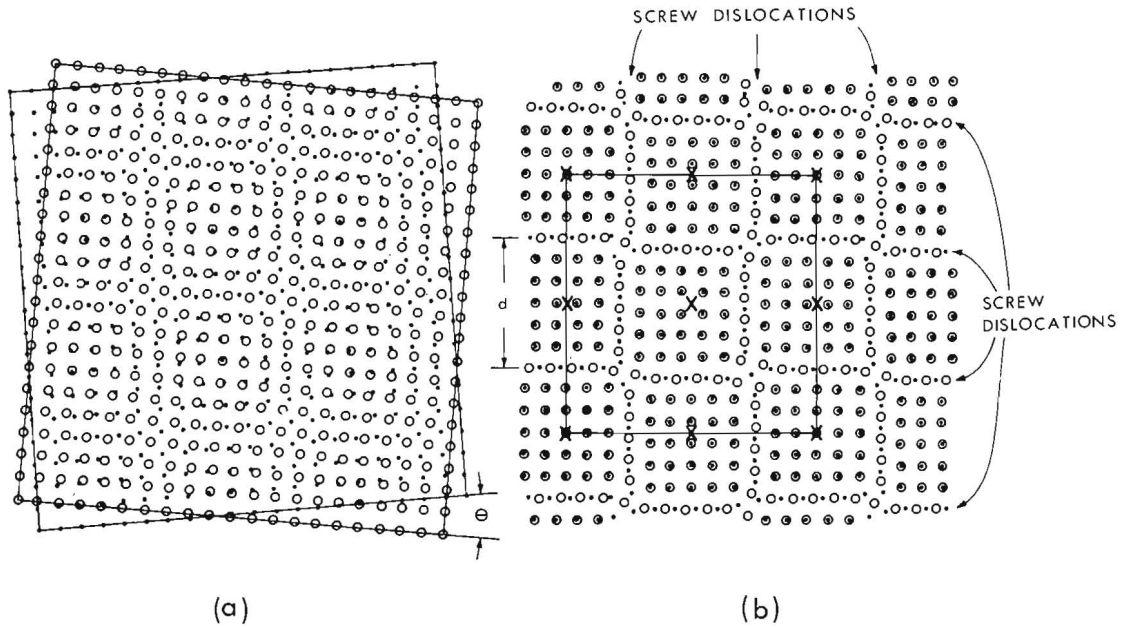


Fig. 4: Unrelaxed (a) and relaxed (b) atomic configurations in the planes just above and below the boundary in a low angle $[001]$ grain boundary, illustrating formation of the screw dislocation network. The unit cell of the CSL is indicated by the square. The periodicity of the screw dislocations is given by the O-lattice marked by crosses.

The equilibrium boundary structure is fundamentally determined by the manner in which the atoms at or near the boundary attain minimum energy. To examine these structures, various models have been developed based on an arrangement of different types of dislocations (low angle boundaries: $\Theta < 15^\circ$) and/or structural units (high angle boundaries). These models have been reviewed by Gleiter (1982) and Smith and Shiflet (1987), for example. Much attention has been paid to the O-lattice theory (Bollmann 1970, 1982; Smith and Pond 1976). This theory has linked together elegantly many ideas from different areas of interface studies. The theory provides a framework within which the dislocation content of an arbitrary interface can be defined in the context of conservation of optimal structures. The O-lattice theory generalized the concept of the coincidence-site lattice (CSL) developed by Kronberg and Wilson (1949) to include

coincidences of all equivalent points, lines or planes. Such coincidences are referred to as O-points, O-lines and O-planes, respectively, and are regions of exact matching between two interpenetrating crystal lattices.

In the case of a low angle twist boundary (Fig. 4), the O-lattice points are separated by bands of mismatch (with spacing d) which are screw dislocations possessing Burgers vectors of the perfect crystal (and hence are called primary dislocations). Fig. 4 shows that the periodicity of the screw dislocations and the O-lattice are identical and that $d = b/2 \sin(\Theta/2)$, where Θ is the misorientation angle and b is the magnitude of the Burgers vector of the screw dislocations. The O-lattice is, in general, a sublattice of the CSL, although in certain circumstances the O-lattice and CSL periodicities are identical.

Grain boundaries may be characterized by the Σ -value where $1/\Sigma$ is the fraction of coincidence sites. If the orientation relationship between the two crystals is slightly changed, the periodicity of the CSL pattern is lost. However, in the case of a low-energy boundary it may be energetically more favourable to conserve the initial coincidence structure and to accommodate the deviation by introducing a network of secondary dislocations. Such dislocations should be distinguished from primary dislocations which accommodate small deviations from the perfect crystal structure. Possible Burgers vectors of the secondary dislocations are defined by the DSC lattice. The DSC lattice is so named because it is the lattice of pattern conserving displacements, i.e. a displacement of one crystal lattice with respect to the second by a DSC vector causes a pattern shift which is complete. Support for this crystallographic approach has come from analyses of secondary dislocation networks in twist boundaries of metals (e.g. Schober and Balluffi, 1970), ceramics (Sun and Balluffi, 1982) and semiconductors (Föll and Ast, 1979; Vaudin and Ast, 1983). The DSC dislocations in general do not have primitive (shortest) DSC-Burgers vectors showing that Frank's dissociation rule (b^2 -rule) cannot directly be applied to DSC dislocations in grain boundaries (Skrotzki et al., 1988).

Although the strength of the O-lattice theory has been demonstrated frequently, the prediction of actual interface structures and their energies using this theory requires more knowledge of the principles which govern the free energy of interfacial structures. For example an appropriate rigid body translation between two grains does not change the periodicity of the O-lattice but calculations suggest that large energy changes occur (Smith and Pond, 1976). Evidently not all O-lattice points are associated with low energy. Naturally a geometrical theory cannot predict which of the alternative structures has lowest free energy since the nature of the chemical binding can vary widely; for a discussion of geometric criteria for low interfacial energy see Sutton and Balluffi (1996). However, the O-lattice approach leads to a general starting point for

the calculation of energy and the interpretation of periodic structures.

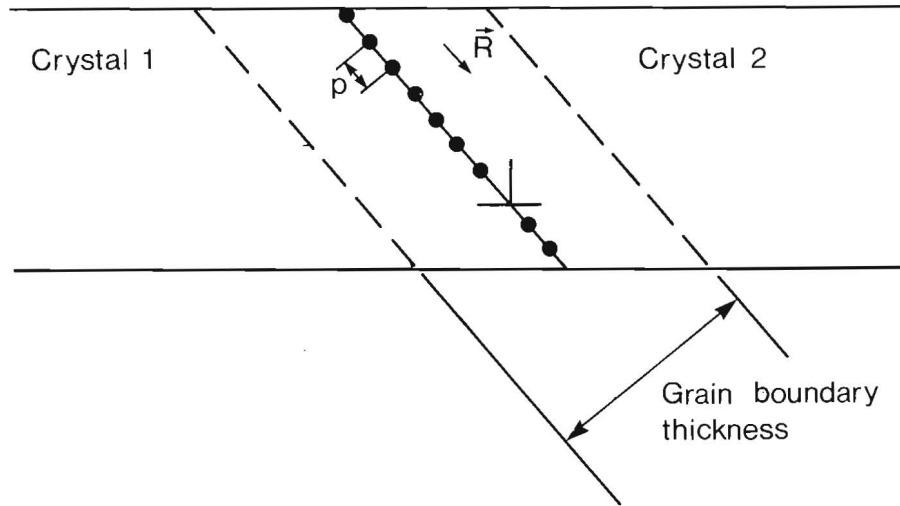


Fig. 5: Schematic of a grain boundary contained in a thin foil, illustrating the information needed to characterize the structure of a grain boundary. The filled circles represent the periodicity p of the boundary consisting of grain boundary dislocations or structural units. The inverted T represents an isolated defect such as an extrinsic dislocation or a ledge. The vector $\vec{R}$ is the rigid body translation of crystal 2 with respect to crystal 1 and is not necessarily parallel to the boundary plane. The pair of dashed lines represents the grain boundary thickness, i.e. the "structural width" associated with the penetration of the displacement field into the two neighbouring crystals. (After Carter and Sass, 1981)

Fig. 5 summarizes the information that must be obtained to characterize the structure of a grain boundary. For a complete characterization of a boundary, it is also necessary to determine its geometry, the boundary plane and the axis and angle describing the misorientation between the two crystals. Finally, in general, the composition near the boundary may also vary from the bulk value.

III TEM TECHNIQUES TO CHARACTERIZE DEFECT STRUCTURES AND APPLICATIONS

In this section the main electron microscopy techniques available for studying defect structures are reviewed. Attention is focussed on the advantages and disadvantages of the different techniques and the principles underlying their application. Examples of the use of these techniques will be given and the information they provide will be discussed. For the physical background of these techniques the reader is referred to standard text books (e.g. Hirsch et al., 1965; Edington, 1974).

III.1 Strong-beam techniques

The term "strong-beam technique" for the study of lattice defects applies to the situation shown in Fig. 6, where two beams, the transmitted beam 0 and the diffracted beam g are strongly excited; the excitation error s_g is usually set to be slightly positive.

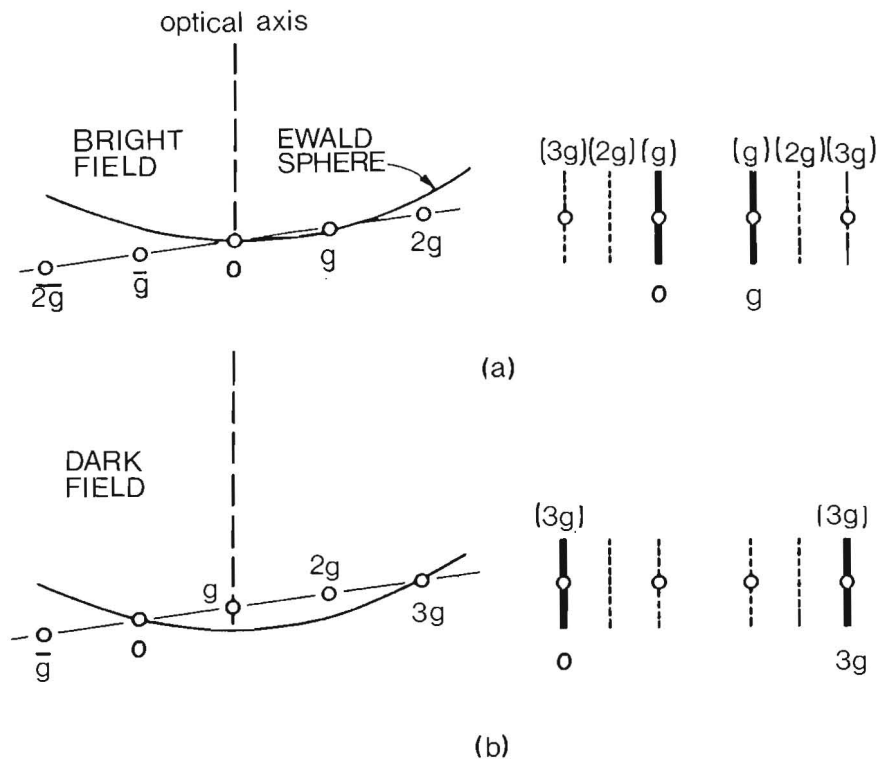


Fig. 6: Diffraction geometry for (a) strong-beam bright-field and (b) weak-beam $g(3g)$ dark-field. The dashed lines (g) , $(2g)$ and $(3g)$ indicate the positions of the respective Kikuchi lines and are fixed to the crystal; they remain in the positions shown when the incident beam is tilted off the optical axis. In (b) the Ewald sphere cuts the g -systematic row of reflections at a distance $3g$ from the 000 spot. (After Carter and Sass, 1981)

This diffraction geometry combined with the $\mathbf{g} \cdot \mathbf{b} = 0$ invisibility criterion is generally used to determine dislocation Burgers vectors. The Burgers vector is given by $\mathbf{g}_1 \times \mathbf{g}_2$, with $\mathbf{g}_i$ fulfilling the criterion. The conventional strong-beam technique can be also applied to very low angle boundaries where the dislocation spacing is more than $1/3 \xi_g$, where ξ_g is the extinction distance (Tholen, 1970). An example is given in Fig. 7 for a low angle grain boundary in olivine. The boundary is a 0.06° (010) twist boundary consisting of a square net of screw dislocations with $[100]$ and $[001]$ Burgers vectors.

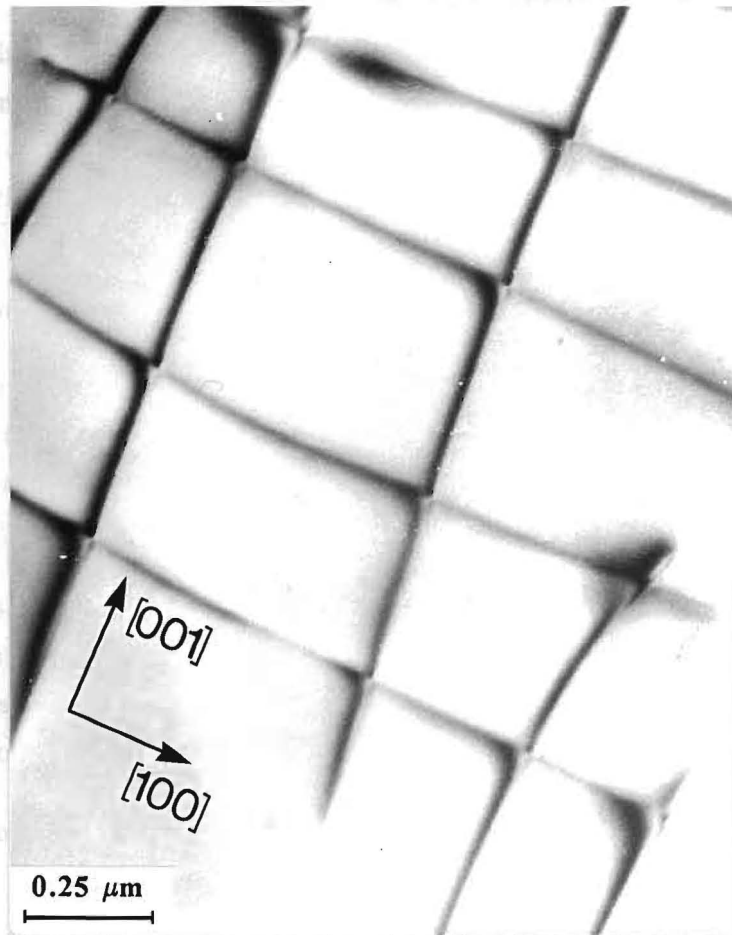


Fig. 7: Low angle (010) twist boundary in olivine of a peridotite xenolith consisting of a square network of $[100]$ and $[001]$ screw dislocations. Bright-field image taken with $\mathbf{g} = \bar{1}22$ and $\mathbf{g} = 121$.

For boundaries with larger misorientations the technique has to be modified. Two approaches to the problem may be used:

- In the first the diffraction condition is set only in one grain, assuming that the second grain acts as an absorbing wedge. The contrast observed depends on whether

the upper or lower grain is diffracting strongly (McDonald and Ardell, 1973; Varin et al., 1975). Images of secondary dislocations can thus appear as dark or light, depending on which grain is diffracting.

- In the second a diffracted beam is strongly excited in both crystals. This method has been found to give very good agreement with computer-simulated images (Humble and Forwood, 1975; Forwood and Humble, 1975). The simultaneous two-beam method has the advantage that the diffraction conditions can be adjusted to make the background uniform over two grains, so that the features in the images are more easily recognized. However, except in the case of using common reflections, care has to be taken with a quantitative analysis of secondary dislocation images.

The strong-beam technique may be also used to determine the translation vector $\mathbf{R}$ of stacking faults and antiphase boundaries and the rigid body translations at grain boundaries (Clark, 1976).

III.2 Weak-beam techniques

The weak-beam technique was developed to study lattice dislocations in more detail. The theoretical and experimental aspects of this technique are described by Cockayne (1972) and Ray and Cockayne (1971), respectively.

An example of the diffraction geometry of the weak-beam method which may be set up is shown in Fig. 6. Starting with the strong-beam condition (a) the dark-field beam-deflection is used to tilt the reflection $\mathbf{g}$ onto the optical axis. The Ewald sphere is thus rotated so that condition $\mathbf{g}(3\mathbf{g})$ is obtained. The Kikuchi lines are fixed to the specimen and do not move while the beam is tilted.

The method produces narrow images of dislocations, the contrast width depending on $s_{\mathbf{g}}$. To resolve narrow spaced partial dislocations $s_{\mathbf{g}}$ should be greater than about $2 \cdot 10^{-2} \text{\AA}^{-1}$ (Ray and Cockayne, 1971). Depending on the type of reflection and the material, it is frequently necessary for the Ewald sphere to intersect the systematic row of reflections at a position significantly larger than $3\mathbf{g}$, to obtain the same value of $s_{\mathbf{g}}$. The resolution limit of dislocation dissociation is about 30 Å.

The successful application of the weak-beam method to the study of dense dislocation networks in grain boundaries has been demonstrated for example by Carter et al. (1980). Experimentally it is found that $s_{\mathbf{g}}$ can be about one order of magnitude smaller than required for lattice dislocations and still gives narrow images. An example for the application of the weak-beam method to high angle boundaries is given in Fig. 8. The boundary is a $\Sigma = 51$ [011] tilt boundary in Ge which has been subjected to low temperature deformation. Fig. 8a is a dark-field image while Fig. 8b is a weak-beam

image taken with the diffraction condition $g(3g)$. The more detailed imaging of the deformation-induced dislocation tangles in the boundary is obvious in Fig. 8b. The difficulty in applying the technique quantitatively results from the fact that two values of s_g must be considered since s_g is rarely the same in both grains.

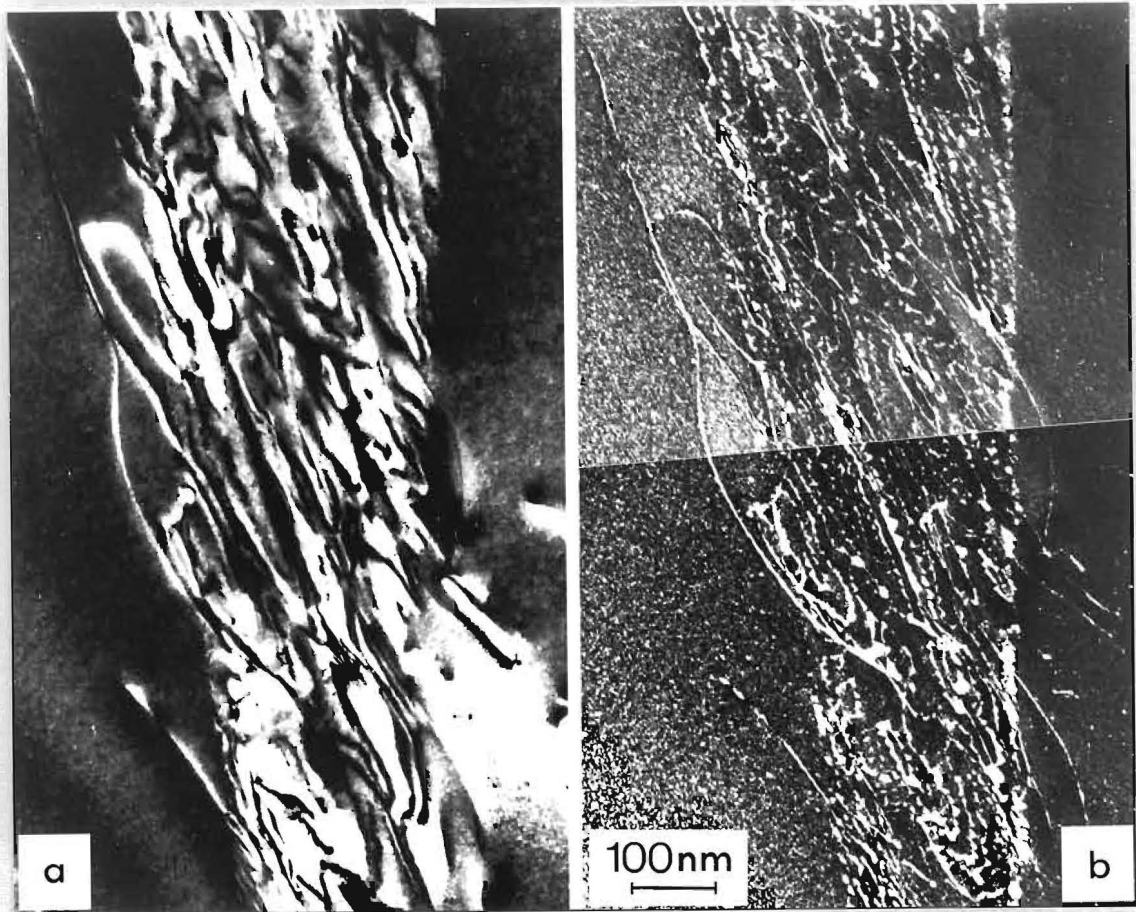


Fig. 8: Dislocation microstructure of a $\Sigma = 51$ [011] tilt boundary in Ge subjected to low-temperature deformation ($T = 500^\circ\text{C}$, $\dot{\epsilon} \approx 10^{-5} \text{ sec}^{-1}$, $\epsilon \approx 1\%$). Images are taken with $g = 022$: (a) dark-field image, (b) weak-beam image with the diffraction condition $g(3g)$.

III.3 High resolution imaging techniques

To study crystal defects at the atomic level high resolution transmission electron microscopy (HRTEM) has become a very powerful tool. If the point-to-point resolution of the microscope is high enough, a "structure image" (Cowley, 1970) can be obtained, that is under certain conditions of defocus and for certain values of foil thickness, it can represent a projection onto the image plane of the atomic structure of the crystal. Such images have been studied in detail for many crystals with large unit cells (for a review on silicates see Buseck and Iijima, 1974). Present generations of

microscopes have point-to-point resolutions down to $2\text{\AA}$.

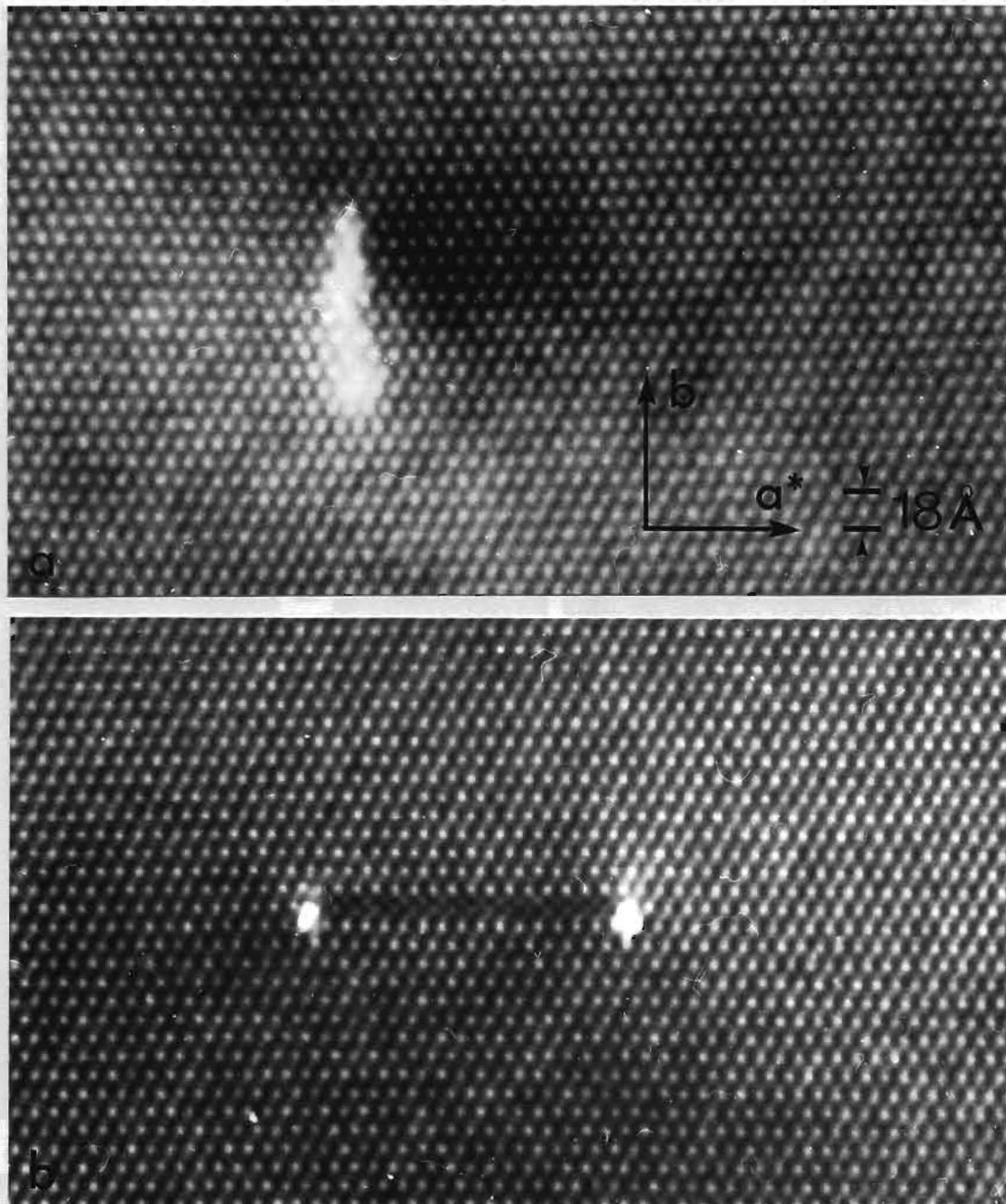


Fig. 9: HRTEM image of the core structure of an undissociated $[100]$ edge dislocation (a) and a dissociated $[101]$ 75° -dislocation (b) in clinoamphibole, respectively.

Structure images are taken by aligning low index poles parallel to the optical axis. If the defect in its "end-on" or "edge-on" orientation (line or planar defect) does not exactly meet this condition then the reflections are not symmetrically positioned relative to the optical axis. As a result only one excited diffracted beam may be in strong interference with the transmitted beam thus producing a lattice fringe image.

The planar defect produced by the partial dislocations has a pyroxene-type structure consisting of two single silicate chains. The inner core structure of the dislocation is

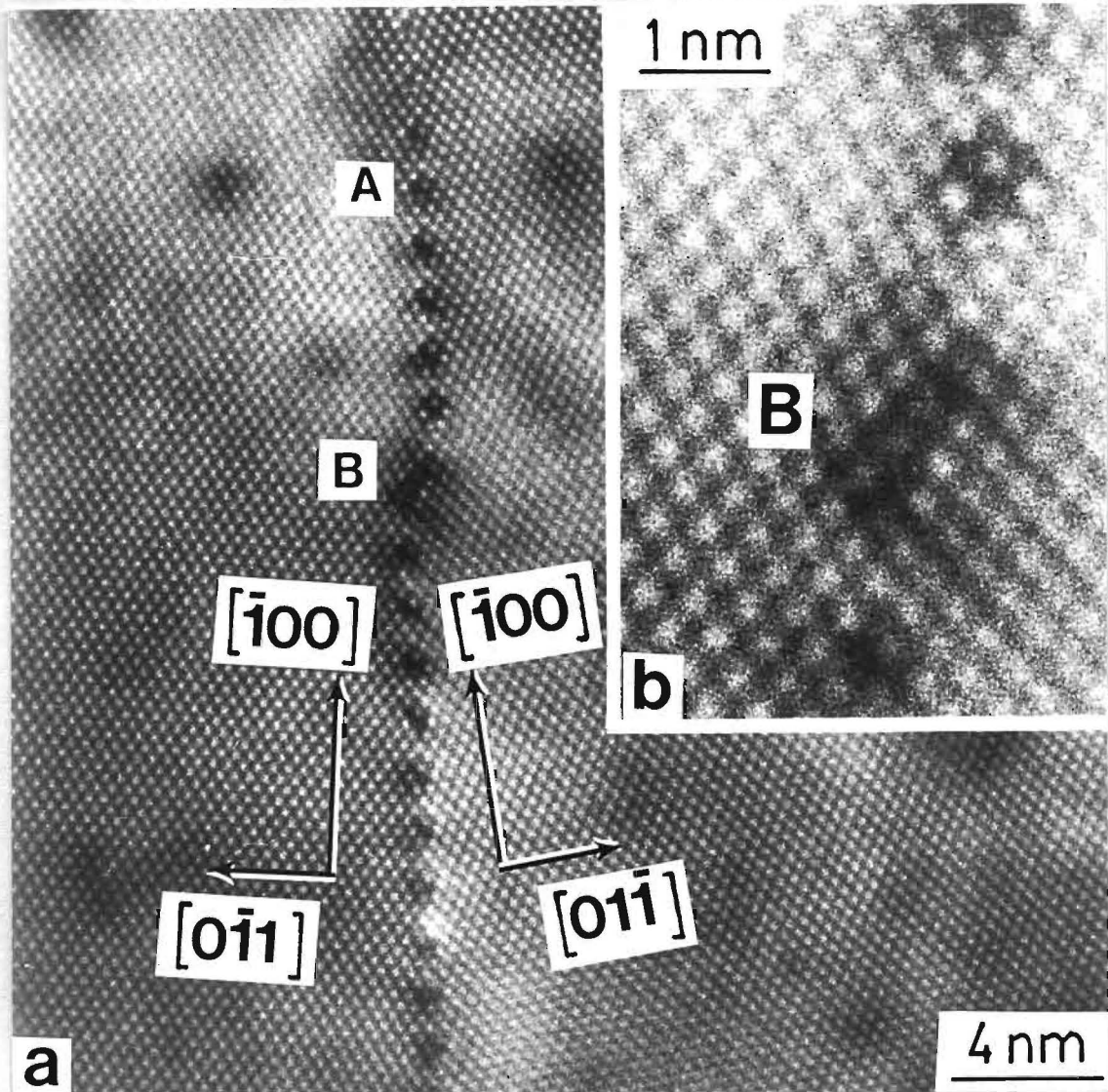


Fig. 10: HRTEM image of the core structure of a $\Sigma = 51$ $[011]$ tilt boundary in Ge viewed edge-on. The periodic structure is produced by Lomer-type edge dislocations (A). Defect (B) which is enlarged in (b) is a secondary dislocation.

Application of HRTEM to dislocations and interfaces allows a detailed characterization of these defects. Figs. 9a and b show examples of the core structure of an undissociated

[100] edge dislocation and a dissociated [101] 75° -dislocation in clinoamphibole (hornblende $\text{NaCa}_2(\text{Mg, Fe})_4\text{AlSi}_6\text{Al}_2\text{O}_{22}(\text{OH, F})_2$; double chain silicate), respectively. The Burgers vector component normal to the dislocation line has been determined by a Burgers circuit. The [100] dislocation is characterized by two, the $1/2$ [101] partial dislocations of the dissociated [101] dislocation by one {110}-type half-plane inserted. The planar defect produced by the partial dislocations has a pyroxene-type structure consisting of two single silicate chains. The inner core structure of the dislocations in amphiboles is rather open due to a lower density. The same has been found by Carter et al. (1987) for the lateral twin boundary in spinel ($\text{Mg}_2\text{Al}_2\text{O}_5$). This observation indicates the role of dislocations in the fast diffusion of certain cations and anions necessary for the alterations found in these minerals (Skrotzki, 1989).

Fig. 10 shows the structure of a near-coincident $\Sigma = 51$ tilt boundary in Ge. The 16.1° tilt is accommodated by primary Lomer-type edge dislocations (A) with a $1/2$ [011] Burgers vector. The core structure of this type of dislocation has been analysed in detail by Bourret et al. (1982). The small deviation from the exact coincidence is taken up by secondary dislocations one type of which is shown at (B). The secondary dislocations represent irregularities in the periodicity of the primary dislocations. The Burgers vectors of the secondary dislocations may be determined from HRTEM images by the method described by Skrotzki et al. (1988). The potential use of HRTEM in studying grain boundary structures has been clearly demonstrated in numerous works on high angle boundaries in semiconductors and ceramics.

II.4 Diffraction techniques

Electron diffraction techniques to study grain boundary structures have been elaborated only recently, and their strength has been demonstrated clearly. Because of the lack of a current review in this field of research, a more detailed description of the diffraction techniques will be given here.

Electron diffraction may be used in general to study periodic arrangements of defects or the periodic nature of defects itself. Any periodic array of defects such as dislocations in a small angle boundary can act as a diffraction grating which gives rise to reflections that are related to the period and structure of the defect array. This will be demonstrated for an edge-on and an inclined high angle boundary in Ge.

The diffraction geometry of an edge-on tilt boundary is sketched in Fig. 11. A grain boundary can be envisioned as a layer of elastically strained material where the atoms are displaced from their normal positions. Because this layer is thin, the boundary reflections are elongated along a direction normal to the boundary as presented in Fig. 11. These rods of diffracted intensity (or reciprocal lattice rods, so-called relrods) can

be recorded as streaks on the diffraction pattern. The periodicity of the streaks is inverse of the boundary periodicity, e.g. the dislocation spacing. The situation becomes complicated for faceted boundaries. In this case the periodicity of the facets is superimposed onto the diffraction pattern of the planar boundary sections (Eastman, 1985). The diffraction effects of a faceted tilt boundary are sketched in Fig. 12, an example is given in Fig. 13. The boundary shown is a near-coincident $\Sigma = 51$ tilt boundary similar to that of Fig. 11 but with a larger extra twist misorientation. The secondary dislocations are associated with ledges in the grain boundary of height $8.9\text{\AA}$. Their spacing is rather regular ($A = 110\text{\AA}$), with one step occurring every six to eight primary dislocation ($d = 14.3\text{\AA}$). The average boundary plane is inclined to the symmetric plane given by the long facets by about $\Phi = 4.7^\circ$. Analysis of the diffraction pattern according to Fig. 12 yields $d = 14.5\text{\AA}$, $A = 100\text{\AA}$, $\Phi = 5^\circ$ and $Nd < 135\text{\AA}$, in excellent agreement with the HRTEM-analysis. The last value gives only an upper limit because it is difficult to detect the whole streak length.

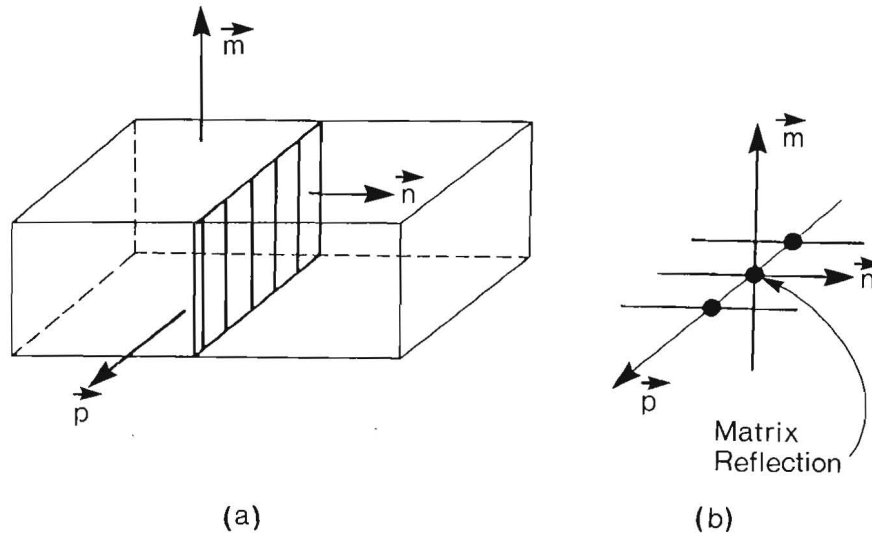


Fig. 11: (a) Geometry of an edge-on tilt boundary. The periodicity is along $\vec{p}$, $\vec{m}$ is the normal to the foil and $\vec{n}$ is the normal to the boundary plane. (b) The reciprocal lattice in the vicinity of a matrix reflection for the boundary in (a). (After Carter and Sass, 1981)

For a simplified model considering the boundary core as a uniform thin crystal with a plane spacing different from that in the neighbouring perfect crystals Lamarre and Sass (1983) found that the width in reciprocal space of diffraction effects due to the interface region can be directly related to the inverse of the real-space width of the distorted interface zone. Using a more realistic model in which the strain varies

smoothly across the grain boundary, Vaudin et al. (1986a) found that such a simple relationship does not exist. However, they still found that the length of the diffraction streak increases as the boundary becomes narrower.

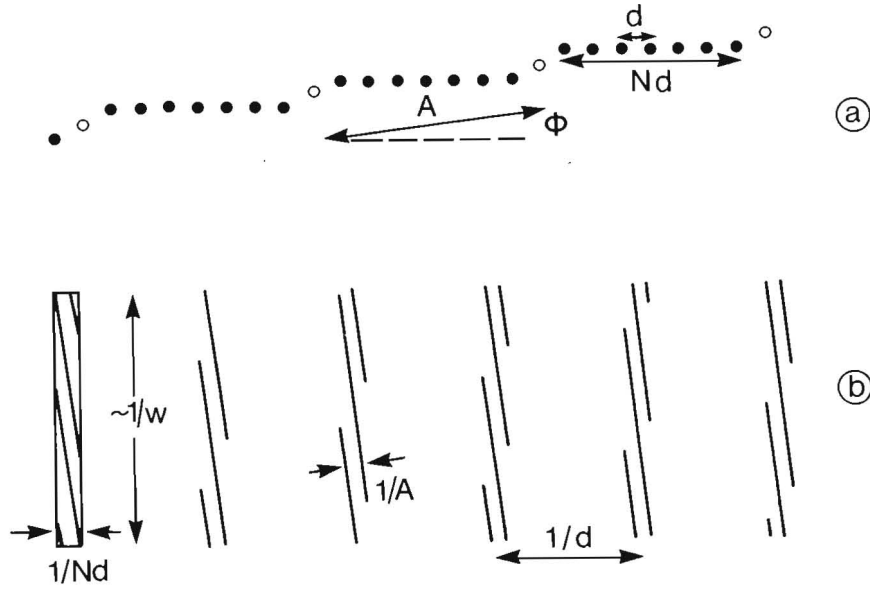


Fig. 12: (a) End-on dislocations in a faceted tilt boundary. The spacing between dislocations in the long facet plane is d . The true periodicity of the boundary is A . The average boundary plane is inclined to the symmetric facets by the angle Φ . (b) Diffraction effects expected from boundary shown in (a); w is the grain boundary width. (After Eastman, 1985)

Estimates of the structural width w of low and high angle (010) twist boundaries in Ge, Au and NiO (Vaudin et al., 1986b), MgO (Eastman, 1985) and olivine (Ricoult and Kohlstedt, 1983a) yield values in the order of 1 to 4 interplanar spacings normal to the boundary. On the other hand the expansion of the lattice normal to the boundary changes with bonding type being little if at all for semiconductors, small for metals ($\approx 10\%$) and large for ionic crystals ($\approx 25 - 40\%$), (Vaudin et al., 1986b). For low angle tilt boundaries the structural width is correlated to the dislocation spacing d . It is found for metals (Au, Cu-Bi) $w/d \approx 1$ (Carter et al., 1979; Hagège et al., 1982; Hall et al., 1982), semiconductors (Si) $w/d \approx 0.5$ (Carter et al., 1981), ceramics (Al_2O_3) $w/d \approx 0.5 - 1$ (Carter et al., 1980) and minerals (olivine) $w/d \approx 0.25 - 0.4$ (Ricoult and Kohlstedt, 1983a; 1983b).

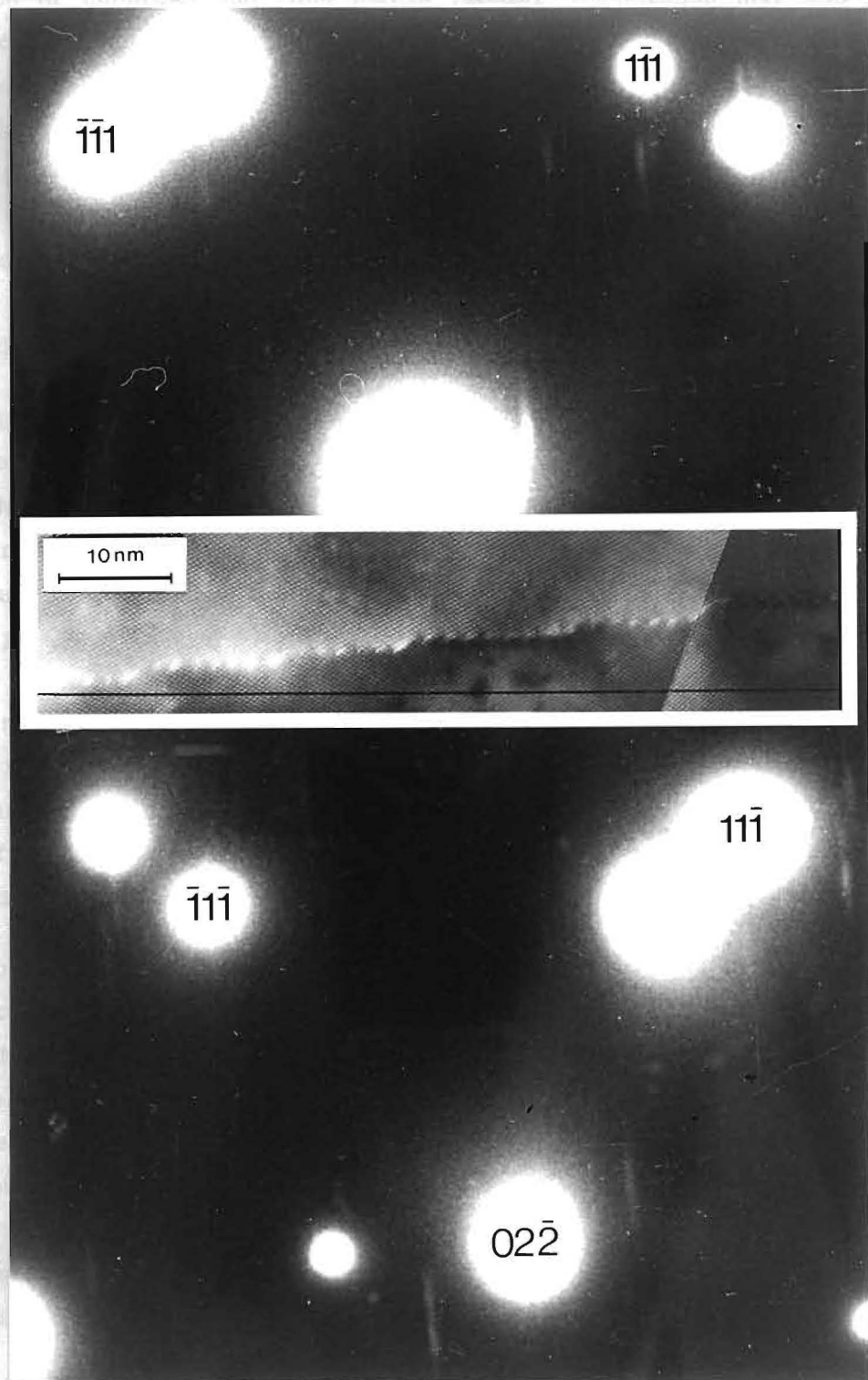


Fig. 13: [011] diffraction pattern and HRTEM image (inset) of a faceted $\Sigma = 51$ tilt boundary. Close to the reflections bundles of streaks are observed with the periodicities shown in Fig. 12.

The main difficulties in measuring grain boundary widths arise from grain boundary segregation of impurities and from narrow faceting. Segregation may lead to a structural change (Sickafus and Sass, 1984) or even to a separate grain boundary phase. For a faceted boundary the streak length is not necessarily proportional to the inverse of the grain boundary width, as in the case of a planar boundary. According to Fig. 12 the actual observed streak lengths will depend on both Nd and Φ and can vary within a bundle of streaks. The boundary width is related to the length of an entire bundle of streaks projected onto the direction normal to the average boundary plane. Thus, boundary widths obtained by simply measuring individual streak length are not meaningful if the boundary is faceted.

The diffraction geometry of an inclined grain boundary in a thin foil is shown in Fig. 14. The boundary has a structure that is periodic along the direction $\mathbf{p}$; the angle between the foil normal $\mathbf{m}$ and the grain boundary normal $\mathbf{n}$ is α . Because the foil is thin, the matrix reflection is streaked normal to the foil surface and is cut by the Ewald sphere to give the spot M . The periodicity along the direction $\mathbf{p}$ in the grain boundary produces an array of relrods which are aligned along $\mathbf{p}$ and are themselves normal to the boundary plane. The spacing of the relrods is inversely related to the grain boundary periodicity. They are cut by the Ewald sphere to give the grain boundary reflections N . The situation shown in Fig. 14b is for the kinematical case with the excitation error s_g negative. The displacement of the row of spots observed in the vicinity of the matrix reflection depends on the sign of s_g and the inclination of the boundary plane (Figs. 14c and d).

An example for an inclined grain boundary is shown in Fig. 15. The boundary is a near-coincident $\Sigma = 41$ [011] tilt boundary in Ge. The extra twist misorientation is accommodated by a network of secondary dislocations one set of which is aligned parallel to [011]. The periodically arranged secondary dislocations give rise to a row of extra reflections. Because $\mathbf{m} \times \mathbf{n}$ is almost parallel to the dislocation line and α is small the row of reflections goes through the matrix reflections M_1 and M_2 the separation of which is a measure of the extra twist orientation $\Delta\theta$. The spacing of the boundary reflections is $x = \Delta\theta/b \approx \Delta\theta/d_{(220)}$, thus deviding the distance between the higher order reflections in equidistant sections.

Previous investigations of interfaces by diffraction techniques have shown that the structural informations obtained are comparable with those of other direct methods. This agreement allows the application of diffraction techniques to interfaces where the facet and dislocation periodicities are too small to be resolved by imaging techniques other than lattice imaging, which is applicable only under certain conditions.

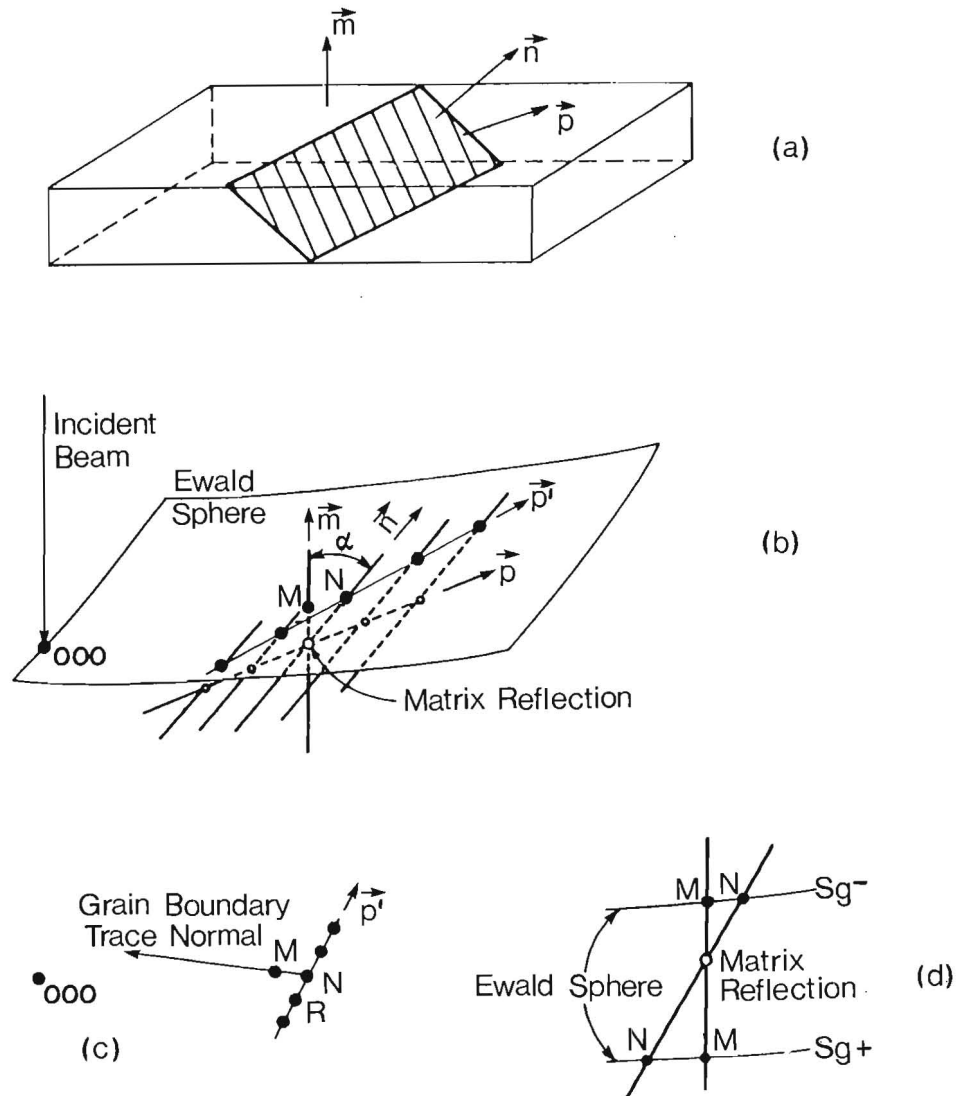


Fig. 14: (a) Geometry of an inclined grain boundary with a structure which is periodic along the direction $\vec{p}$. The angle between the foil normal $\vec{m}$ and the grain boundary normal $\vec{n}$ is α . (b) The reciprocal lattice near a matrix reflection for the inclined boundary in (a). The Ewald sphere intersects the reldods parallel to $\vec{m}$ and $\vec{n}$ at M and N , respectively. The direction of the projected periodicity is $\vec{p}'$. (c) Expected diffraction pattern for the boundary shown in (b). (d) Relation between the sign of the excitation error s_g and the relative positions of spots M and N . (After Carter et al., 1979)

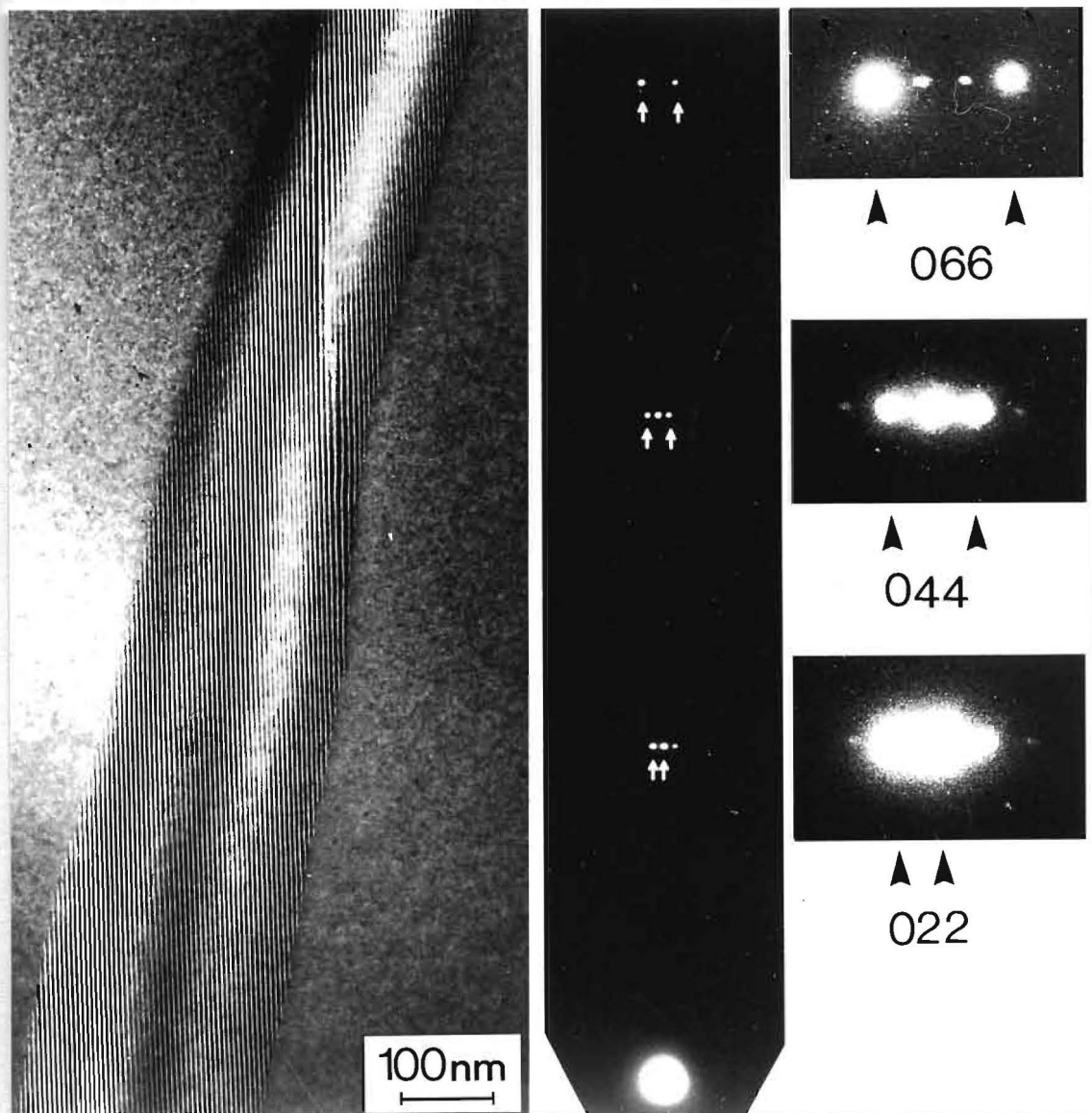


Fig. 15: Diffraction effects of an inclined near-coincident $\Sigma = 41$ [011] tilt boundary in Ge. The periodically arranged secondary dislocations give rise to a row of extra reflections. The matrix reflections are marked by arrows. TEM dark-field image taken with $g = 022$.

The particular importance of diffraction studies is the possibility of measuring the structural width of interfaces. Because diffusion through an interface occurs predominantly in regions of maximum disorder the structural width should provide a bound for the diffusion width. Values for the grain boundary diffusion width δ have been calculated by comparing experimental results for grain boundary controlled creep and rates of initial stage sintering - all of which yield values of $D_{gb}\delta$ - with results for grain growth and secondary recrystallization - both of which depend on $D_{gb}\delta^{-1}$ (Mistler

and Coble, 1974). D_{gb} is the grain boundary diffusion coefficient. For metals this analysis gives values for δ of 0.1 - 1 nm which are in reasonable agreement with those obtained from diffraction studies on high angle boundaries. For ceramics, Atkinson and Taylor (1981) have determined δ directly from grain boundary diffusion experiments on NiO. A comparison of the value D_{gb} obtained in short-time anneals with the value $D_{gb}\delta$ obtained in long-time anneals yields $\delta = 0.7$ nm. This value is in good agreement with that measured for the structural width of high angle twist boundaries in NiO (Vaudin et al., 1986b).

IV MECHANISMS OF MICROSTRUCTURAL DEVELOPMENT

Crystalline solids deform plastically by a number of alternative, often competing, mechanisms. According to Frost and Ashby (1982) these mechanisms operating under certain conditions of stress, temperature, strain rate, etc., may be divided into four main groups (Fig. 16):

- Low-temperature plasticity by dislocation glide
- Low-temperature plasticity by twinning
- Power-law creep
- Diffusional flow

In the following these mechanisms which may be influenced by metamorphic reactions will be briefly characterized and the main characteristics of the microstructure they produce will be described.

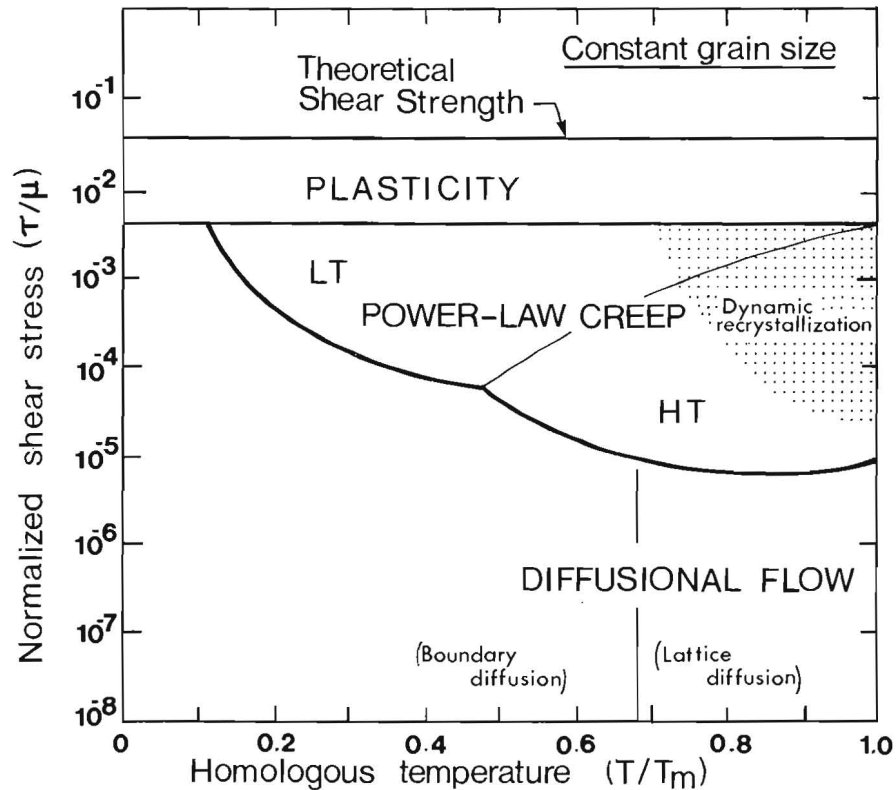


Fig. 16: Schematic illustration of a typical deformation mechanism map of normalized stress versus homologous temperature at constant grain size. The field of dynamic recrystallization characterizes migration recrystallization.

IV.1 Deformation mechanisms

IV.1.1 Low-temperature plasticity by dislocation glide

At low temperatures flow of polycrystals is by conservative or glide motion of dislocations provided an adequate number of independent slip systems is available (von Mises, 1928). This motion is limited either by intrinsic or extrinsic obstacles.

The main intrinsic obstacle to be considered is the Peierls potential. The Peierls force reflects the fact that the energy of a moving dislocation changes with lattice periodicity. Thus, the crystal lattice represents an array of long, straight barriers which at elevated temperatures may be overcome by thermally activated throwing out of kink-pairs (for a review see Guyot and Dorn, 1967). The Peierls mechanism of dislocations with spread cores where the spreading is not totally contained in the glide plane has been discussed in detail by Vitek (1984) and Veyssiere (1988). The microstructure characteristic of the Peierls mechanism often consists of long straight dislocation segments aligned along low index crystallographic directions (Peierls valleys).

Extrinsic obstacles to dislocation motion are other dislocations, solute atoms, precipitates and grain boundaries (see for example Haasen, 1974). Depending on the strength of these obstacles they introduce a friction-like resistance to slip or even block the motion of dislocations. As a consequence, dislocations bow out between the obstacles or pile up in front of them.

The hardening mechanisms described above determine the onset of plastic flow and work-hardening characteristics, which at low temperatures are measured in dynamic (constant strain rate) tests. With increasing flow stress different stages of work-hardening are observed. While for long time the work-hardening curve has been assumed to consist only of three stages recently two more stages have been observed in highly deformed metals and semiconductors (e.g. Haasen et al., 1986; Haasen, 1988). In stage I the dislocation microstructure is characterized by dislocation layers parallel to the slip plane often consisting of dislocation dipoles. In stage II the activation of oblique slip systems leads to dislocation multipoles, dislocation reactions, dislocation bundles and finally to a cell structure with dislocation walls containing more than one Burgers vector and sign. Further strain is accomplished by (screw) dislocation traffic through the (edge) dislocation walls. Dynamical recovery in stage III is controlled either by cross slip induced annihilation of screw dislocations in the soft phase (cell interior) or by climb induced annihilation of edge dislocations in the hard phase (cell wall). In semiconductors easy climb leads to the dissolution of the walls leaving behind screw dislocation networks in stage IV (Brion and Haasen, 1985). Finally in stage V cross slip

becomes prominent and annihilates the screw dislocation networks. In metals the situation is reverse, here cross slip is easier than climb. This leads to a drastic decrease in the rate of growth of the free dislocation density and the rate of shrinkage of the cell size (Zehetbauer et al., 1988). In stage V the walls dissolve and the microstructure changes to a subgrain structure characteristic of steady state deformation generally reached at elevated temperatures (see below).

IV.1.2 Low-temperature plasticity by twinning

For certain materials twinning is an important deformation mechanism at low temperatures (Tullis, 1980). Twinning is a variety of dislocation glide involving the motion of partial, instead of complete dislocations. The kinetics of the process often indicate that the nucleation, not propagation, determines the rate of flow. The microstructure of twinned crystals consists of twin lamellae with interfaces containing twinning dislocations. If the number of twinning systems is too small to produce a general strain in polycrystalline materials then in addition slip has to take place by this raising the flow stress.

A mechanism similar to twinning is the deformation-induced shear transformation. The transformation is accomplished by partial dislocations the motion of which along the interface broadens the new phase. An example for such a transformation is the ortho-to-clinoenstatite transformation (Coe and Kirby, 1975; see V.2 and VI.3).

IV.1.3 Power-law creep

At elevated temperatures dynamical recovery of the microstructure is sufficient to achieve steady state creep (balance of hardening and softening). Recovery is either by cross slip of screw or by climb of edge dislocations. These processes allow the overcoming of obstacles, dislocation annihilation and the arrangement of dislocations into low energy dislocation structures. Climb-controlled creep may be limited by dislocation core diffusion or by lattice diffusion the latter dominating at higher temperatures. Lattice dislocations may also enter grain boundaries and decompose into grain boundary dislocations (secondary dislocations). The motion of these dislocations in the interface produces grain boundary migration and grain boundary sliding if they have a Burgers vector component normal and parallel to the interface, respectively. The creep microstructure in general consists of a subgrain (low angle boundary) structure with free dislocations between.

Power-law creep may be accompanied by dynamic recrystallization. According to Poirier (1985) two mechanisms may be distinguished: (i) "rotation" and (ii) "migration"

recrystallization.

- (i) If the migration of (high angle) grain boundaries is impeded for example by impurities then new grain boundaries are produced by the incorporation of lattice dislocations into (low angle) subgrain boundaries, by this changing its misorientation. In addition also coalescence of slowly migrating subgrain boundaries occurs accelerating the rotation recrystallization.
- (ii) If the grain boundaries are mobile, then they may migrate into regions of high dislocation density. The driving force for grain boundary migration results from the energy stored in the dislocation microstructure. Growth of nuclei as well as bulging and coalescence of the migrating grain boundaries leads to the formation of new grains.

IV.1.4 Diffusional flow

At high temperatures and low stresses steady state creep can be diffusion controlled. The diffusion is either through the lattice (Nabarro-Herring creep) or at lower temperatures predominantly along grain boundaries (Coble creep). In fine-grained materials (grain size in the order of $1\ \mu\text{m}$) under certain conditions diffusion controlled grain boundary sliding can lead to superplasticity, that is to an considerable increase in strain rate. Crystals deformed by diffusional creep do not show any pronounced dislocation microstructure and texture.

IV.2 Metamorphic reactions

Metamorphism is the solid state mineralogical and often structural transformation of rocks due to a change of physical and chemical conditions. Metamorphic transformations may have a pronounced effect on the deformation of rocks. There are several ways in which the resistance of a rock to deformation can be altered by syntectonic metamorphic reactions; a review with illustrations on basic rocks is given by Brodie and Rutter (1985):

- Metamorphic reactions may produce transiently fine-grained products so that grain boundary sliding accommodated by diffusion may be greatly enhanced. Under certain conditions of temperature, grain size and grain size stabilizing factors the deformation mechanism may change to superplastic flow. Grain boundary sliding may be impeded by reaction-induced serrated interfaces or precipitation at interfaces.
- Dehydration reactions lead to water which may be in solid solution or if the solubility is exceeded will be also precipitated as fluid inclusions. Water introduces hydrolytic weakening in various minerals. The strongest effect has been observed in quartz (Paterson, 1985). The weakening is by reducing the glide resistance of

dislocations, facilitating climb processes as well as processes enabling grain boundary migration and therefore promoting dynamic recrystallization. The ductility may be changed by the fluid itself or by a modification of the point defect chemistry through fluids. It should be noted, however, that not all impurity-controlled defects soften materials. Some can produce hardening. The effect may be seen in the contrasting behaviour of doped Ge where Ga (p-type) raises the upper yield stress whereas As (n-type) lowers it, in proportion to the amount by which the extrinsic defect concentration exceeds the intrinsic concentration at a particular temperature (Patel and Chaudhuri, 1966). Water-induced alterations of basic rocks lead to lamellar or massif transformations of pyroxenes into amphiboles and sheet silicates (Buseck et al., 1980). These new phases may influence further deformation either by softening or hardening. On the other hand fluids promote brittle deformation. Pore fluid pressure reduces the hydrostatic pressure while segregation of fluids along grain boundaries reduces the cohesion of the material.

- Transformations can enhance the plasticity of a material if the transformation is associated with a significant volume change. It has been suggested that these volume changes can generate stresses that will assist in overcoming the resistance to intracrystalline slip (Poirier, 1985). A geological important transformation is the olivine-spinel transformation which might lead to enhanced plasticity deep in the mantle.

V CORRELATION BETWEEN MICROSTRUCTURE AND THERMO-MECHANICAL HISTORY

This section is the central part of the thesis because it shows the possibilities to relate the microstructure of rocks with their thermo-mechanical history. Different ways are given of estimating stresses, strains and temperatures the rocks have experienced. The relations described are mainly empirical, their generality by suitable normalization will be discussed. The parameters estimated characterize tectonic events. However, in nature deformation phases are complicated processes which may change through time in intensity, stress state and due to metamorphic reactions. Post-deformation recovery may lead to microstructural changes, too. Moreover, the inhomogeneity of the mineralogical and chemical composition of rocks combined with anisotropies at different scales favours heterogeneous deformation. Thus, the meaning of the thermo-mechanical parameters has to be discussed with respect to these problems. Another point of discussion will be multiple deformation and the question of how and how many different deformation events may be identified from relics in the final microstructure.

V.1 Microstructure - differential stress relations

It is known from the deformation of metals and ceramics that there exists a certain functional relation between steady state stress and dislocation density, subgrain size and recrystallized grain size, and between critical shear stress and twinning. Recently similar investigations have been done on geologically relevant materials with the aim to estimate the stress responsible for the natural deformation of rocks. In the following these "paleopiezometers" will be described and problems of their application will be discussed.

V.1.1 Dislocation density - stress

If a constant differential stress, $\sigma = \sigma_1 - \sigma_3$, is applied to a crystal, the dislocation density ρ within the subgrains increases to a steady state value (e.g. H  ther and Reppich, 1973). The stress dependence has in general been found to be $\rho \sim \sigma^m$. However, for a wide variety of metallic and ionic materials the exponent m is about 2 (Takeuchi and Argon, 1976). Thus ρ correlates with σ through a relation of the form

$$\rho b^2 = (\sigma/\mu)^2/\alpha^2 \quad (1)$$

where α is a constant and μ is the shear modulus. At a constant stress the dislocation density depends upon temperature only through the temperature sensitivity of μ and b .

Data of metals and ionic crystals fit to eq. (1) yield $0.8 < \alpha < 5.2$ (Takeuchi and Argon, 1976). The relations between ρb^2 and σ/μ for experimentally deformed silicates are shown in Fig. 17. The stress-dislocation density data for olivine were obtained from deformation experiments which cover a temperature range of 600°C, a strain rate range of 4 orders of magnitude, hydrostatic pressures between 10^{-1} and $1.5 \cdot 10^3$ MPa, and different water contents. Clearly, the dislocation density is primarily determined by the differential stress. The data fits are best represented by $\alpha = 3$. The scatter of the data shown is rather small, within a factor of 2. An α value between 1.5 and 3 has been also reported for calcite CaCO_3 (Goetze and Kohlstedt, 1977).

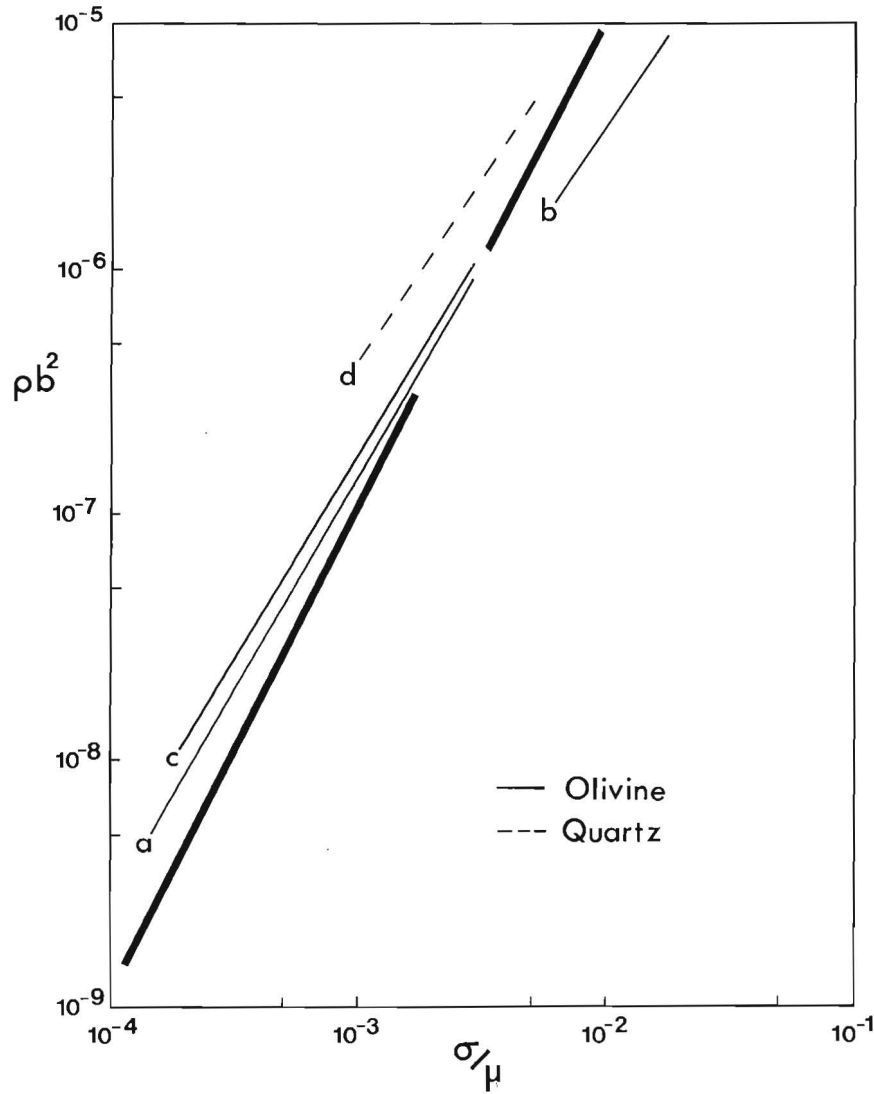


Fig. 17: Experimentally determined stress dependence of the dislocation density ρ in olivine and quartz following the relation $\rho b^2 = (\sigma/\mu)^m/\alpha^2$, with b mean Burgers vector, μ mean shear modulus, α and m constants (olivine: $b = 5.34 \text{ \AA}$, $\mu = 60 \text{ GPa}$, quartz: $b = 5 \text{ \AA}$, $\mu = 44 \text{ GPa}$). (a) Durham et al. (1977); (b) Zeuch and Green II (1979), dry; (c) Toriumi et al. (1984); (d) McCormick (1977), wet. Thick line is calculated with $\alpha = 3$ and $m = 2$.

The range of α -values for different materials is quite large. On the one hand this is due to the different methods used to measure dislocation densities. Dislocation densities obtained from etch-pits are generally underestimated because narrow spaced dislocations are not counted separately and dislocations intersecting the surface at an acute angle are not etched. Moreover, etching often depends on the crystallography of the etched surface. As a consequence, α may be too high. Dislocation densities are measured by TEM in two ways. First the density is measured as dislocation line length per unit volume. However, such a measurement presumes an accurate determination of the foil thickness at each area of interest. In general this is not easy in minerals because of large thickness variations within TEM samples due to preferential ion milling. The reason for this is not well understood and may vary for different rock compositions. The second method commonly used for geological materials is counting the number of dislocation intersections per unit area. For randomly oriented dislocations this density is exactly 1/2 the density measured as dislocation line length per unit volume (Schoeck, 1962). Since in general dislocations with different Burgers vectors occur different imaging conditions may be necessary to get all dislocations into contrast. Moreover, it is difficult to record heterogeneities in the dislocation density on the TEM scale. The latter may be avoided by increasing the sample statistics.

On the other hand a comparison of the α -values obtained by the TEM method shows that the magnitude of α is specific for certain classes of materials being 1.5 ± 1 for metals and ionic crystals and 3 ± 1 for minerals.

Eq. (1) has been derived from different models based on the elastic interaction of parallel dislocations and the interaction with forest dislocations either by dislocation-cutting or dislocation reactions (for a review see Lavrentev, 1980). The interaction constant α depends on the type of work-hardening model, its magnitude is in the order of unity, in agreement with experiment. Although much effort has been devoted to the physical foundation of the stress dependence of the dislocation density at present a more reliable theoretical prediction of the interaction constant for rocks is not possible.

Using dislocation densities as an indicator of stress in naturally deformed rocks one has to keep in mind that the dislocation density is not very stable against changes in temperature and stress. As a result, the average dislocation density in a deformed rock often reflects a late stage of stress-free anneal or a low-temperature high-stress emplacement stage, but is seldom representative of the main strain-producing episode, except when it is a major episode and when other paleopiezometers (see below) give the same answer.

V.1.2 Subgrain size - stress

It is now well established that high temperature deformation of a wide variety of materials is accompanied by the formation of subgrains coexisting with free dislocations. In the steady state of deformation the subgrain size d generally depends on the differential stress σ according to $d \sim \sigma^r$ (Twiss, 1977; Takeuchi and Argon, 1976; Raj and Pharr, 1986). For many materials, the value of the exponent r is close to unity. This observation is consistent with a number of theories of subgrain formation as will be discussed below. As a consequence, it has been suggested and quite commonly accepted that the proper relation between subgrain size and stress is of the form

$$d/b = k (\sigma/\mu)^{-1} \quad (2)$$

with k being a constant. In a recent compilation and analysis of data for the stress dependence of the subgrain size Raj and Pharr (1986) found that $k = 20$ works quite well for many metals (see also Staker and Holt, 1972; Takeuchi and Argon, 1976). For ionic crystals k is about 50 (Nicolas and Poirier, 1976). Similarly the data fits for silicates (Fig. 18) are within a factor of 3 best represented by $k \approx 100$. While the data may deviate from the anticipated $d \sim \sigma^{-1}$ behaviour, the empirically derived subgrain size-stress relations provide a sound basis for estimating paleostresses.

Although much of the scatter in the k values can be related to random error in the experimental determination of the subgrain size (Raj and Pharr, 1986) and to quantitative stereological problems, there seem to be specific values for certain classes of materials. Most problematic is the measurement of subgrain sizes in the $1 - 10 \mu\text{m}$ range ($2 \cdot 10^3 < d/b < 2 \cdot 10^4$). Here optical measurements of etched or decorated subgrains have their lower limit while conventional TEM has its upper limit. As a consequence, subgrain sizes in this range will be generally over- and underestimated, respectively (see VI.2.2).

Although water affects dislocation glide in silicates strongly, it does not seem to influence the subgrain size. In quartz, which generally is most sensitive to water, the subgrain size has been found independent of "wetness" and initial grain size (Koch, 1983).

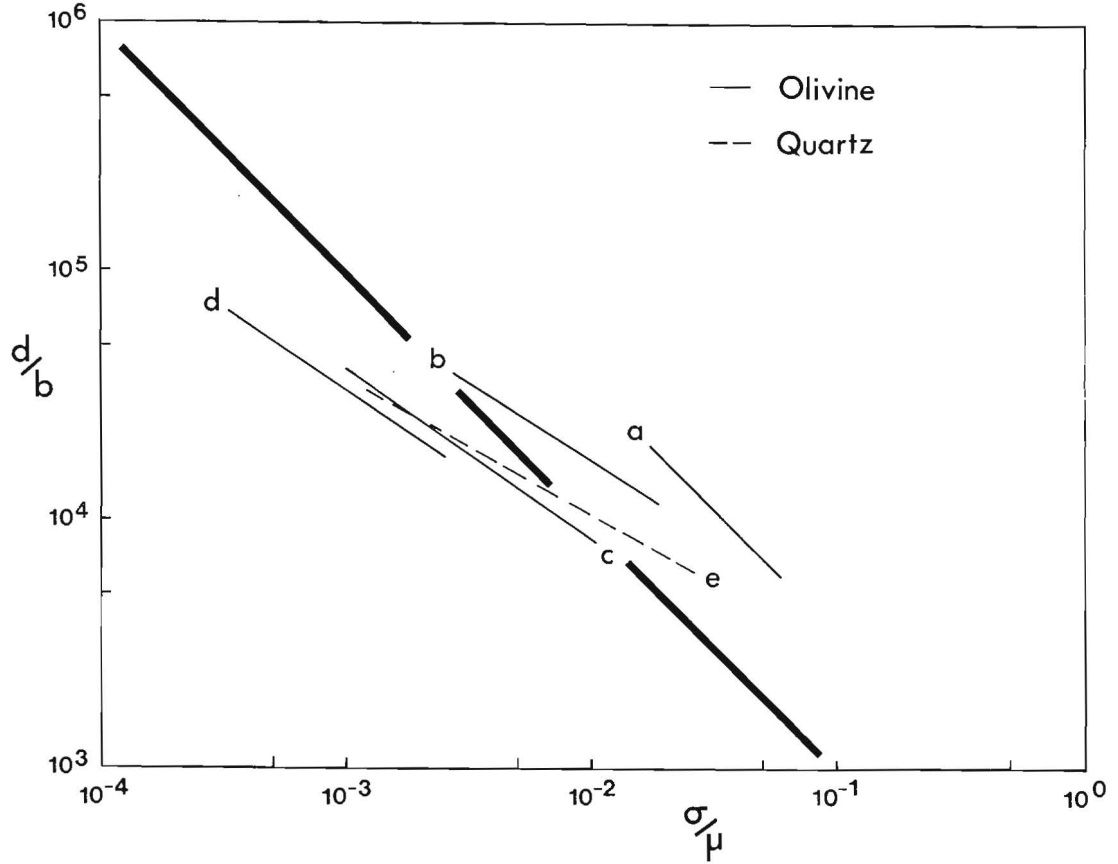


Fig. 18: Experimentally determined stress dependence of the subgrain size d in olivine and quartz following the relation $d/b = k (\sigma/\mu)^{-r}$, with k and r constants. For olivine d is the distance of decorated (100) tilt boundaries; for quartz d is the mean size of etched subgrains. (a) Mercier et al. (1977), (b) Ross et al. (1980), dry, (c) wet; (d) Karato et al. (1980); (e) Koch (1983), wet. Thick line is calculated with $k = 100$ and $r = 1$.

A number of theories have been proposed for the stress dependence of the subgrain size (for a review see Orlova and Cadek, 1986; Raj and Pharr, 1986). A common feature of these theories is that the driving force for subgrain formation is the reduction in strain energy resulting from the clustering of dislocations into subgrain boundaries. The models may be classified in static models in which the relation between subgrain size and stress is arrived at primarily through energy minimization alone, and dynamic models in which attention is paid to the dynamics of dislocation processes during deformation. All models end up with eq. (2). However, the constant k can only easily be estimated from the static model of Kuhlmann-Wilsdorf and van der Merwe (1982). According to their theory k is related to α by $k \approx 20\alpha$, with α being the constant of dislocation interaction (eq. (1)). This relation means that the subgrain size is about 20 times the mean distance between the free dislocations, $\rho^{-1/2}$. In the cases

where α and k have been measured on the same material a ratio of the same order has been found for metals and ionic crystals (Staker and Holt, 1972; Eggeler and Blum, 1981). The same seems to hold for minerals suggesting an universal character of k/α . The model of Kuhlmann-Wilsdorf and van der Merwe (1982) also predicts that the dislocation spacing in the subgrain boundaries is about $d/100$. For $d \approx 10\mu\text{m}$ the misorientation across the subgrain boundary should be a few tenth of a degree, in agreement with the observations on olivine (Fig. 32).

The stability of the subgrain size against changes of stress is contradictory. Laboratory experiments on NaCl (Pontikis and Poirier, 1976) and on olivine (Ross et al., 1980) show that the subgrain size increases rapidly during stress increase but is stable against a stress decrease. On the other hand Eggeler and Blum (1981) report a coarsening of the subgrain structure after stress reduction. Similar to the dislocation density the new steady state values are reached within a few percent of transient strain. High-temperature annealing leads to a coarsening of the subgrain structure and a decrease of the dislocation density. For olivine a factor 3 increase in subgrain size parallels a factor 10 decrease in dislocation density. Although the kinetics of recovery are not known it is believed that the recovery rate of the subgrain size is slower than that of the dislocation density. Thus, the subgrain size is more or less representative of the maximum stress experienced by a mineral, unless a later episode at a lower stress has produced a large enough strain allowing a new structure to evolve.

V.1.3 Dynamically recrystallized grain size - stress

At sufficiently high temperatures ($T > 0.5 T_m$) above a critical strain the deformation of materials may be accompanied by dynamic recrystallization. Depending on the temperature, stress and purity of the material the mechanism changes from "rotation" to "migration" recrystallization (see IV.1.3). While both types of mechanisms have been reported for non-metallic materials, e.g. for NaCl (Guillopé and Poirier, 1979) and for NaNO_3 (Tungatt and Humphreys, 1981), in metals only migration recrystallization has been observed clearly (Gottstein and Mecking, 1985). Independent of mechanism the dynamically recrystallized grain size D is a function of a applied stress only:

$$D/b = K (\sigma/\mu)^{-n} \quad (3)$$

Eq. (3) is similar to eq. (2) for subgrains, but the exponent n is usually somewhat larger than 1. For rotation recrystallization n is about 1 and the constant K is only slightly larger than k characterizing the subgrain size in eq. (2). In the migration recrystallization regime for metals n has been found about 4/3 and K in the order of 20

(Luton and Sellars, 1969; Bromley and Sellars, 1973). A compilation of the data for silicates (Fig. 19) shows that with a few exceptions the same parameters may be used for this group of minerals.

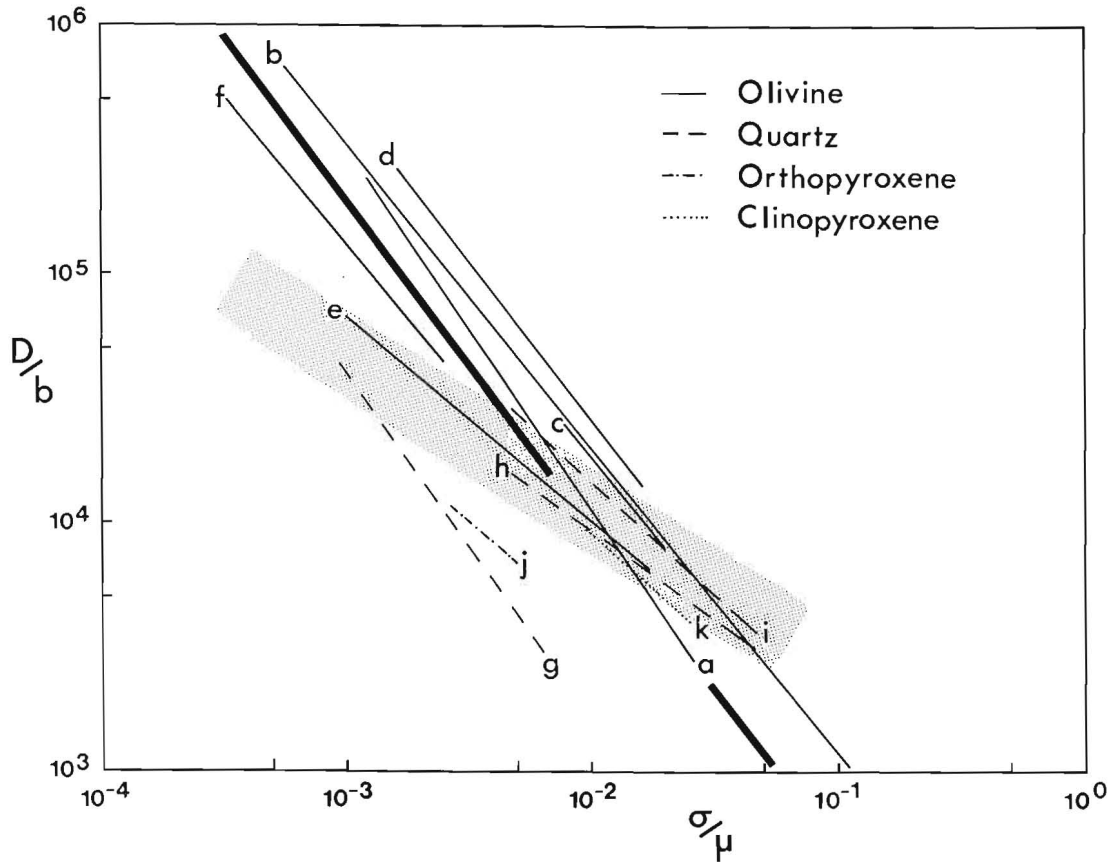


Fig. 19: Experimentally determined stress dependence of the dynamically recrystallized grain size D in different silicates following the relation $D/b = K (\sigma/\mu)^{-n}$, with K and n constants. (a) Post (1977), dry and wet; (b) Mercier et al. (1977), dry and wet; (c) Zeuch and Green II (1979), dry; (d) Ross et al. (1980), dry, (e) wet; (f) Karato et al. (1980); (g) Mercier et al. (1977), wet; (h) Christie et al. (1980), dry, (i) wet; (j) Ross and Nielsen (1978), wet; (k) Avé Lallemant (1978), dry. Thick line is calculated with $K = 20$ and $n = 4/3$. The hatched area represents the subgrain size-stress relations of Fig. 18.

The scatter of the data is largely due to random error in the experimental determination of the grain size and quantitative stereological problems, but may also result from other parameters. Luton and Sellars (1969) have shown that in Ni K decreases by alloying with Fe, while Bromley and Sellars (1973) did not observe any alloying effect of Al in Cu. The effect of water on migration recrystallization in

silicates is not clear. In the cases where silicates have been deformed under both dry and wet conditions no large differences have been found. On the other hand the curves for wet orthopyroxene and wet quartzite (curves i and g in Fig. 19) deviate strongly from the general trend.

It is also a big problem, that in most cases of the literature the grain size is given without mentioning whether it results from rotation or migration recrystallization. Therefore part of the curves in Fig. 19, especially those with small n - and K -value may belong to rotation recrystallization. The difference between both mechanisms in the range measured should be not large as is indicated by the hatched area which is representative of the subgrain sizes.

It should be mentioned that $n = 1$ and $K \approx 10^3$ has been suggested for rotation recrystallization (Mercier, 1980) from an analysis of discontinuities in grain size versus depth data for peridotite xenoliths. Similarly, data of Kohlstedt et al. (1976a) obtained from an analysis grain size as a function of dislocation density in naturally deformed olivine bearing rocks (which has been related to stress via Fig. 17), yield $n = 1$ and $K \approx 10^3$ for $\sigma/\mu < 4 \cdot 10^{-3}$ and $n = 4/3$ and $K = 60$ for $\sigma/\mu > 4 \cdot 10^{-3}$. The indirectly obtained K -values suggested for rotation recrystallization are an order of magnitude higher than those obtained experimentally for the subgrain size. The reason for this discrepancy is not understood.

A widely quoted theory to explain the recrystallized grain size-stress relation has been developed by Twiss (1977). Twiss determines the critical grain size for which the decrease in volume strain energy is exactly compensated by the increase in surface energy and assumes that it will be the "equilibrium grain size". However, as emphasized by Poirier (1985), this is an unstable equilibrium as the critical size found corresponds to an energy maximum: a grain larger than the critical size can always lower the energy of the system by growing. The expression for the critical grain size is of a similar form as eq. (3), with $4/3 < n < 3/2$ but entirely devoid of physical meaning.

The grain size is the most refractory feature of the microstructure. It adjusts to both increasing and decreasing stress levels only after relatively large strains ($\approx 10 - 50\%$; e.g. Guillopé and Poirier, 1979; Ross et al., 1980). It is also comparatively stable against static annealing. Only a small amount of recrystallization in quartz during static annealing occurs after 1 hour at 800°C (Hobbs, 1968) and numerous relic grains remain. Thus the recrystallized grain size reflects the last high-temperature major stress episode. However, even this paleopiezometer is thought to be the most reliable, it is beset by problems:

- As the paleopiezometers for rotation and migration recrystallization are different, the recrystallization mechanism has to be identified in the microstructure

otherwise the error in the stress estimates may be significant depending on the grain size. However, a distinction between the mechanisms is quite difficult because in nature slow migration and rotation of grain boundaries may operate simultaneously (Urai et al., 1983). If progressive misorientation of subgrains is dominant the change in misorientation should proceed more or less smoothly from subgrains to grains. The resulting microstructure should consist of clusters of "new" grains with similar orientation. In partially recrystallized rocks the main evidence for recrystallization by subgrain rotation has come from so-called core-and-mantle structures (White, 1976; Urai et al. 1986). The cores of the host grains pass out transitionally into mantles with increasing subgrain development and then into aggregates of recrystallized grains with similar size and orientations to nearby subgrains. If migration dominates the material will consist of deformed grains (porphyroclasts and recrystallized grains) in which subgrain misorientation does not exceed a few degrees. The smallest recrystallized grains may be strain-free. Grain boundary shapes are generally serrated or lobate. Frequently, bulging of grain boundaries leads to new grains (Kohlstedt and Weathers, 1980).

- Post-deformation annealing may lead to static recrystallization or to a coarsening of the dynamically recrystallized grain structure. Diagnostic of dynamic recrystallization is a deformation microstructure in the material assuming that a second deformation can be ruled out. According to Urai et al. (1986) a moderately well-developed 120° maximum of angles between grain boundaries does not seem to be inconsistent with dynamic recrystallization.
- In many cases grain boundaries may be stabilized against migration by a second phase. The grain size then does not correlate with stress (White, 1979).

V.1.4 Mechanical twinning - stress

Under certain conditions of stress, temperature and strain rate in a variety of materials twinning is preferred over slip. Reviews on twinning in metals and minerals have been given by Lubenets et al., (1985) and Tullis (1980), respectively.

To a first order of approximation, twinning occurs when the shear stress reaches a critical value on the twin plane in the twin direction and correct sense. Unlike the case for slip of crystals twinning is not thermally activated, and so the critical resolved shear stress does not depend strongly on temperature or strain rate. This has been confirmed for calcite CaCO_3 (Turner et al., 1954; Griggs et al., 1960), dolomite $\text{CaMg}(\text{CO}_3)_2$ (Higgs and Handin, 1959; Heard et al., 1978; Barber et al., 1981) and hedenbergite $\text{CaFeSi}_2\text{O}_6$ single crystals (Fig. 20, Kolle and Blacic, 1979). Thus extrapolations from laboratory measurements to the field situation are not necessary. There is also little effect of hydrostatic pressure on twinning (Turner et al., 1954).

Whether water content affects the ease of twinning in crystals has not been thoroughly investigated. It would not be expected to have an influence in calcite and dolomite, since there is no evidence of water affecting their slip behaviour. In silicates, however, water does play an important role in slip (Paterson, 1985) and might possibly affect twinning as well. However, since diffusion is unimportant in twinning and no silicon-oxygen bonds are broken (Kirby and Christie, 1977), it is fairly likely that water content of silicate crystals will not greatly affect their twinning behaviour. Thus there need be no uncertainty in the shear stresses that caused twinning in nature, even if the depth of burial or the fluid pressure and water content is unknown.

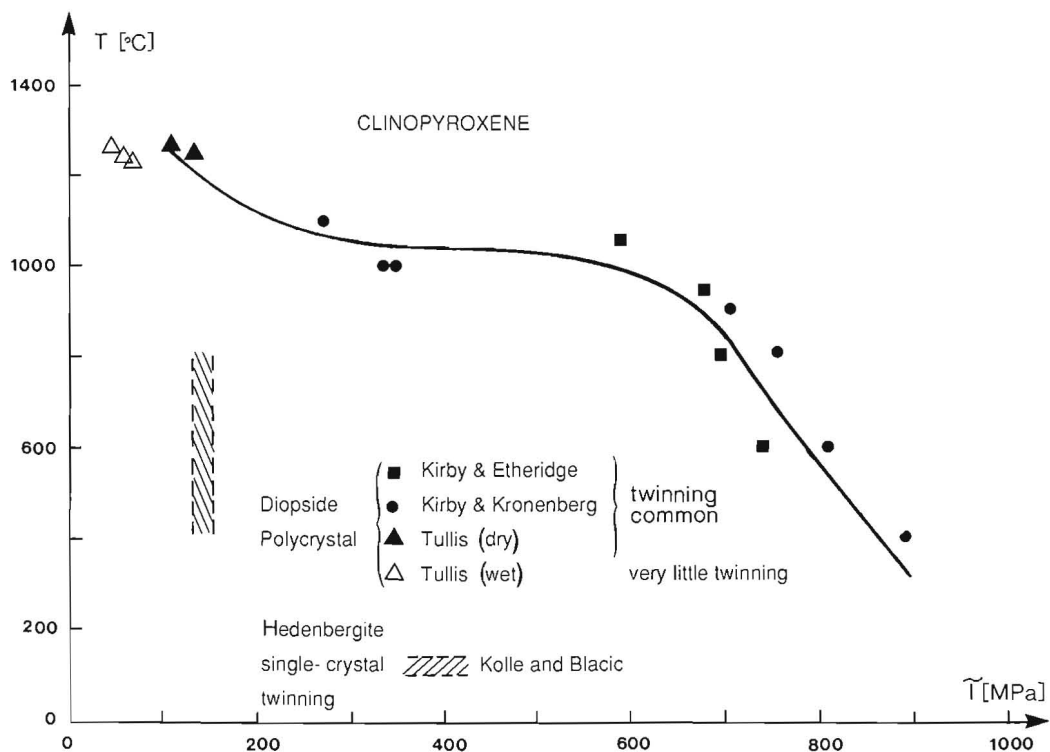


Fig. 20: Temperature dependence of the shear strength for clinopyroxene single crystals and aggregates. The hatched area shows the range of critical shear stresses for twinning of single crystals. The solid line shows the maximum shear stress for aggregates. (After Tullis, 1980)

Calcite, dolomite and clinopyroxene are rock-forming minerals with the greatest promise for the use of twinning to determine the magnitude of paleo-stresses. The critical resolved shear stresses measured for calcite, dolomite and clinopyroxene are about 10, 100 and 140 MPa, respectively. However, there is a difficulty of applying single crystal data to polycrystalline aggregates. As is shown in Fig. 20 for clinopyroxene the critical flow stress for aggregates may be up to one order of

magnitude higher than for single crystals and decreases with temperature. The conversion of the critical flow stress of polycrystals into critical shear stress has been done by taking the maximum Schmid factor $1/2$. In general, for polycrystals the reciprocal Taylor factor has to be used (Haasen, 1974) which for polycrystals without texture is about $1/3$. However, the temperature dependence would not change, only the stress difference between single and polycrystals would be larger. At low temperatures, where the critical shear stress for slip is much higher than for twinning, the aggregates support high stresses. This is because slip as well as twinning is required to allow differently oriented crystals to deform to sufficiently arbitrary shapes that the polycrystal can deform without creating voids at grain boundaries. With increasing temperature or decreasing strain rate a critical point is reached at which the critical shear stress for slip on the independent slip systems required for polycrystalline plasticity (von Mises, 1928) is lower than that for twinning, and therefore twinning does no longer develop. As slip in silicates is affected by water the critical temperature decreases for increasing water content. In naturally deformed polycrystals where mechanical twinning has been observed, the stress must have been at least as high as that for single crystal twinning, because at this stress level twinning starts in favourably oriented grains. A more detailed analysis of the orientations of crystals in a given aggregate that show or do not show twinning on one or more possible sets of twin planes might allow a more precise determination of the stress levels supported by the aggregate. If it can be shown or reasonably assumed that the principal stresses have not changed their orientation with time, then the direction of the principal stresses can be determined using the analysis of Turner (1953). This would then allow a determination of the Schmid factor for each crystal. A plot of the proportion of activated twin planes for given intervals of Schmid factors might yield an estimate of the actual value for the average applied stress on the aggregate instead of a lower bound. A method somewhat related to this was used by Jamison and Spang (1976) to estimate the stresses felt by limestones deformed in the laboratory as well as in the field. In the case of the experimental samples they were able to estimate the true stress magnitudes to within about 20%, and for strains of 2.5% and less their estimates were within 10% of the experimental values.

Considerable study has also been made of mechanical twinning in plagioclase feldspars (solid solutions of albite $\text{NaAlSi}_3\text{O}_8$ and anorthite $\text{CaAl}_2\text{Si}_2\text{O}_8$) (e.g. Marshall and McLaren, 1977) and clinoamphiboles (for a review see Kirby and Christie, 1977). However, in plagioclases there is evidence that the structural state of the feldspar (amount of order in the cation occupancy) effects the ease of twinning. As the extent of order is a function of the temperature history of the crystals it would not be easy to determine in what structural state a sample had become twinned.

In the case of clinoamphiboles Rooney et al. (1975) reported a critical shear stress for

twinning on (101)[101] of about 350 MPa at 400°C and 150 MPa at 800°C. However, as discussed by Kirby and Christie (1977) it seems likely that these stresses are instead the stresses required for shearing on the reciprocal (100)[001] slip system when it is in a kinking position. For the (100)[001] twinning system it has been suggested by Dollinger and Blacic (1975) that the stress required for its activation is greater than that for slip on the same system and therefore tends not to occur. Because of the insufficient experimental situation as well as the difficulties mentioned, at present feldspars and clinoamphiboles are not useful for estimating paleostresses.

V.1.4 Distinction between different deformation events

The preceding sections have shown that the deformation-induced microstructure (dislocations, subgrains, recrystallized grains) may be changed by static recovery or a deformation event following the deformation of interest erasing or resetting the stress indicators. In general, the last deformation phase is recorded. However, as the three paleopiezometers equilibrate at different rates to changes of stress, differences in stress level from the three piezometers can be related to the tectonic history of rocks. Of the three microstructural parameters the dislocation density is most easily altered by static annealing and stress changes. The subgrain size is more refractory while the grain size is the most stable feature of the microstructure. In the following differences in the stress levels predicted from the three microstructural parameters will be discussed.

(i) $\sigma_D = \sigma_d = \sigma_p$

In the most straightforward situation all three parameters yield the same paleostress level, indicating that the microstructure is representative of the last major deformation period and has not adjusted to stresses during uplift, erosion and cooling. Good examples are given by quartz mylonites of various shear zones (Tab. 2). With only two exceptions the stress levels agree quite well. Remarkable is also the agreement between the different shear zones. The average stress to produce a quartzite shear zone is 135 ± 25 MPa. It should be noted that such a narrow stress range is the result of using suitably normalized piezometers derived from different classes of materials. Former estimates based on fewer experimental calibrations show larger scatter.

(ii) $\sigma_D < \sigma_d = \sigma_p$

This situation is indicative of two deformation phases. The first major phase has lead to dynamic recrystallization, while the second phase at a higher stress has changed the subgrain and dislocation structure. If the second deformation is intense enough, it may also lead to partial dynamic recrystallization producing a bimodal grain size distribution and $\sigma_{D1} < \sigma_{D2} = \sigma_d = \sigma_p$.

Tab. 2: Differential stresses σ in different shear zones estimated from quartz microstructures according to $\sigma_D = 1390 D^{-3/4}$, $\sigma_d = 2200 d^{-1}$, $\sigma_\rho = 6.6 \cdot 10^{-3} \rho^{1/2}$, with σ in MPa, D and d in μm , and ρ in cm^{-2} (D = recrystallized grain size, d = subgrain size, ρ = dislocation density). Stress values in brackets are calculated with $d_{\text{Opt}} = 6 d_{\text{TEM}}$.

	D [μm]	d_{Opt} [μm]	d_{TEM} [μm]	ρ [$10^8/\text{cm}^2$]	σ_D [MPa]	σ_d [MPa]	σ_ρ [MPa]	References
Moine Thrust (Scotland)								
Loch Eriboll	20			2.5	150		100	W, B, C & K, 1979
	20	13	4.5	0.5	150	170	50	W, 1979
Stack of Glencoul near the stack	15		2	5	180	(180)	150	W, B, C & K, 1979
	13	20	2.5	10	200	110	210	O & C, 1984
	17	17	2.5	5	170	130	150	
	35	18	2	4	100	120	130	
	33	16	3.5	4	100	140	130	
Allt nan Sleagh	61	23	4	4	60	100	130	
Knockan Crag				8			190	W, B, C & K, 1979
Idaho Springs-Ralston shear zone (Colorado)	15		2	5.5	180	(180)	160	K & W, 1980
Coyote Mountain mylonite zone (California)	22			2.1	140		100	C & O, 1980
Ikertoq shear zone (Greenland)	25-50		5-10	1-4	70-120	(40-70)	70-130	K, C, W & B, 1979
Portalegre shear zone (Brasil)	25		5	5	120	(70)	150	S & H, 1987

An example for case (ii) (Skrotzki, 1986) is presented in Tab. 3 for quartz-bearing rocks of the Variscan collision zone exposed in the Oberpfalz (NE-Bavaria). Here the Moldanubian (Mo) unit has been thrust onto the Saxothuringian (Sax) unit. The immediate boundary between both units is represented by the small zone of Tirschenreuth-Märing (ZTM) (Vollbrecht et al., 1988). According to Stein (1988) the tectonic evolution of this area comprises four variscan folding events with the peak of the high-temperature low-pressure (HT-LP) metamorphism coinciding with F_3 in Mo and with F_2 in Sax. The dislocation structure observed, which reflects the last deformation event, indicates a unique folding event F_4 under chlorite-facies conditions. This deformation event has been more intense in the ZTM leading here to a mylonitization. In this case all the different stress estimates give a unique value of 120 ± 10 MPa for F_4 , in agreement with the mylonitization stresses given in Tab. 2. The grain structure becoming coarser from north to south probably has been produced during F_3 and

reflects the decrease of the HT-LP metamorphism.

Tab. 3: Paleostress estimates from quartz microstructures in rocks of different structural units of the Oberpfalz (see text). Parameters and equations used are described in Tab. 2.

	D_1 [μm]	D_2 [μm]	d_{TEM} [μm]	ρ [$10^8/\text{cm}^2$]	σ_{D1} [MPa]	σ_{D2} [MPa]	σ_d [MPa]	σ_ρ [MPa]
Sax	60	—		3	60	—		110
ZTM	290	25	4	4	20	120	(110)	130
Mo	400	—		4	15	—		130

Another example is the analysis of the deformation history of olivine-bearing kimberlite nodules by Götze (1975). The grain size distribution is bimodal, and the dislocation density in the small grains is the same as in the large grains. The size of the small (second generation) grains and the dislocation density yields $\sigma \approx 300$ MPa, the size of the large (first generation) grains gives $\sigma \approx 10$ MPa. Götze (1975) concluded that the high dislocation density and small grain size (high σ) were introduced during the explosive ascent of the nodules, while the large grain size (low σ) was probably characteristic of the stress level in the mantle.

A further example is presented in section VI where the microstructure in peridotites of the Balmuccia massif reflects the low stress intrusion of mantle material into the crust and a younger high stress crustal deformation.

(iii) $\sigma_D \approx \sigma_d < \sigma_\rho$

Such a result is indicative of a stress pulse changing only the dislocation microstructure. Such conditions may be found for example in cataclasites (Skrotzki, 1988, unpublished KTB results).

(iv) $\sigma_D > \sigma_d \approx \sigma_\rho$

If the deformed material has been annealed the subgrain size and the dislocation density recovers and the stress derived from these parameters will be underestimated. If, however, the microstructure does not show any signs of recovery, condition (iv) may be also the result of a second intense low

stress deformation event not leading to dynamic recrystallization.

$$(v) \quad \sigma_D \neq \sigma_d \neq \sigma_p$$

This situation reflects a complicated combination of deformation and annealing periods and, in general, should be interpreted cautiously.

In any way, because of the scatter in the piezometers, statements about the deformation history of rocks seem to be reasonable only if differences in the stress estimates are at least a factor 3.

V.2 Microstructure - strain relations

At low temperatures in certain crystals deformation is accomplished by a deformation-induced shear transformation. This has been shown by Coe and Kirby (1975) for orthopyroxene transforming to clinopyroxene. The reverse situation is found in large pigeonite exsolution lamellae in augite where misfit stresses induce a transformation to orthoenstatite (Robinson et al, 1977). Similarly, transformations of clinopyroxene to clinoamphibole and clinoamphibole to sheet silicates (Chisholm, 1973) can be achieved by simple shear. Whether these transformations may be induced by deformation remains open. In the following it will be shown how to estimate the strain in orthopyroxenes from deformation-induced clinoenstatite lamellae.

According to measurements of Coe and Kirby (1975) the transformation of ortho- into clinoenstatite involves a shear of $\delta = 13.3^\circ$ in the (100) plane along the [001] direction. Taking the width y and the separation L of the clinoenstatite lamellae and assuming they are extended across the grain then the shear strain of the orthoenstatite phase may be calculated according to Fig. 21 by

$$\gamma = \frac{x}{y+L} = \frac{y \tan \delta}{y+L} \quad L \gg y \quad \approx \frac{y \tan \delta}{L} \quad (4)$$

with x being the amount of shear.

Due to large variations in the stacking fault widths and thicknesses the strain $\epsilon \approx \gamma/2$ gives only an estimate of the order of magnitude. This method of estimating strains is limited by shear strains larger than 23.6% at which the orthopyroxene is totally transformed. The strain of mechanically twinned materials may be estimated in the same way by considering the width and extension of twin lamellae.

If an aggregate of particles like grains is strained homogeneously, the final shape and orientation of each particle depends on five independent parameters. These are: (a) the initial shape of the particle, (b) the initial axial orientation, (c) the strain intensity, (d)

the orientation of the stress field and (e) the ductility contrast of the particles to the total particle/matrix system. From the resultant shape and orientation of a group of particles it is difficult if not impossible to determine all these parameters. The total strain of a rock may be the sum of a sequence of processes affecting different components in different geological environments. A technique of finite strain analysis using elliptical particles has been established by Dunnet (1969) and Elliot (1970). According to Elliot (1970) the natural finite strain is given by

$$\epsilon = 1/2 \ln A/B \quad (5)$$

with A and B being the short and long axis of the strain ellipsoid, respectively. If the deformed particles were originally spherical, the axial ratio of the strain ellipsoid equals the ratio of the short and long particle axis.

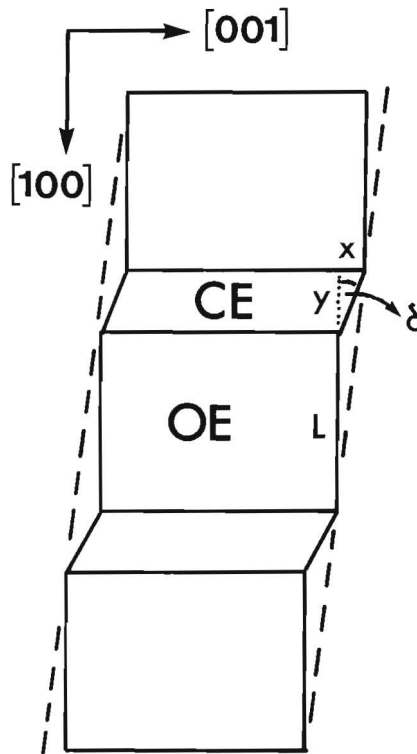


Fig. 21: Shear produced by the ortho-clinoenstatite (OE/CE) transformation.

To determine finite strains from grain shapes it has to be excluded that the material is recrystallized. If dynamic recrystallization takes place in waves the "finite strain-clock" is reset periodically and the grain shape and preferred grain shape orientation observed

represents the last strain increment (Lister and Snoke, 1984). If the recrystallization process is continuous grain growth by preferred grain boundary migration may produce a grain shape anisotropy, too. Grain boundary sliding is another mechanism whereby an aggregate can change shape, but without profound changes of forms of the individual crystals. Another problem of strain estimates arises from pressure solution taking place particularly at low metamorphic grades or even diagenesis. In this case individual grains undergo solution on the sides which face the greatest shortening direction, and this material is transferred to and crystallized on the sides which face the greatest extension direction. Because of these problems strain estimates from grain shape analyses are very questionable and therefore should be treated with great caution. For a more detailed discussion of strain analyses the reader is referred to Ramsay and Huber (1983).

V.3 Microstructure - temperature relations

V.3.1 Predominant slip system - temperature

Generally slip in crystals takes place on crystallographically different slip systems defined by the slip plane and the slip direction. To activate these slip systems different critical resolved shear stresses are necessary. As has been shown for ionic crystals with NaCl-structure (Skrotzki, 1980; Skrotzki and Haasen, 1981) this "plastic anisotropy" depends on several parameters, like temperature, the level of impurities and the degree of ionicity of the material. While in ionic crystals the choice of the slip systems can be understood in terms of Peierls and dislocation-impurity interactions the situation in minerals with ionic-covalent bonding is more complicated. So far no systematic studies have been carried out to solve this problem. However, as a rule of thumb in minerals such systems are preferred, slip on which produces a minimum of covalent bond breaking.

Studies on the plastic anisotropy of crystals are generally applied to single crystals and single slip conditions. However, a homogeneous plastic deformation of a polycrystal with a random distribution of grain orientations requires the activation of five independent slip systems (von Mises, 1928). Therefore, in general several types of slip systems are necessary to deform a polycrystalline solid. The role of the plastic anisotropy in the deformation of polycrystalline halides has been demonstrated by Skrotzki and Haasen, 1984. The results suggest that the required five independent slip systems have not been activated equally, in particular in crystals with a texture. This assumption is supported by computer simulations of a homogeneous deformation of quartzite (Lister et al., 1978) showing that the fourth and fifth slip system contribute to strain about one to two orders of magnitude less than the most active (primary) system.

According to Paterson (1978) the absence of these slip systems is compensated by a spatially heterogeneous deformation and other mechanisms like grain boundary sliding. Thus, in polycrystalline materials there is a predominance of slip on the primary slip system. Maps showing the predominance of different slip systems have been determined experimentally for quartz and olivine (see for example Nicolas and Poirier, 1976). The map for olivine is presented in Fig. 22.

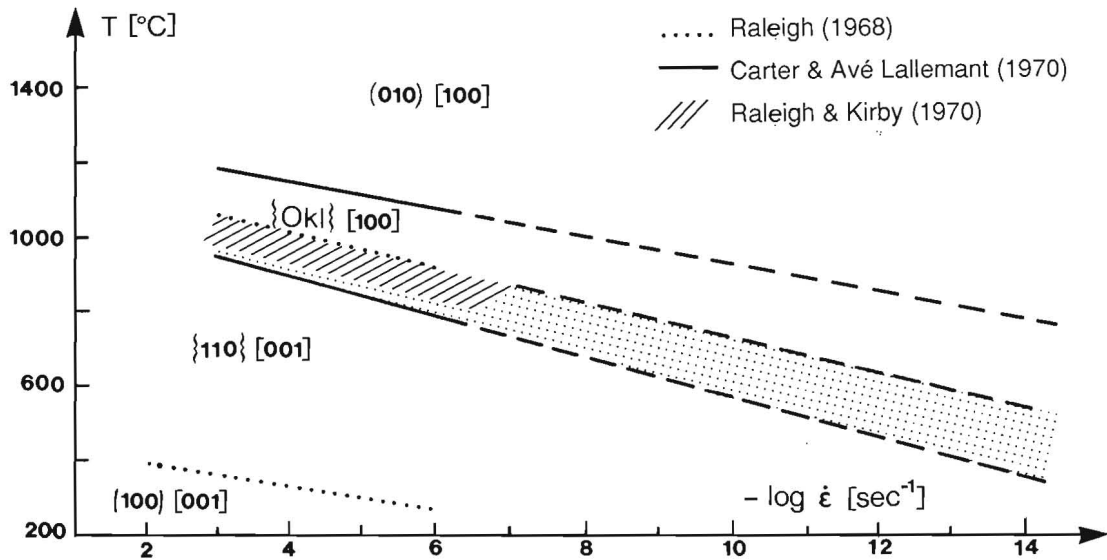


Fig. 22: Predominant slip system in olivine as a function of temperature and strain rate. Hatched area represents the uncertainty in the transition from predominant [001] to [100] Burgers vector. Dashed lines are extrapolations to geological strain rates.

The application of "predominant slip system maps" to natural conditions should be done very cautiously. In general, the boundaries between different regions are difficult to determine and shifts are common for different authors. Moreover, the range of strain rates and hydrostatic pressures covered by experiment is small. Hence extrapolations to geological conditions (strain rates between 10^{-13} to 10^{-14} sec^{-1}) are rather uncertain. Estimates of natural strain rates are based on geodetic measurements from surface strains, as along the San Andreas fault (Whitten, 1956), from rebound rates associated with crustal unloading (Crittenden, 1967), from displacement rates of sea floor spreading (Heirtzler et al., 1968; Le Pichon, 1973), and from estimated rates of crustal shortening in orogenic regions (Gilluly, 1972). If solid state mantle convection is responsible for crustal plate motion and deformation, then mantle strain rates should be of the same order. Based on the results on olivine estimates of upper and lower deformation temperatures are possible only with an uncertainty of about 200°C.

V.3.2 Transition temperature from shear transformation to slip

It has been shown for orthopyroxene and may also hold for other minerals that at a given strain rate there is a critical temperature above which the material slips instead of transforming (Fig. 23). The critical temperature is strongly dependent on strain rate and only weakly on water content. Thus maps like Fig. 23 offer the possibility to estimate upper and lower limits of the deformation temperature depending on the observed microstructure. Problematic again is the extrapolation to geological strain rates.

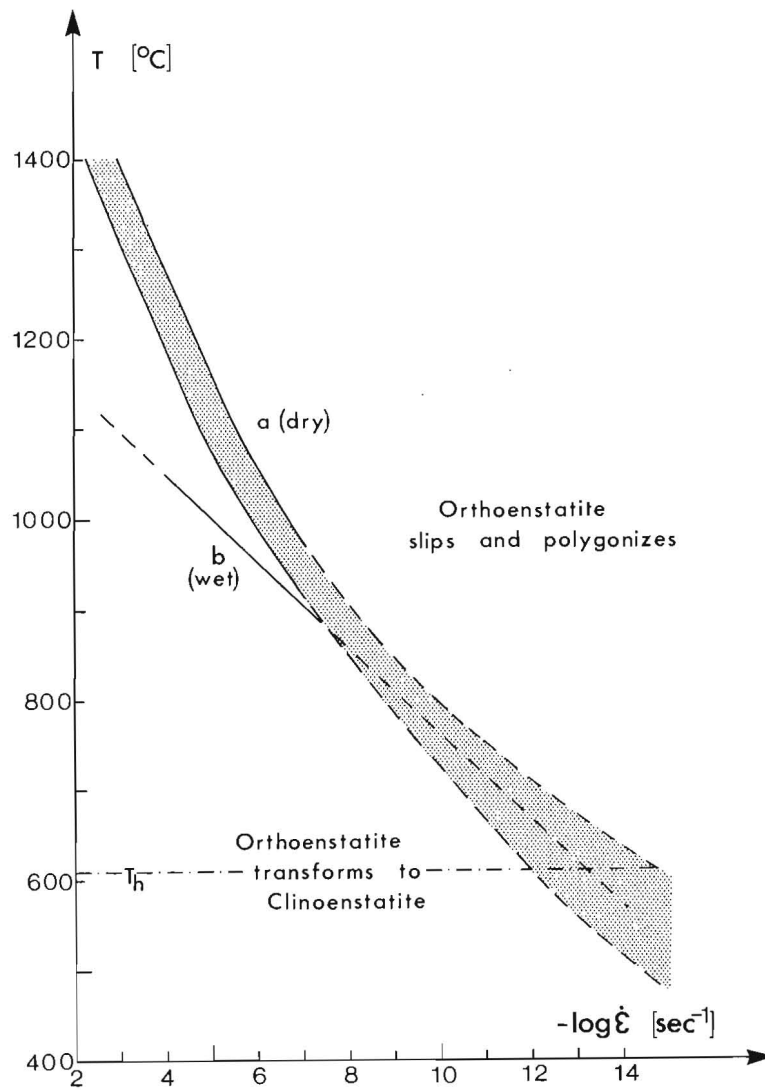


Fig. 23: Experimentally determined transition in a $T - \dot{\epsilon}$ diagram between the regimes in which orthoenstatite transforms to clinoenstatite and orthoenstatite slips and polygonizes, respectively. Measurements (solid lines): (a) Raleigh et al. (1971), (b) Ross and Nielsen (1978). Dashed-pointed line is the hydrostatic phase boundary at 1 GPa proposed by Grover (1972) according to $T_h = 566^\circ\text{C} + (46^\circ\text{C/GPa}) P$.

V.4 Application of deformation mechanism maps

Deformation mechanism maps (Frost and Ashby, 1982; Fig. 16) display the relationship between the three macroscopic variables: stress, temperature and strain rate. If any of these variables is specified, the map can be used to determine the third. The procedure is as follows. First from microstructural observations the predominant deformation mechanism has to be determined. Then constitutive equations characterizing the deformation regimes are taken to calculate the third from two known parameters.

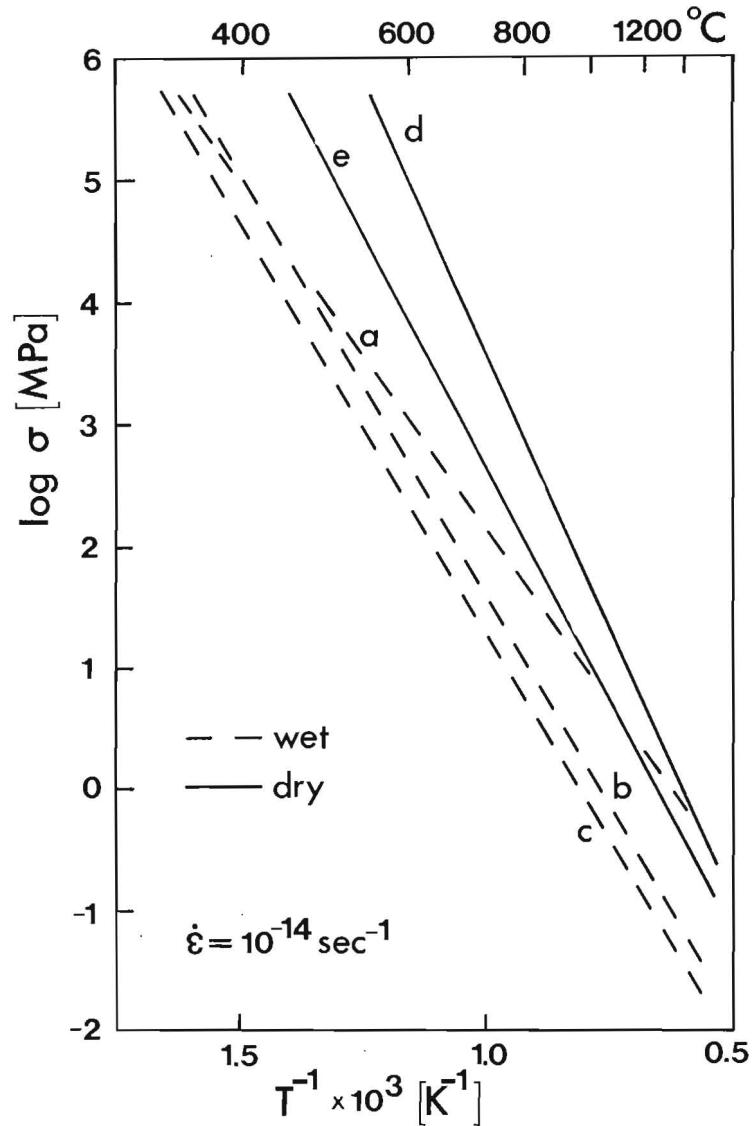


Fig. 24: Temperature dependence of the steady state stress of experimentally deformed dry and wet dunite extrapolated for a strain rate $\dot{\epsilon} = 10^{-14} \text{ sec}^{-1}$. (a), (b) Chopra and Paterson (1981); (c), (d) Post (1977); (e) Chopra and Paterson (1984).

The temperature may be estimated, for example, from Fig. 24 if the stress is known and the strain rate is assumed to be $10^{-14} \text{ sec}^{-1}$. Constitutive equations have been determined for various minerals, but after suitable normalization also equations obtained for isomechanical materials may be used (Frost and Ashby, 1982).

The main problem in the application of deformation mechanism maps in determining deformation parameters of naturally deformed rocks is the lack of sufficient experimental results. In particular, the influence of parameters like water content and rock composition has not been investigated thoroughly. As has been shown for crustal rocks by Hansen (1982) these parameters may strongly change the flow laws.

VI MICROSTRUCTURE AND TEXTURE IN PERIDOTITES OF THE BALMUCCIA MASSIF AND RELATION TO ITS TECTONIC EVOLUTION

Naturally deformed mantle rocks being investigated most are peridotites both xenoliths and crustal intrusions (massifs). Xenoliths in general reflect mantle deformation while the microstructure of crustal intrusions may be overprinted by the intrusion process as well as further crustal deformation (for a recent review see Mercier, 1985). In this section a microstructural and textural model analysis will be described on olivines and orthopyroxenes of peridotites of the Balmuccia massif (Fig. 25).

The purpose of the study is threefold:

- To give a detailed characterization of the microstructure and texture of the Balmuccia massif,
- to analyse the microstructure with respect to the thermo-mechanical history of the massif and
- to look for a correlation between microstructure and texture.

VI.1 Geological framework

The Balmuccia massif is one of several ultrabasic bodies intercalated within the "Basischer Hauptgesteinszug" of the Ivrea zone (Fig. 25). The massif has the form of a lens (6 km x 0.7 km) with its longest extension in the NS direction. It is surrounded by metabasites of the "Basischer Hauptgesteinszug".

The ultrabasic rocks of the Balmuccia massif consist of spinel bearing lherzolites, dunites and harzburgites, with clinopyroxene-poor lherzolites being the main constituent (Shervais, 1979a). According to microprobe analyses of Shervais (1979b), Garuti and Friolo (1979) and Sinigoi et al. (1983) the compositions of the different rock forming minerals are as follows: Olivine is composed of about 90 % forsterite. The orthopyroxenes are enstatites with compositions around $Wo_1En_{88}Fs_{11}$. Diopside is found as exsolution lamellae along (100) planes and as inclusions in large orthopyroxene grains. The diopside compositions range from $Wo_{50}En_{50}Fs_0$ to about $Wo_{50}En_{45}Fs_5$. Generally clinopyroxene is in contact with orthopyroxene. The spinels are observed in two variations: Fe (II)-spinel and Al-spinel with about 9% Cr_2O_3 . They exist at triple points as well as inclusions in olivine. Small accessory amphiboles are Ca-rich.

According to Boudier et al. (1984) the peridotites display a pronounced foliation, defined as the plane of mineral flattening, and a lineation, which is the mineral elongation direction contained in the foliation. The foliation dips steeply to the west from about 85° in the south to about 60° in the north (Fig. 26). Its strike varies

progressively between 5° in the south to 350° in the north. Simultaneously the orientation of the lineation changes from $5^\circ/45 - 65^\circ$ to $350^\circ/10 - 15^\circ$. Foliations and lineations in the northeastern domain are concordant with those in the granulitic gabbros of the eastern contact (Boudier et al., 1984).

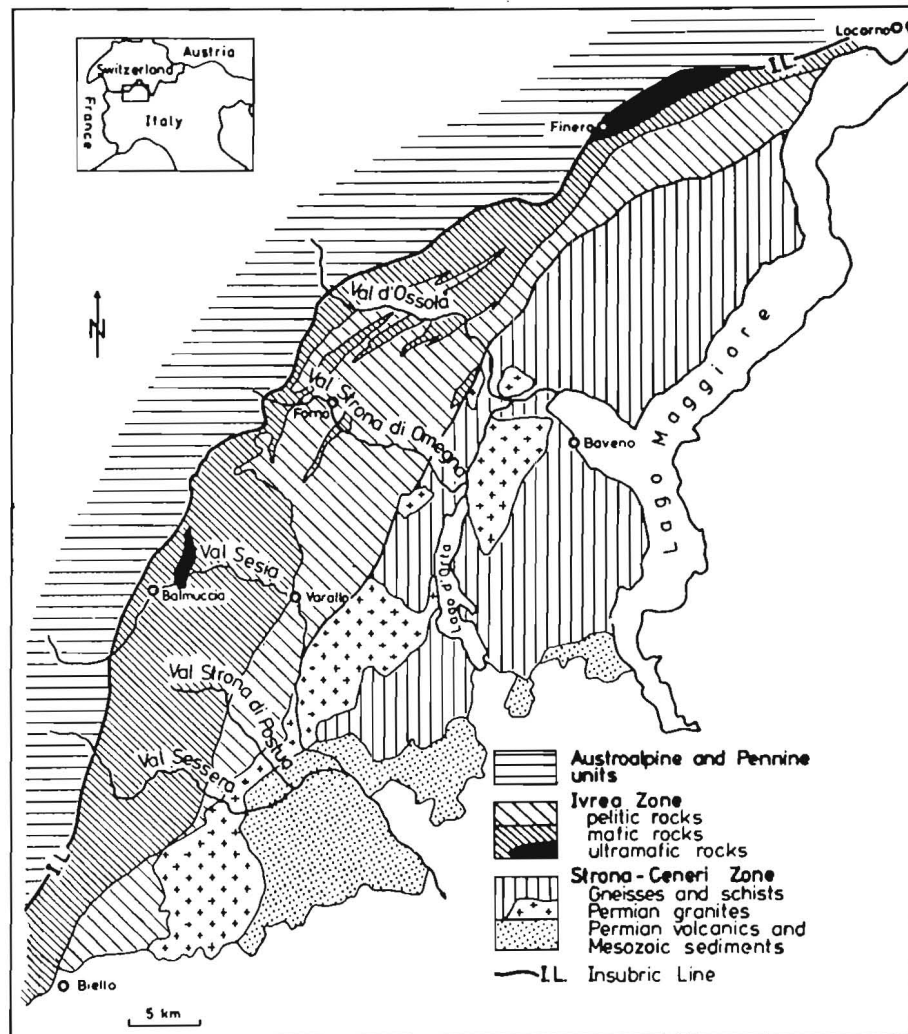


Fig. 25: Map of the Ivrea zone. The Balmuccia and Finero ultramafic bodies are marked in black in the W and N part of the region, respectively. (After Hunziker and Zingg, 1980)

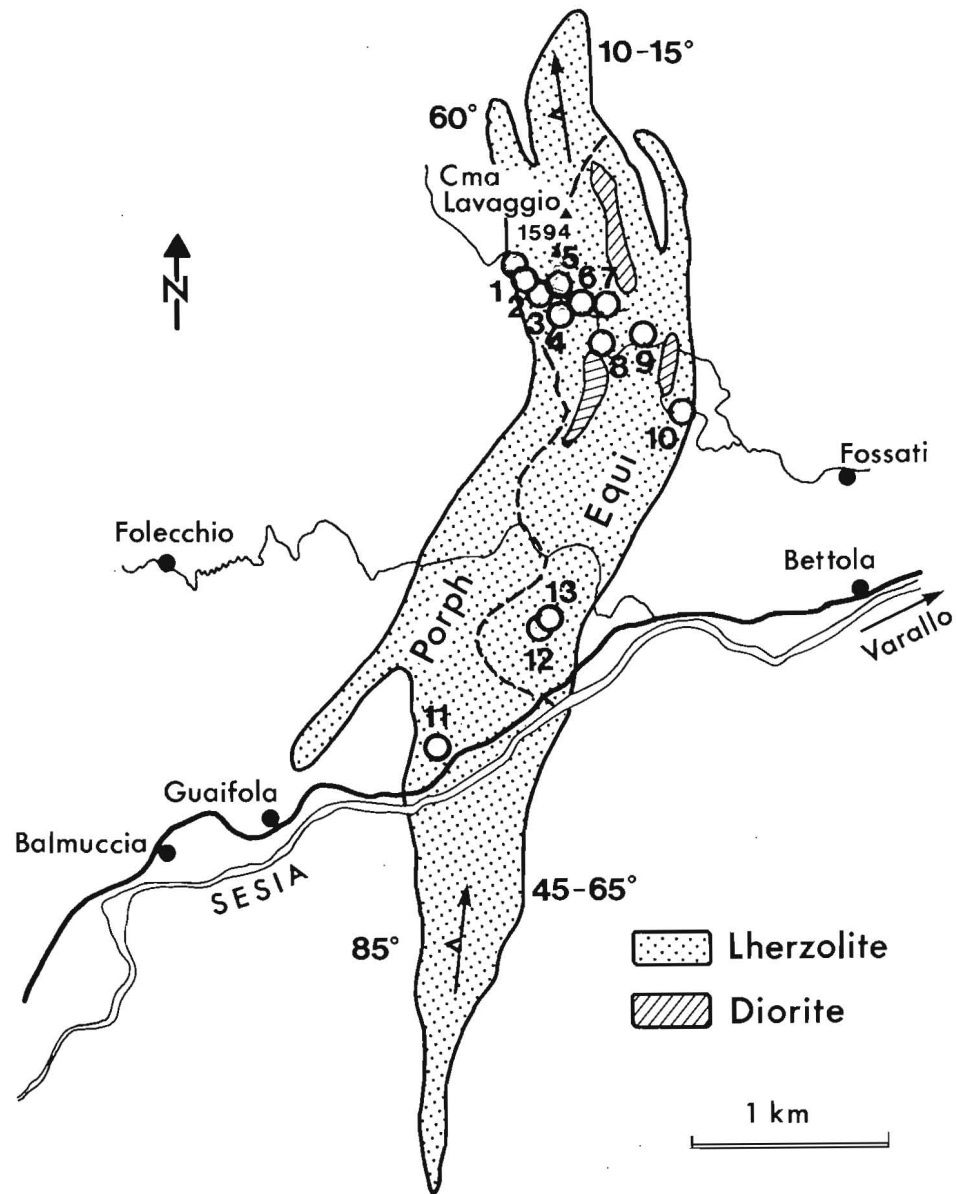


Fig. 26: Map of the Balmuccia massif with the sample sites marked. The dip and strike of the foliation and the plunge of the lineation are indicated. The border between the porphyroclastic and equigranular domain is marked by the dashed line. (After Boudier et al., 1984)

VI.2 Microstructure in the olivine

The olivine shows a typical high temperature creep microstructure consisting of dynamically recrystallized grains, subgrains and free dislocations between. In the following these features investigated in thin sections and by TEM will be characterized in more detail.

VI.2.1 Grain structure

The grain structure of the olivine phase changes from a partially recrystallized structure in the western part to a fully recrystallized structure in the eastern part of the Balmuccia massif (Fig. 27). According to the terminology of Mercier and Nicolas (1975) these structures are porphyroclastic and equigranular grain structures, respectively. The border between these structural domains in the massif (Fig. 26) has been mapped by Boudier et al. (1984).

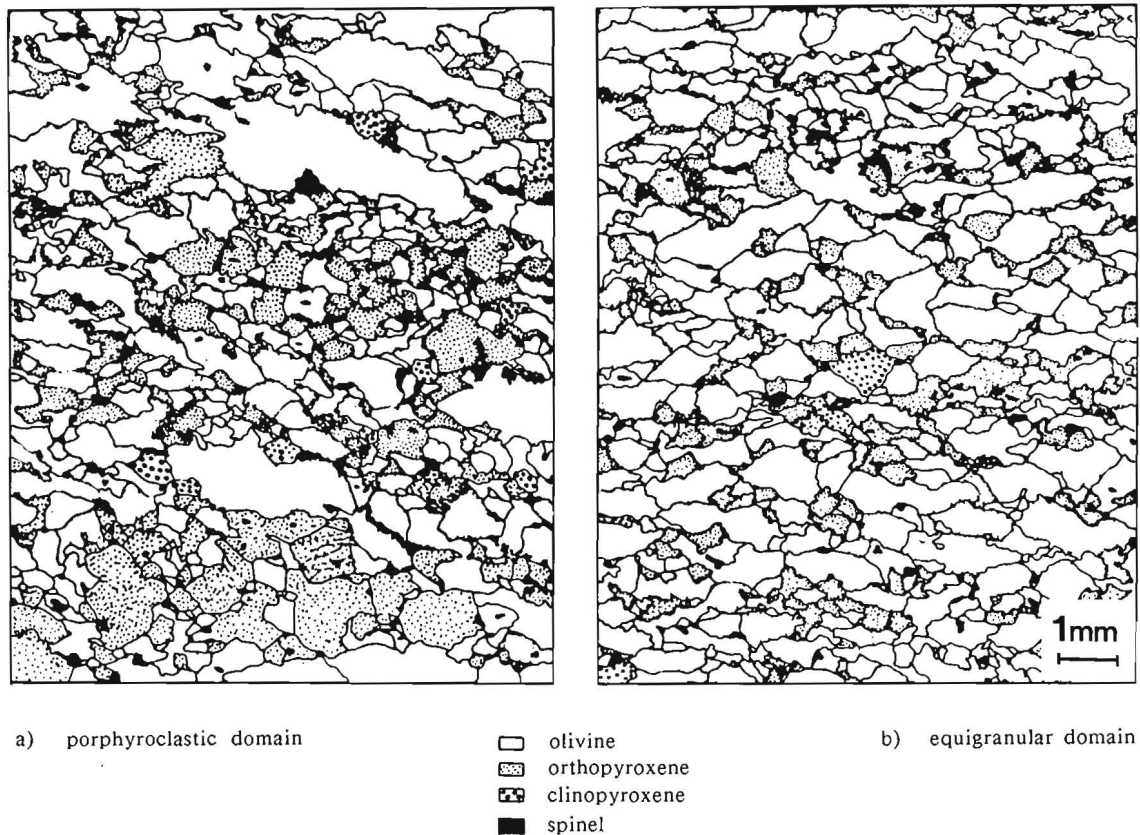


Fig. 27: Grain structure in the porphyroclastic (a) and equigranular domain (b) viewed normal to the foliation. The mineral lineation is almost horizontal.

Specimens from 13 locations within the massif have been investigated in thin sections. The results may be summarized as follows:

- The porphyroclastic domain is characterized by a bimodal grain size distribution of large elongated olivine clasts (3.5 mm x 1.8 mm) surrounded by smaller elongated grains (1.3 mm x 0.6 mm; $D_1 \approx 0.9$ mm represents the mean diameter of spherical grains the area of which equals that of the elongated grains). Locally bands of fine recrystallized grains ($D_2 \approx 30 \mu\text{m}$) are observed along the grain boundaries of the olivine matrix (Fig. 28).
- In the equigranular domain the grains are somewhat larger and also elongated (1.8 mm x 0.9 mm; $D_1 \approx 1.3$ mm)

The standard deviation of the long and short axes is about 50%, that of the axial ratio about 30%. The mean grain sizes measured on thin sections are given without stereological corrections.

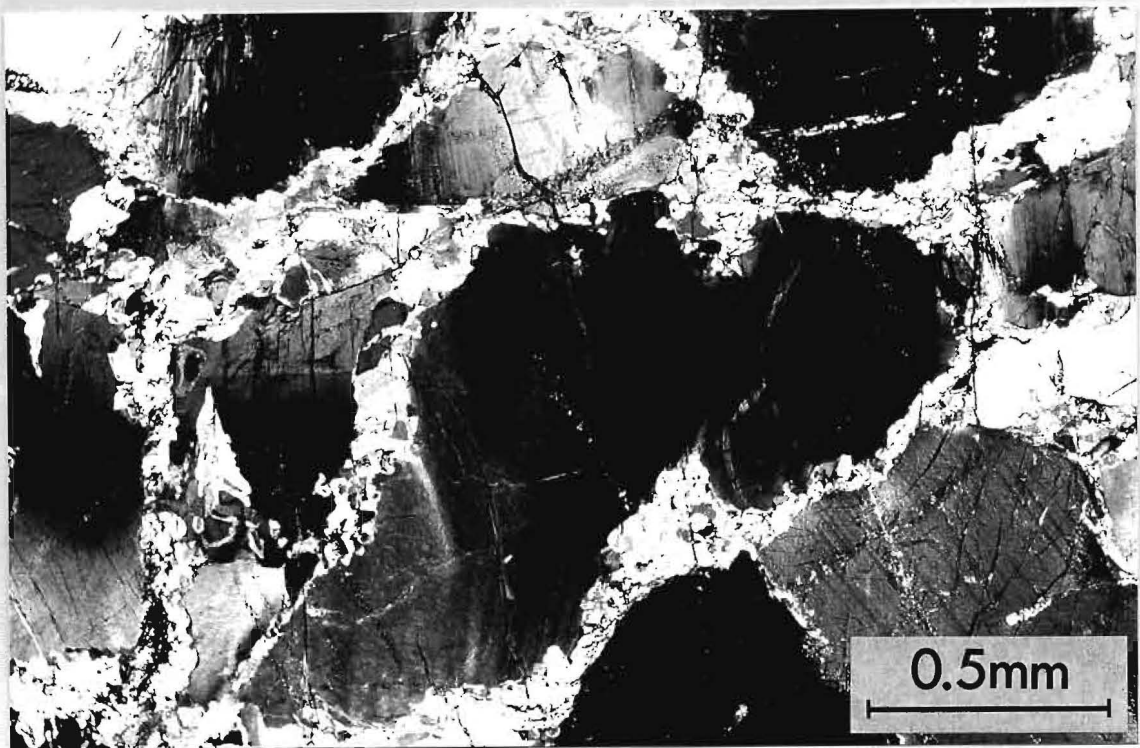


Fig. 28: Bands of fine recrystallized grains along grain boundaries of olivine matrix grains in the porphyroclastic domain. Optical micrograph, crossed polarized light.

VI.2.2 Subgrain structure

The subgrain and dislocation structure has been studied in detail on specimens 11 and 13 (Fig. 26) taken as representative of the porphyroclastic and equigranular domain, respectively. The most common low angle boundaries found on decorated thin sections are (100) tilt boundaries (Fig. 29).

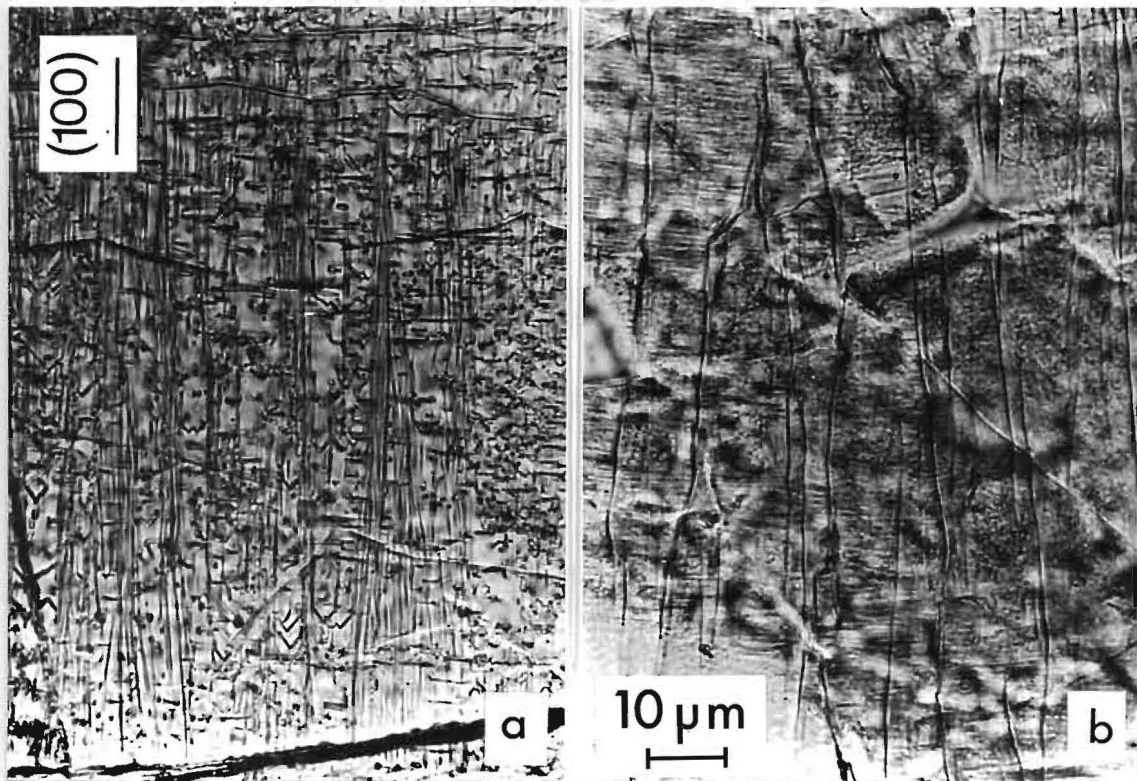


Fig. 29: Transmission optical micrograph of the decorated microstructure in the porphyroclastic (a) and equigranular domain (b). The spacing of the subgrain boundaries is larger in the equigranular domain. The decoration is produced by oxidation-induced precipitates according to the technique of Kohlstedt et al. (1976b).

In the equigranular domain (001) tilt boundaries also have been observed frequently, producing rectangular-parallelepiped subgrains (Fig. 30).

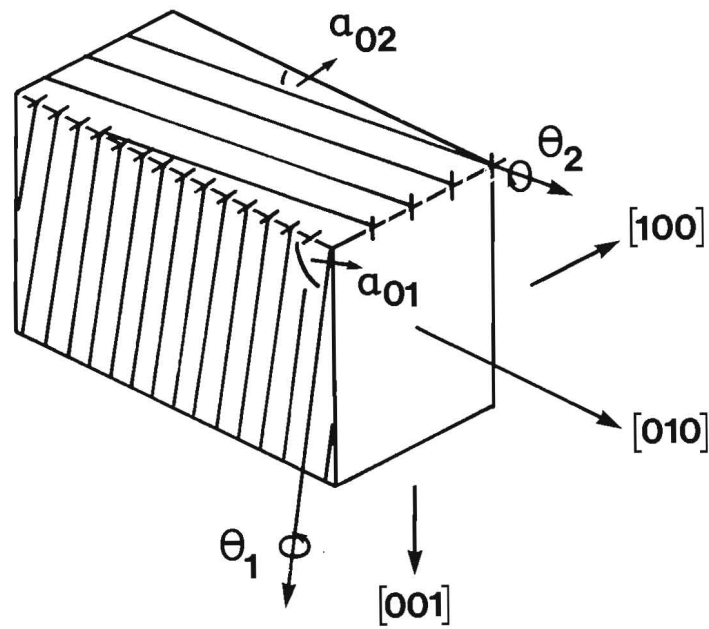


Fig. 30: Schematic sketch of the parallelepiped subgrain structure observed. The tilt angles Θ_i and the angles α_{oi} between tilt axes and $[010]$ characterizing the tilt boundaries are defined.

Both types of boundaries consist of edge dislocations with $[100]$ and $[001]$ Burgers vectors, respectively. The edge dislocations in the boundaries are almost evenly spaced and aligned along certain directions. Spacing and line direction change from one boundary to another (Fig. 31) thus changing the tilt angle and the tilt axis. Tilt angles and directions of the tilt axis measured are given in Fig. 32. The tilt angles have been calculated using the equation

$$\Theta = 2 \arcsin \frac{b}{2d_0} \approx \frac{b}{d_0} \quad (\text{for small } \Theta) \quad (6)$$

with b magnitude of the Burgers vector ($b_{[100]} = 4.77 \text{ \AA}$, $b_{[001]} = 5.99 \text{ \AA}$) and d_0 spacing of the edge dislocations:

$$d_0 = \frac{d \cos \alpha_0}{\cos \alpha \cos \beta} \quad (7)$$

where d is the projected dislocation spacing in the TEM and α is the angle between the projected dislocation line direction and $[010]$.

$$\alpha_0 = \arctan \frac{\tan \alpha}{\sin \beta} \quad (8)$$

is the angle between the tilt axis and $[010]$.

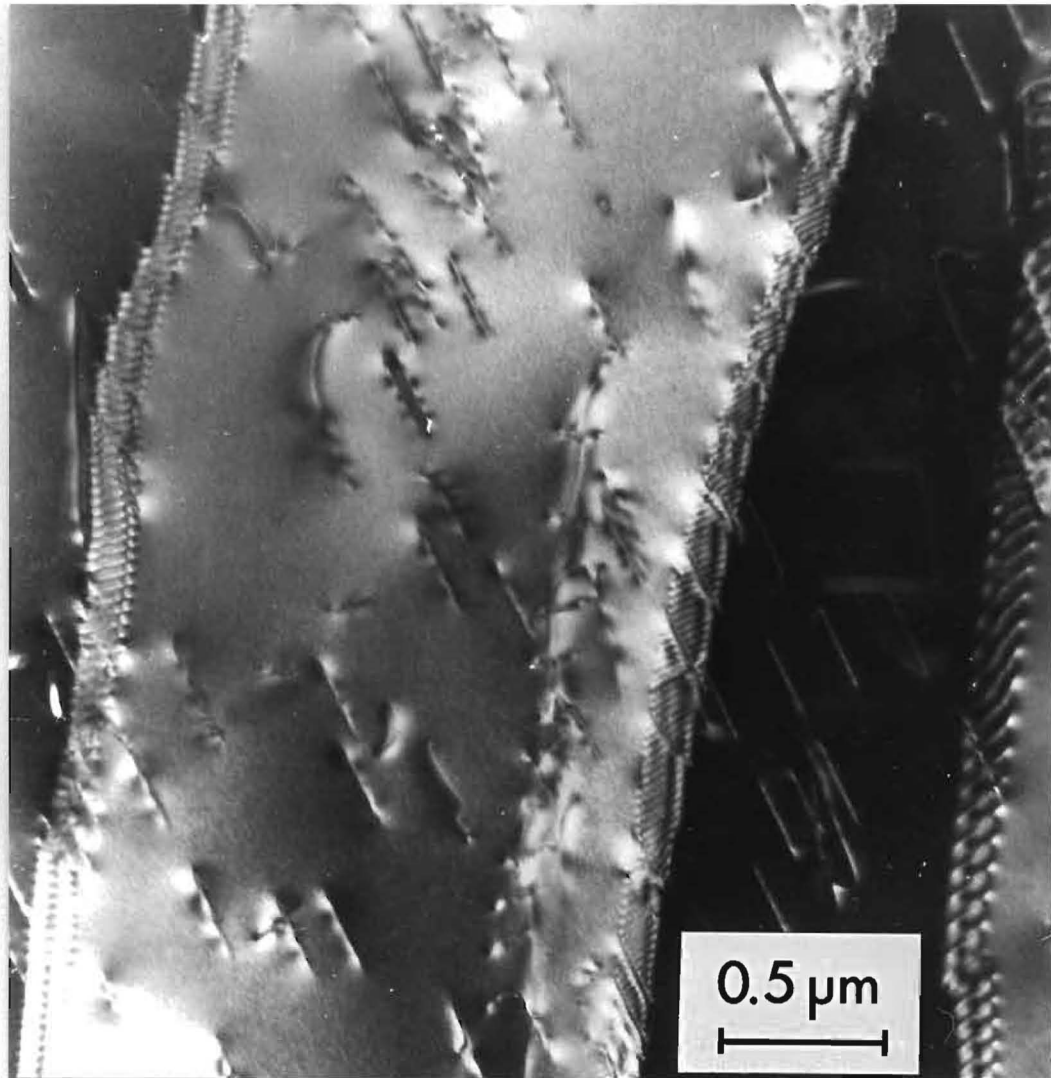


Fig. 31: Bright-field TEM micrograph of faceted $(100) [0kl]$ tilt boundaries in the porphyroclastic domain taken with $g = (131)$.

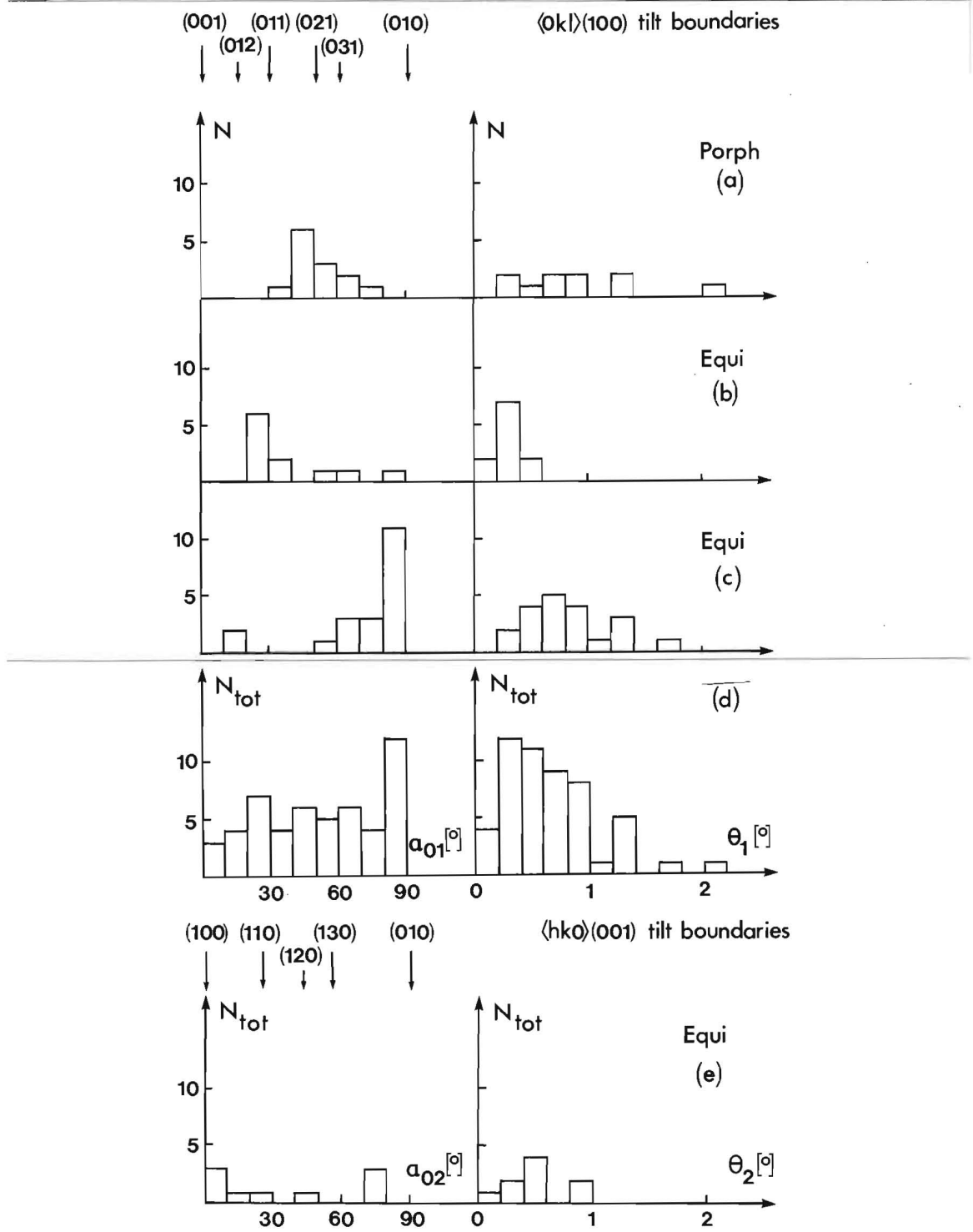


Fig. 32: Histograms of α_{oi} and Θ_i for $[0kl](100)$ and $[hk0](001)$ tilt boundaries found in the porphyroclastic and equigranular domain, respectively. Histograms (a), (b) and (c) differentiate between different grains, (d) and (e) give the total numbers. Arrows indicate possible low index slip planes given by the edge dislocation line direction and the Burgers vector.

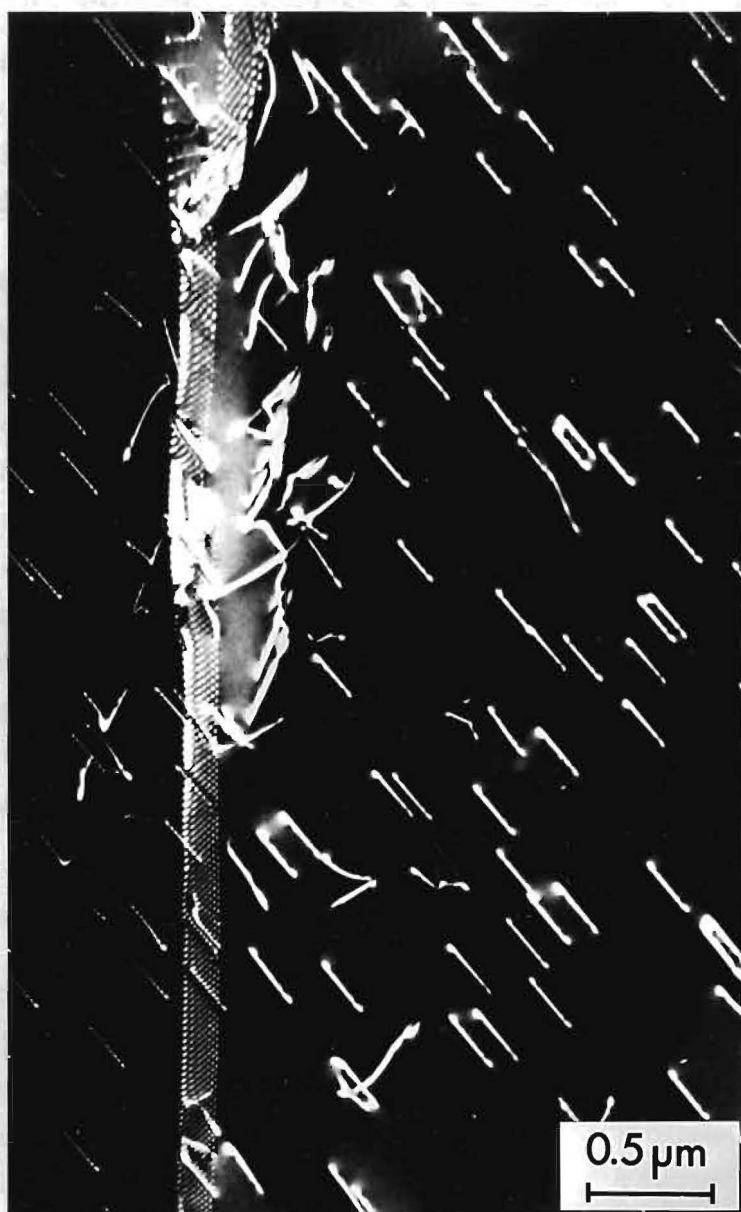


Fig. 33: Dissociation of a (100) tilt boundary. Weak beam dark-field image taken with $g = (131)$.

According to the histograms of Fig. 32 the tilt axis directions vary between 0 and 90° with respect to [010] with some maxima existing at certain low index directions. These directions and the Burgers vector suggest certain low index planes which might have been the preferred slip planes. For the [100] Burgers vector (011), (021), and mainly (010) are observed, or in general $\{0kl\}$, while for the [001] Burgers vector they are $\{hk0\}$. Apparently, the plane which dominates depends on the grain orientation with respect to the maximum shear stress (Figs. 32a to c). The tilt angles range up to 2°, with the maximum of the Poisson distribution lying at about 0.3°.

Some of the tilt boundaries either dissociate into two others or coalesce to form a new boundary with the tilt angles related by $\Theta_1 = \Theta_2 + \Theta_3$ (Fig. 33). Occasionally tilt boundaries on planes other than (100) and (001) have been observed, e.g. (120), ($\bar{1}\bar{2}0$) and (110). Twist boundaries on (010), consisting of [100] and [001] type screw dislocations which are often found in xenoliths (Buiskool Toxopeus and Boland, 1976), have not been detected.

The spacings of the $\langle 0kl \rangle$ (100) tilt boundaries are listed in Tabs. 4 and 5. The scatter of the values measured optically and by TEM is about 50%. There exists a discrepancy between the values measured on decorated thin sections and on TEM samples. Values measured on decorated thin sections are generally up to a factor 5 larger than those measured by TEM. The difference may be due to the limited resolution of the decoration technique in resolving narrow spaced subgrain boundaries, in particular if the boundaries cannot get into the edge-on orientation, and/or to the inhomogeneity of the subgrain sizes which is difficult to record on the TEM scale. Similar observations have been made by Mercier et al., 1977. In accordance with these authors it is believed that the spacings measured on decorated samples give a more reliable average than the conventional (100kV) TEM estimates because these are biased towards regions where the subgrain spacing is exceptionally small.

VI.2.3 Free dislocations

The free dislocations observed have Burgers vectors of the type [100] and [001], the density of [001] dislocations being about ten times higher than that of [100]. The total dislocation density is given in Tab. 4; it has been determined by counting the number of dislocation intersections per micrograph. The standard deviation of the mean density measured within several grains is about 20%. The dislocations are homogeneously distributed. Dislocation dipoles and glide loops are common (Fig. 34). While small loops ($\varnothing \leq 1 \mu\text{m}$) have a round shape, larger loops are polygonal with their sides aligned along certain low index directions. Flat cuts of such loops with [100] and [001] Burgers vectors lying on different planes are schematically sketched in Fig. 35A, and actual photographs are shown in Fig. 35B. On (010) the loops are rectangular with sides in edge and screw orientations, while on (001) and (100) mixed segments also exist. On (010) there is a significant difference between these dislocation types. For [100] dislocations the screw parts are more mobile than the edge parts, whereas the opposite is observed for [001] dislocations. As a result [100] dislocations are found predominantly in an edge orientation, whereas [001] dislocations are found in a screw orientation.

The loops observed prove that the active slip systems are $(010)[100]$, $(001)[100]$, $(010)[001]$ and $(100)[001]$. Dislocation multiplication occurs by the classical Frank-Read mechanism (d in Fig. 35B) and by helix formation (Fig. 34) resulting from double cross slip of screw dislocation segments.

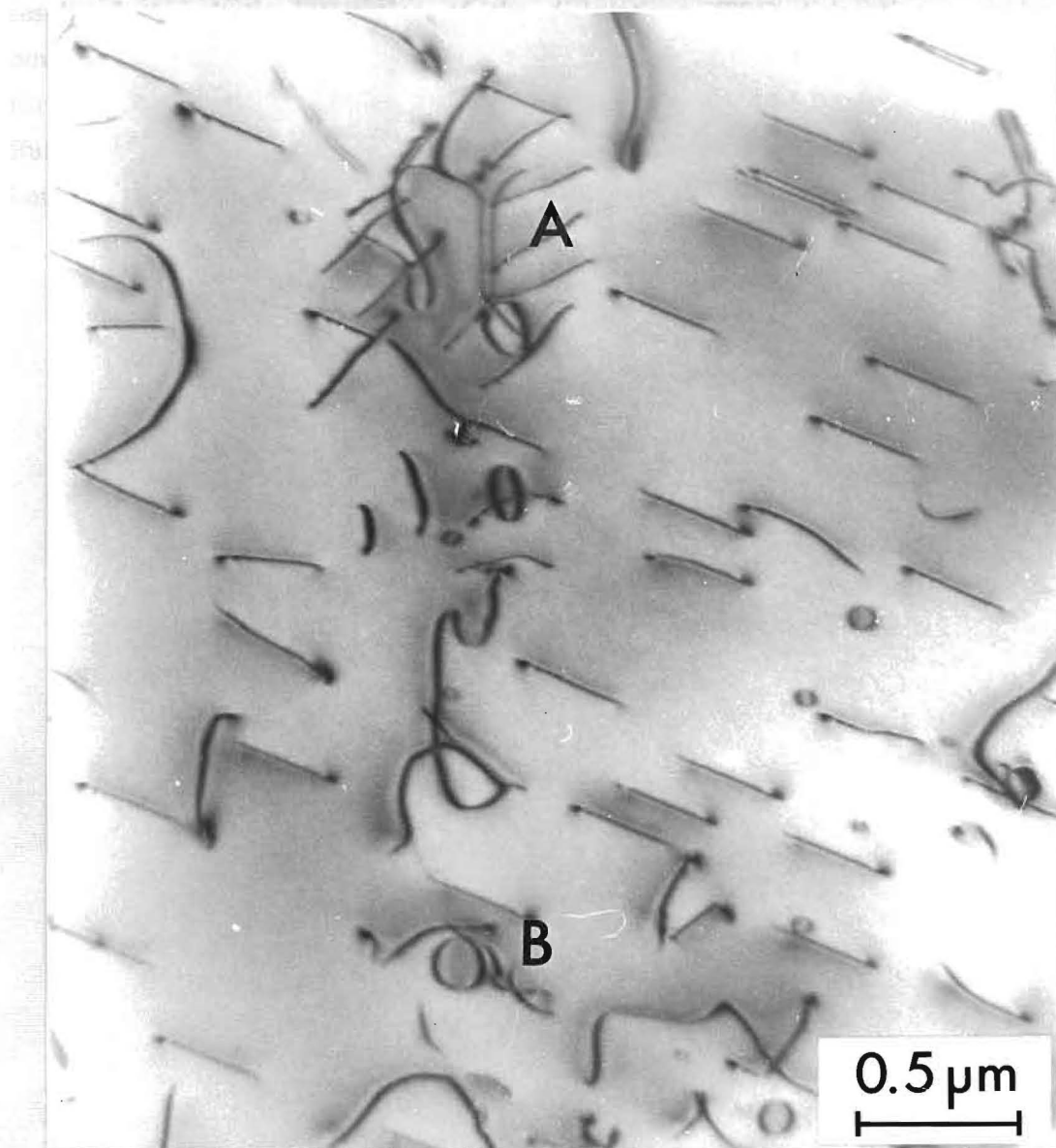
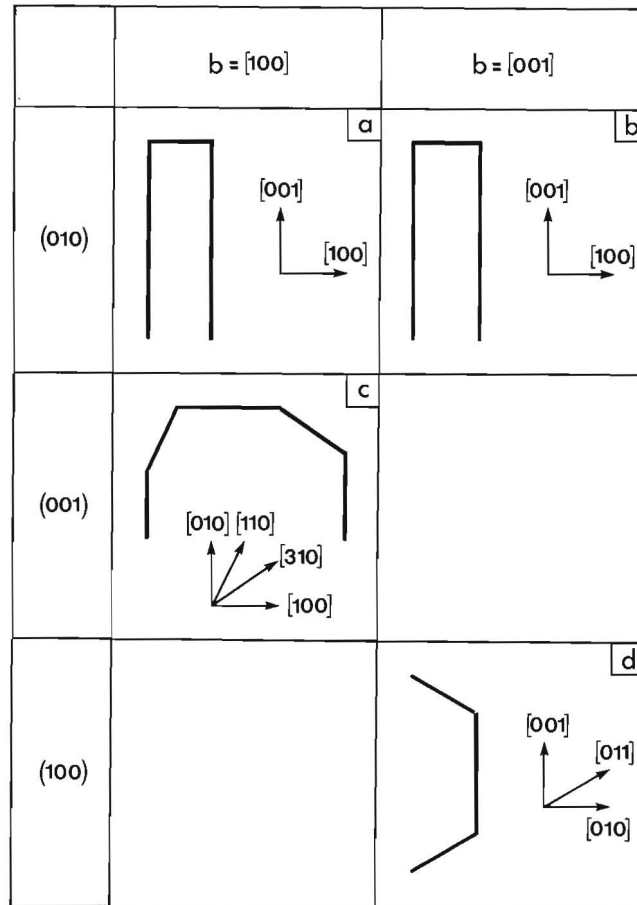
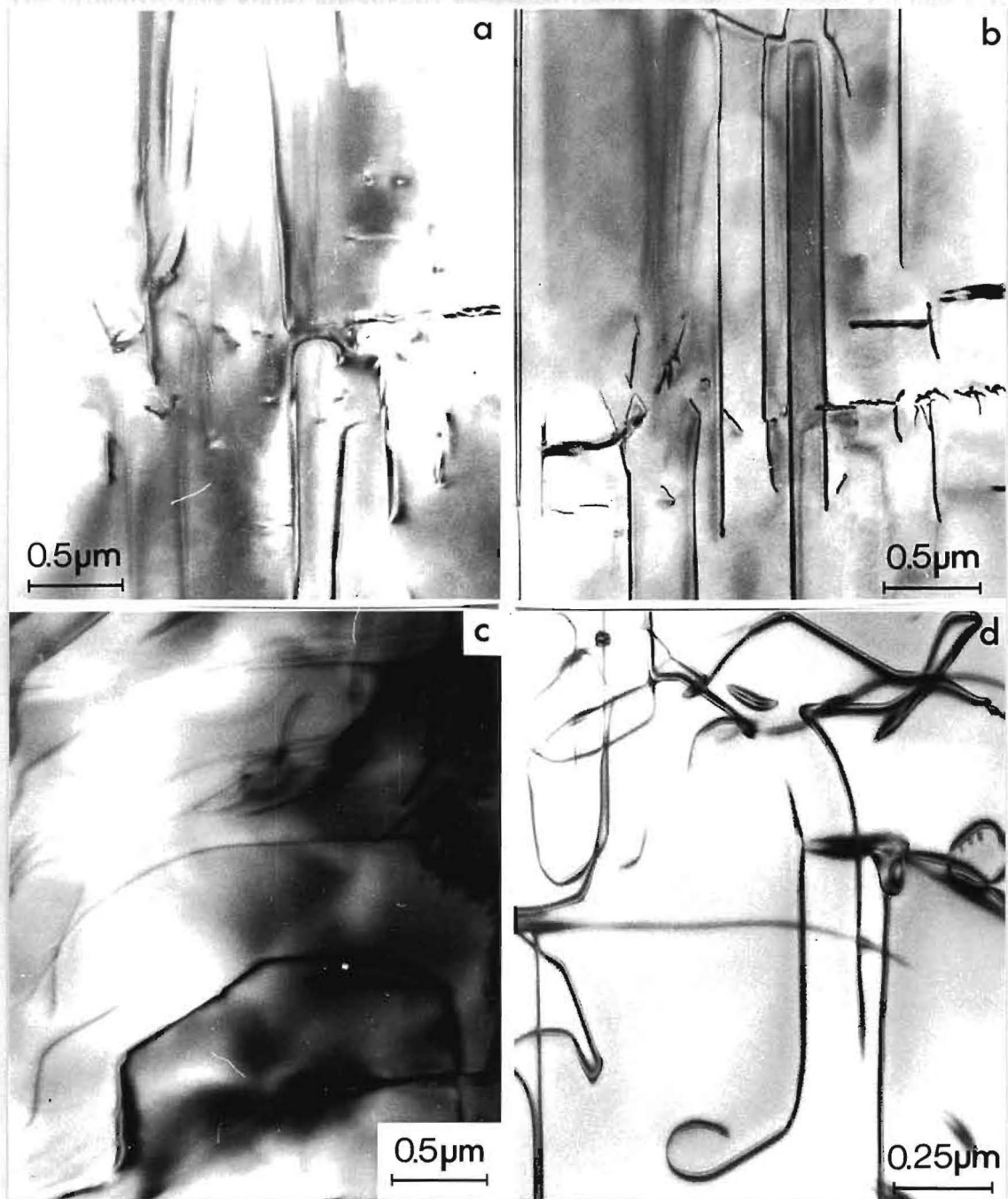


Fig. 34: Free dislocation arrangement: Dislocation dipole (A), dislocation loop (B). Straight dislocations have a $[001]$ Burgers vector, others have $[100]$. Bright-field image taken with $g = (131)$.

Dislocations may be trapped by the grain boundaries (Fig. 33). This process leads to a change of the misorientation and to faceting in the case of different Burgers vectors (Fig. 31). The absorption of free dislocations into a subgrain boundary is clearly seen in flat-on (100) tilt boundaries. [100] dislocations entering the boundary force the other grain boundary dislocations to climb into new equilibrium positions. Crossing of like-sign dislocations produces kinking at the region of closest approach. Such kinking is the result of the elastic interaction of the dislocations. [001] dislocations entering the boundary are found to produce ledges. The [001] and [100] dislocations may interact to form narrow spaced edge dislocation arrangements in the boundary with [101] pseudo-Burgers vector as has been resolved with the weak-beam method in (101) tilt boundaries by Gueguen and Darot (1982b).





B

Fig. 35: (A) Schematic sketch of dislocation glide loops with Burgers vectors $[100]$ and $[001]$ observed on different slip planes. (B) Corresponding dislocation glide loops observed. Bright-field images: (a) and (c) $g = (400)$, (b) and (d) $g = (004)$

VI.3 Microstructure in the orthopyroxene

The orthopyroxene grains are slightly elongated (porphyroclastic domain: 1.6 mm x 1.1 mm, equigranular domain: 1.2 mm x 0.7 mm; the standard deviation is the same as for the olivines). No signs of recrystallization are observed within this mineral phase. Within the grains the microstructure displays three basic features: perfect dislocations (non-dissociated within the resolution of bright-field TEM), stacking faults and diopside exsolution lamellae. Fig. 36 provides an overview of these defects, details will be discussed below.

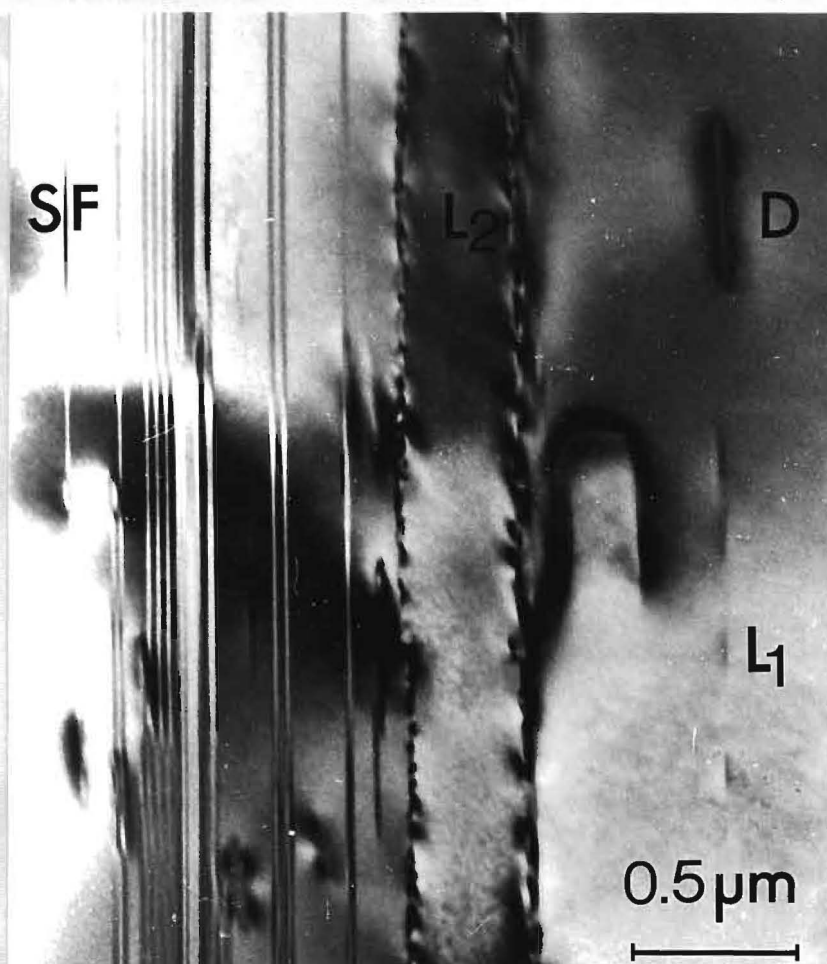


Fig. 36: Microstructure of orthopyroxenes consisting of isolated dislocations (D), stacking faults (SF) and diopside exsolution lamellae (coherent L_1 , semicoherent L_2) viewed along [010]. Bright-field image taken with $g = (202)$.

VI.3.1 Dislocations and stacking faults

The perfect dislocations observed mainly lie in the (100) plane; their line directions vary. The Burgers vector has been analysed to be [001]. Thus these dislocations belong to the (100)[001] slip system. Occasionally they are arranged into low angle tilt boundaries with (001) boundary plane and [010] tilt axis. Tilt angles are on the order of magnitude of 1° . In a few cases edge dislocations on (100) with [010] Burgers vector and dislocation loops on (010) with [001] Burgers vector (Fig. 36) have been observed. The resulting slip systems are (100)[010] and (010)[001], respectively.

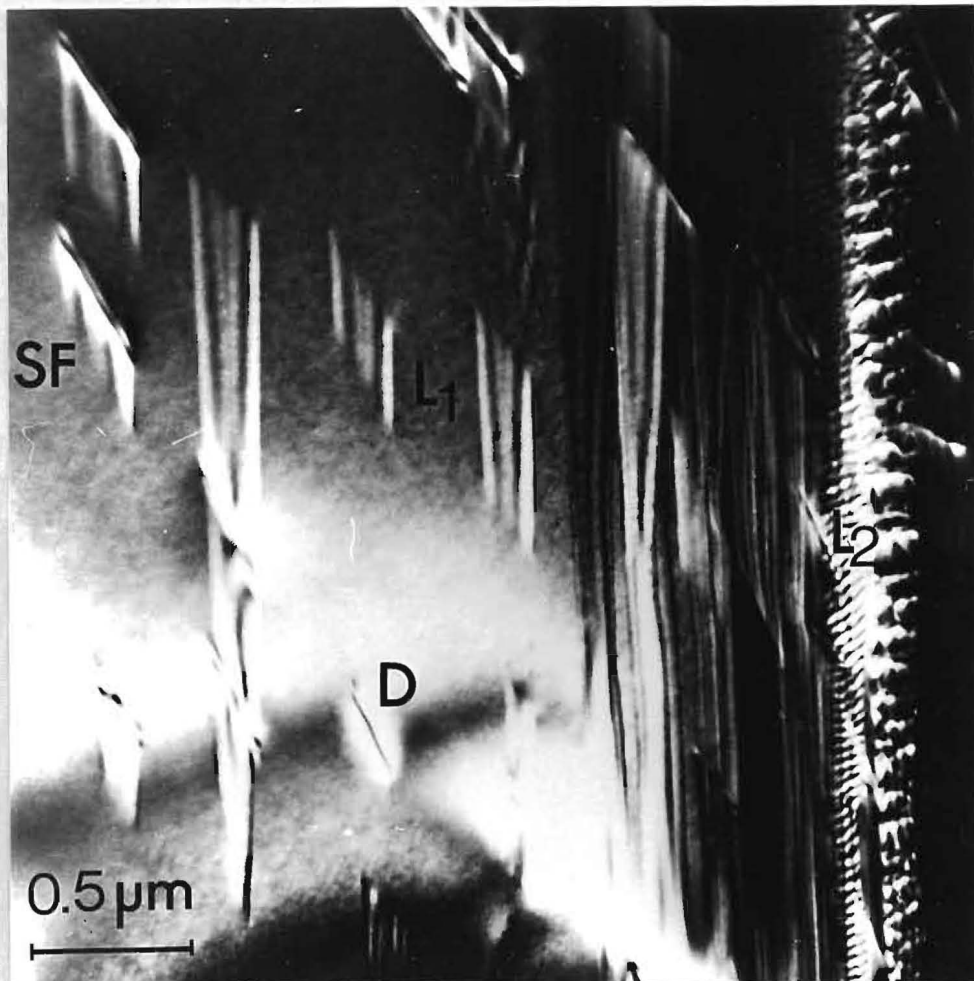


Fig. 37: Dark-field micrograph taken with $g = (202)$ showing the dislocations (D), stacking faults (SF) and diopside exsolution lamellae (coherent L_1 , semicoherent L_2) in an inclined position.

The stacking faults observed lie in the (100) plane. Their displacement vector $\mathbf{R}$ has been proven to be parallel to [001]. They are bounded by partial dislocations (Fig. 37); the Burgers vector $\mathbf{b}_p$ of the strong partial dislocation is parallel to [001]. The stacking fault width varies from one fault to the next; lower and upper bounds cannot be given. The spacing between the faults in [100] direction is about $0.1 \mu\text{m}$; it decreases close to the semicoherent exsolution lamellae. Based on a detailed analysis of stacking faults in experimentally deformed orthoenstatite Coe and Kirby (1975) concluded $\mathbf{R} = 5/6 [001]$ and the partial dislocations to be formed by the dissociation $[001] \rightarrow 5/6 [001] + 1/6 [001]$, the latter partial being practically invisible. This result is in agreement with theoretical predictions of the ortho-clinoenstatite phase transformation (Coe, 1970). The exsolution of Ca-clinopyroxene from orthopyroxene has been analysed by Kirby and Etheridge (1981). They assume that the exsolution involves nucleation and growth by shear transformation combined with cation exchange. Their analysis supports the shear transformation mentioned above. Former suggestions by Kohlstedt and Vander Sande (1973, $\mathbf{R} = 1/4 [001]$ and $\mathbf{b}_p = 1/4 [001]$), Boland (1974, $\mathbf{b}_p = 1/4 [001]$) and McLaren and Etheridge (1976, $\mathbf{R} = 1/6 [001]$, $\mathbf{b}_{p1} = 7/6 [001]$ and $\mathbf{b}_{p2} = -1/6 [001]$) based on ambiguous analyses of naturally deformed orthopyroxenes therefore do not seem to hold any longer.

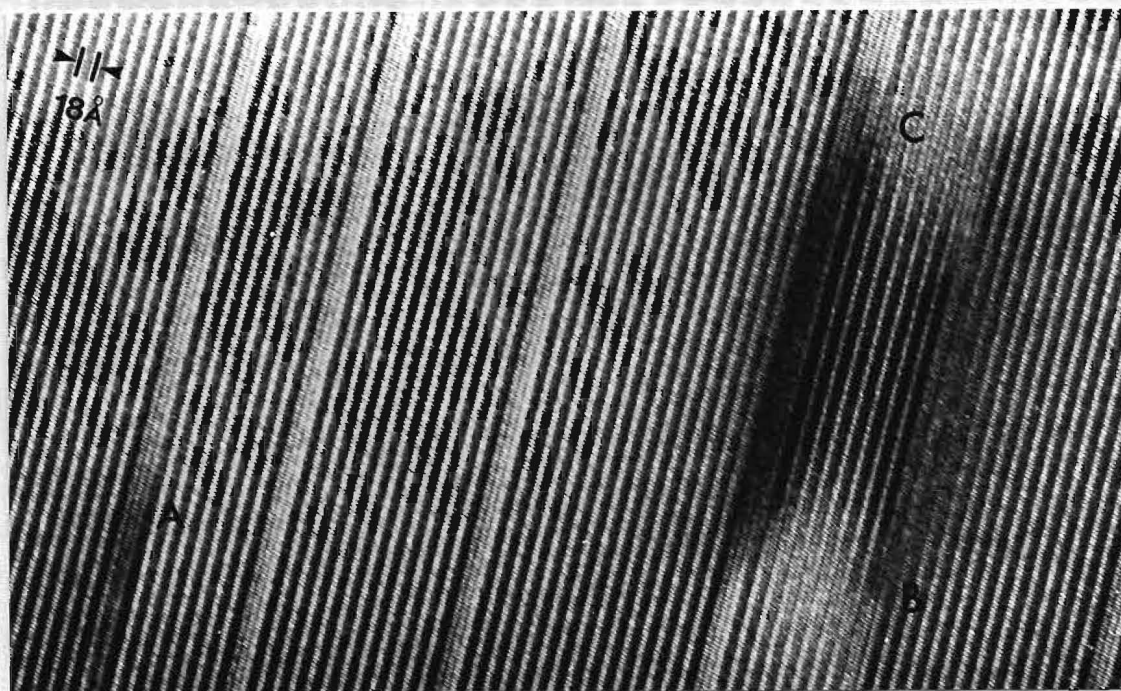


Fig. 38: Lattice fringe image of orthoenstatite interspersed with clinoenstatite. Ending lamellae are marked at A and B, a growing lamella at C. The lattice fringes give the periodicity of the (100) planes in the orthopyroxene phase.

To study the stacking faults in more detail HRTEM has been used. Figs. 38 and 39 show lattice fringe images of clinoenstatite lamellae. The fringes give the periodicity of the (100) planes in ortho- ($d_{(100)} = 18 \text{ \AA}$) and clinoenstatite ($d_{(100)} = 9 \text{ \AA}$), respectively. The width of the clinoenstatite lamellae is an even multiple of $9 \text{ \AA}$ in accordance with observations of Müller (1974). Broadening of the lamellae occurs by motion of dislocations in the interface (point C in Fig. 38).

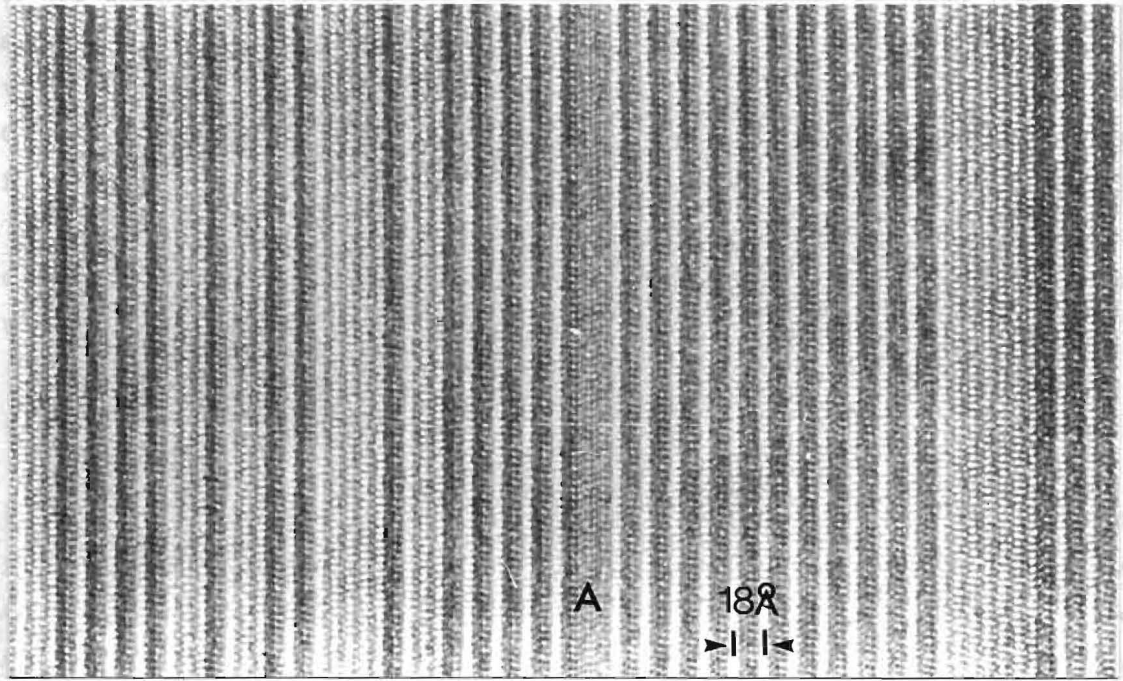


Fig. 39: Lattice fringe image of orthoenstatite interspersed with clinoenstatite and coherent diopside (A) lamellae.

VI.3.2 Diopside exsolution lamellae

Diopside exsolution lamellae parallel to (100) planes have been observed in two different variations: thin coherent lamellae exhibiting stacking fault like contrast and broad semicoherent lamellae approximately $0.1\ \mu\text{m}$ wide. The coherent exsolution lamellae could be distinguished from the stacking faults because they did not show bounding partial dislocations under all diffraction conditions used. A HRTEM image of such a lamellae ($36\ \text{\AA}$ wide) is shown by A in Fig. 39. The (100) interphase boundaries of the semicoherent lamellae are composed of parallel rows of misfit dislocations spaced about $500\ \text{\AA}$ apart. HRTEM shows that the misfit dislocations are associated with ledges in the interphase boundary. Similar observations have been reported by Champness and Lorimer (1973), Kohlstedt and Vander Sande (1973), Vander Sande and Kohlstedt (1974), Smith et al. (1982), van Duysen et al. (1985) and Nazé et al. (1987). These works show that the misfit dislocations have [001] Burgers vector and are dissociated into four partials. Depending on the thickness of the lamellae in addition undissociated [010] dislocations are also found in the interphase boundary.

VI.4 Texture

Pole figures for different crystal axes of olivines (excluding the very fine grains of the porphyroclastic domain) and orthopyroxenes have been measured with an universal optical stage on specimens of locations 5, 11, 12 and 13 in Fig. 26 (Wedel, 1985, Wedel et al., 1986). Typical diagrams of the equigranular domain are presented in Fig 40. The characteristics of the textures can be summarized as follows:

- The [100], [010] and [001] axes of olivine form symmetrical maxima with the (010) plane parallel to the foliation and [100] parallel to the mineral lineation. The corresponding [001] maximum lies within the foliation and perpendicular to [100].
- Pole figures of orthopyroxene show orientations of [001] axes perpendicular to the lineation parallel to the [001] orientation of olivine. The [010] and [100] axes form girdles perpendicular to [001].
- The rocks of the equigranular domain have a stronger preferred orientation than those of the porphyroclastic one.

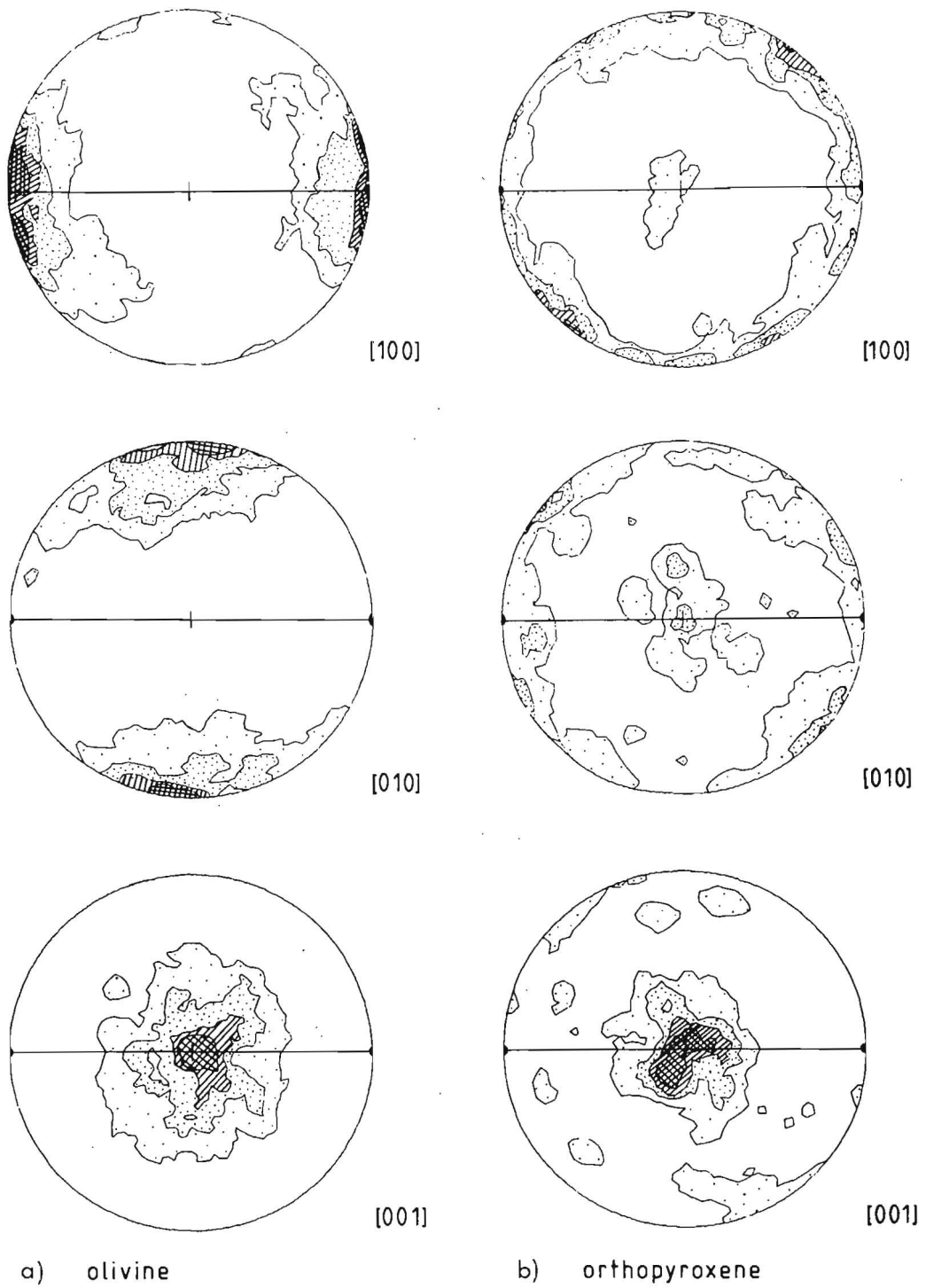


Fig. 40: Texture of olivine (a) and orthopyroxene (b) in the equigranular domain; $n = 200$, isolines: 1%, 3%, 5%, 7% per 1% of total area, lower hemisphere projection, foliation: horizontal line, lineation: black dots.

VI.5 Discussion

VI.5.1 Slip systems

It is interesting to compare the slip systems observed in naturally deformed olivine and orthopyroxene with that deformed in the laboratory. For olivine in section VI.5.2 the comparison of predominant slip systems will be used to give a crude estimate of the deformation temperature.

Olivine

In experimentally deformed single crystals, the main slip systems observed are for $[110]_c$ compression direction: $(010)[100]$, $[011]_c$: $(010)[001]$ and $[101]_c$: $(001)[100]$ and $(100)[001]$. (The $[uvw]_c$ direction refers the compression axis to cubic axes superimposed on the orthorhombic crystallographic axes). These have been found by analyses of changes in the shape of olivine single crystals (Durham and Götze, 1977) and by TEM-analyses of olivine (Phakey et al., 1972) and forsterite single crystals (Gueguen and Darot, 1982a; 1982b). Based on optical microscopy observations of deformation bands and decorated dislocations the predominant slip systems identified in deformed natural polycrystalline olivine are in the order of increasing temperature (Fig. 22): $(100)[001]$, $(110)[001]$, $\{0kl\}[100]$ and $(010)[100]$ (Carter and Avé Lallemant, 1970). The most comprehensive TEM study on the dislocation microstructure in deformed synthetic olivine polycrystals, by Zeuch and Green (1984), showed that both types of Burgers vectors are active at all temperatures investigated.

All slip systems identified in the naturally deformed olivines investigated are identical to those found in experimentally deformed olivine at elevated temperatures. The dislocation microstructure consists of both types of Burgers vectors, $[100]$ and $[001]$. Dislocations with $[100]$ Burgers vector are mainly arranged in low angle tilt boundaries with varying tilt axis. For $[001]$ dislocations subgrain boundary formation is found only in the equigranular domain and even there is not so common. Assuming that the dislocations in the grain boundary have moved on the plane given by the cross product of the Burgers vector and their line direction then the resulting slip systems are $\{0kl\}[100]$ and $\{hk0\}[001]$, respectively. Except for $(010)[100]$ there is only a weak preference of other low index planes. However, as climb is necessary to arrange the dislocations with an even spacing in the subgrain boundary, the line directions of the dislocations may have changed by climb. An analysis of glide loops proves $(010)[100]$, $(001)[100]$, $(010)[001]$ and $(100)[001]$ as activated slip systems.

The straight dislocation segments observed lying in certain directions may be due to deep Peierls valleys in these directions. On (010) planes dislocations with $[100]$ Burgers vector are mainly found in edge orientations those with $[001]$ in screw orientations.

This results from different mobilities of edge and screw dislocations: $[100]$ screws are faster than $[100]$ edges, whereas the opposite holds for $[001]$ dislocations. Similar observations have been made in forsterite and olivine deformed experimentally in $[110]_c$ and $[011]_c$ orientations, respectively, (Darot and Gueguen, 1981; Durham et al., 1977). The rate controlling creep mechanism suggested in the first case is climb by $[100]$ edge dislocations and in the second case cross slip by $[001]$ screw dislocations, the latter process being more difficult (Gueguen and Darot, 1982b). The difference may be due to dissociation of $[001]$ dislocations. However, so far no convincing proof exists in the literature on dislocation dissociation in olivine (Vander Sande and Kohlstedt, 1976; Gueguen and Darot, 1982a). For polycrystalline olivine it has been shown that the creep rate coincides with that which is determined by the creep mechanism of the hardest slip system, that is cross slip of screw dislocations of the $(010)[001]$ system (Kohlstedt and Ricoult, 1984; Toriumi et al., 1984). In analogy the same may be assumed for the peridotite rocks of Balmuccia.

There are differences in the microstructure between the porphyroclastic and the equigranular domain. In the equigranular domain (100) tilt boundaries are wider spaced and $[001]$ dislocations are partly arranged in low angle tilt boundaries. Annealing experiments of Ricoult (1979) on natural dunite showed that with increasing annealing time (001) tilt boundaries are more numerous. These results point to a higher (syn- or post-tectonic) recovery in this domain. According to Boudier et al. (1984) fluid inclusions, which they found more abundant in the eastern peridotites of the massif, could be responsible for the recovered character.

Orthopyroxene

The slip systems which are observed in experimentally deformed orthopyroxene are $(100)[001]$ (Coe and Kirby, 1975), $(100)[010]$ and $(010)[001]$ (Nazé et al., 1987). Depending on stress, temperature and strain rate, slip on the $(100)[001]$ system leads to the transformation of ortho- into clinoenstatite. Studies of the inversion by TEM (Kirby, 1976) show clearly the role of partial dislocations in the transformation.

Investigations of naturally deformed orthopyroxenes reveal the same defect structures (Kohlstedt and Vander Sande, 1973; Nazé et al., 1987). The results on the microstructure in orthopyroxenes of the Balmuccia massif are in full agreement with the observations described above.

VI.5.2 Stress, strain and temperature estimates

Stress

The deviatoric stresses (Tab. 4) leading to the microstructure observed have been estimated using the experimentally determined paleopiezometers presented in Figs. 18 to 20. From the stress levels two deformation events may be inferred: the larger recrystallized grain size D_1 is caused by a stress in the order of 10 MPa, and the fine recrystallized grains D_2 along the grain boundaries in the porphyroclastic domain as well as the subgrain and dislocation microstructure is caused by a stress an order of magnitude higher ($\sigma \approx 300$ MPa). Because of the scatter of the data as well as of the deviations of the piezometers from each other the stress levels estimated are reliable only within a factor of 2. However, the general conclusions made about the deformation history of the massif (VI.5.4) do not depend on the accurate absolute value but on the large stress difference.

Tab. 4: Dynamically recrystallized grain sizes $D_{1,2}$, subgrain sizes d (measured optically on decorated thin sections) and dislocation densities ρ measured for olivine. Paleostresses given in brackets have been estimated by using equations $\sigma_D = 1990 D^{-3/4}$, $\sigma_d = 3200 d^{-1}$, $\sigma_\rho = 9.6 \cdot 10^{-3} \rho^{1/2}$, with σ in MPa, D and d in μm , and ρ in cm^{-2} .

	D_1 [mm]	D_2 [μm]	d [μm]	ρ [$10^8/\text{cm}^2$]
Porphyroclastic domain	0.9 (12)	30 (160)	7 (460)	7 (250)
Equigranular domain	1.3 (9)	— —	12 (270)	8 (270)

Strain

In both domains the olivine grains are elongated with an aspect ratio of about 2:1. Assuming this elongation to be due to intracrystalline slip during deformation event II the finite strain estimated with eq. (5) is $\epsilon_{\text{II}}(\text{olivine}) = 35\%$. It is not known if part of this elongation also results from preferential grain growth during dynamic recrystallization of deformation event I. However, as in the porphyroclastic domain the

olivines show a fine-recrystallized rim along the matrix grains, which according to the stress estimates has been produced during the second deformation event, a strain of the order estimated seems to be reasonable.

According to measurements of Coe and Kirby (1975) the transformation of ortho- into clinoenstatite involves a shear of $\delta = 13.3^\circ$ in the (100) plane along the [001] direction. Taking a mean thickness $y = 18\text{\AA}$ and a separation of the clinoenstatite lamellae $L = 0.1\text{ }\mu\text{m}$ and assuming they are extended across the grain then the shear strain of the orthoenstatite phase calculated with eq. (4) is $\epsilon_{\text{II}}(\text{orthopyroxene}) \approx \gamma/2 = 0.2\%$. Due to the large variation in the stacking fault width as well as to the uncertainty in the nature of the stacking faults this value gives only an estimate of the order of magnitude. The strain estimated is rather small indicating that the orthopyroxene grains behave as competent particles with a high creep resistance in a ductile olivine matrix. The elongation of the orthopyroxene grains may be a remnant of deformation event I or even a crystallization grain shape, indicating that the orthopyroxene phase did not contribute much to strain at higher temperatures.

Temperature

On the basis of the present results temperatures during the deformation phases may be estimated in three ways:

- Assuming an average strain rate of about $\dot{\epsilon} \approx 10^{-14}\text{ sec}^{-1}$ during deformation then experimentally determined creep laws (Fig. 24) yield $T_{\text{I}} \leq 1000^\circ\text{C}$ and $T_{\text{II}} \leq 800^\circ\text{C}$ for deformation events I ($\sigma_{\text{I}} \approx 10\text{ MPa}$) and II ($\sigma_{\text{II}} \approx 300\text{ MPa}$), respectively. These temperatures decrease for increasing water content of the material.
- Maximum temperatures at which orthoenstatite transforms to clinoenstatite depend on the strain rate applied. Assuming the orthopyroxenes to be deformed during event II at the same strain rate as the olivine matrix ($\dot{\epsilon} \approx 10^{-14}\text{ sec}^{-1}$) then Fig. 23 yields an upper limit for T_{II} of about 650°C . This temperature equals that obtained for wet olivine in Fig. 24. It should be noted that the hydrostatic phase boundary in Fig. 23 proposed by Grover (1972) is in the same temperature range and therefore the clinoenstatite observed could also be due to an equilibrium phase transformation. However, as pointed out by Buseck and Iijima (1975), a static transformation should produce twins in the clinoenstatite phase, in contrast to observation.
- In the material investigated both types of Burgers vectors [100] and [001] have been observed. According to Fig. 22 for a strain rate of $\dot{\epsilon} = 10^{-14}\text{ sec}^{-1}$ a change of the Burgers vector occurs between 200 and 400°C . Therefore temperatures within this range may have existed during deformation event II.

The temperatures estimated for deformation event II cover a large temperature range. The most reliable estimate seems to be that from the ortho- to clinoenstatite transformation because of the weak dependence on the water content.

VI.5.3 Correlation between microstructure and texture

To discuss the texture development in the peridotites investigated it seems to be appropriate first of all to compare the results with those on the same massif reported by others. All olivine textures of the Balmuccia massif observed so far (Garuti, 1977; Jackson, 1979, and Boudier et al., 1984) exhibit a [010] maximum normal to the foliation plane. Within this plane Garuti (1977) found maxima of [100] and [001] normal to each other with no marked difference between the porphyroclastic and equigranular domain. The lineation is not given by Garuti. Jackson (1979) and Boudier et al. (1984) found that in both domains there is a distinct [100] maximum slightly inclined to the mineral lineation. Although the sample locations of the above-mentioned authors are not specified, the general agreement of all olivine textures may point to a textural homogeneity within this massif. Measurements of orthopyroxene textures in the Balmuccia massif are not known.

The olivine texture observed is generally explained in a simple way by predominant slip on the (010)[100] system (Hobbs et al., 1976), that is during shearing the slip plane and slip direction rotates towards the foliation and lineation, respectively. According to the analysis of dislocations and subgrain tilt boundaries this requirement does not seem to be fulfilled in deformation event II, which is characterized by {0kl}[100] and {hk0}[001] slip. The operation of (010)[100] slip is commonly observed in high temperature deformation experiments suggesting that the texture observed may have developed by intracrystalline slip during event I remaining unchanged by the low strain deformation of event II. However, this interpretation is rather speculative. Much more experimental work is needed to give a better founded interpretation. In particular, the influence of dynamic recrystallization on the texture development in olivine must be investigated. For salt, extrusion experiments have shown that the texture developed during slip is totally different from that developed during dynamic recrystallization (Skrotzki and Welch, 1983).

Assuming the elongation of the olivine grains to be due to intracrystalline slip during deformation event II the finite strain estimated is $\epsilon_{II}(\text{olivine}) \approx 35\%$. The strain estimated in orthopyroxene from the density and width of deformation induced clinoenstatite lamellae yields a value of $\epsilon_{II}(\text{orthopyroxene}) \approx 0.2\%$. This small value means that the texture in orthopyroxene cannot be the result of slip within this mineral phase during deformation event II. Also the slip plane (010) and the slip direction [001]

of the main slip system does not coincide with the foliation and lineation, as is generally expected by a shear model (Hobbs et al., 1976). It is therefore concluded that the texture observed in orthopyroxene is due to an rigid body rotation of orthopyroxene grains around [001] assumed to be the morphological long grain axis. This rotation mechanism has been observed experimentally for prolate rigid grains within a ductile matrix (Taylor, 1923). The large difference in strain between orthopyroxene and olivine supports this mechanism. However, according to the experimental results of Taylor (1923) it is questionable if the straining of the olivine matrix during event II is large enough to produce such a pronounced texture in the orthopyroxene phase.

Textures have been also measured in other peridotite massifs of the Ivrea zone in Baldissero (Nicolas and Poirier, 1976; Garuti and Friolo, 1979) and Finero (Garuti and Friolo, 1979; Kruhl and Voll, 1979; Kumpmann, 1987). For the location of these massifs see Fig. 25; Baldissero is located SW of Biella. In going from Baldissero over Balmuccia to Finero the degree of recrystallization increases a fact which may be explained by an increasing deformation, provided the deformation conditions in the massifs have been the same. Two recrystallization stages may be distinguished, the first leading to a coarse-grained and the second to a superimposed fine-grained structure. According to Kruhl and Voll (1979) these stages are due to granulite and amphibolite facies deformation, respectively. With decreasing metamorphic grade a change in texture is observed.

In the Baldissero complex olivine is oriented with [100] parallel to the lineation, with [001] normal to the lineation in the foliation and with [010] normal to the foliation. Orthopyroxene does not show any significant crystallographic preferred orientation (Nicolas and Poirier, 1976; Garuti and Friolo, 1979).

With decreasing deformation temperature or increasing strain rate in the Finero complex essentially three different olivine textures are observed (Kruhl and Voll, 1979):

- (i) [010] normal to the foliation, [001] girdle in the foliation with maximum normal to the lineation and [100] girdle in the foliation with maximum parallel to the lineation,
- (ii) [010] and [001] girdles normal to the lineation and [100] maximum parallel to the lineation,
- (iii) [010] and [100] girdles normal to the lineation and [001] maximum parallel to the lineation.

The texture of orthopyroxene in the Finero complex always shows a [001] maximum parallel to the lineation, a [010] maximum normal to the lineation in the foliation and a [100] maximum normal to the foliation.

Comparing the textures in the Balmuccia massif with that of Baldissero and Finero, there exists a good agreement between Balmuccia and Baldissero. Because of the lower strain in Baldissero a texture in the harder orthopyroxene phase did not develop. The olivine textures found in the Finero complex are characteristic of low temperature deformation indicating the dominant operation of the $\{0kl\}[100]$ and $\{hk0\}[001]$ slip systems under granulite and amphibolite facies conditions, respectively. The difference to Balmuccia may be due to the higher water content in this massif leading to hydrolytic weakening effects, that is to a change of the plastic anisotropy and to a more intense recrystallization. As a consequence, a texture characteristic of deformation event II is also found locally. The first argument may be also applied to explain the difference between the orthopyroxene textures of both massifs. It should be emphasized that the type of orthopyroxene texture found in Balmuccia seems to be an exception rather than the rule (compare Christensen and Lundquist, 1982). In general Finero-type textures are found in comparable massifs (Nicolas et al., 1971; Carter et al., 1972; Cordellier et al., 1981; Mercier, 1985).

VI.5.4 Tectonic evolution of the massif

Olivine-pyroxene-spinel equilibria (Shervais, 1979) for samples from the Balmuccia massif indicate that it originated in the upper mantle at pressures of 1.2 — 2 GPa and temperatures of about 1200°C. At that temperature the mass began to rise either as a discrete diapir or possibly as part of a general upwelling of the asthenosphere. During this process the mass was emplaced into the lower crust at depths of 34 — 44 km (1 — 1.3 GPa), heating up the crustal granulites to temperatures above 850°C. During and after emplacement, clinopyroxene-orthopyroxene pairs in the massif equilibrated to lower temperatures of $\approx 865^\circ\text{C}$, which are about the same as those in the surrounding granulites. The intrusion of mantle material into the crust may be correlated with deformation event I at temperatures around 1000°C, leading to the dynamically recrystallized porphyroclastic and equigranular grain structure in the massif. According to Boudier et al. (1984) the equigranular grain structure in the eastern part of the massif is derived from the porphyroclastic one by continuing strain, leading to a more homogeneous distribution of the spinel grains, and by syn- or posttectonic olivine grain boundary migration, leading to the inclusion of spinel grains in olivine. Stronger recrystallization restricted to the eastern part may be due to water as is indicated by the abundance of fluid inclusions observed by Boudier et al. (1984). Since foliation and lineation of the peridotites in the equigranular domain have the same attitude as those in the adjacent granulitic gabbros it has been suggested by Boudier et al. (1984) that deformation and recrystallization have occurred at the same time under granulite facies conditions. The age of this metamorphism has been dated as being Caledonian (478 ± 20 Ma, Rb-Sr paragneiss whole rock isochron) at a maximum temperature of

about 820°C and a pressure of 1 GPa (Hunziker and Zingg, 1980). Recent Sm-Nd data from ultramafic and mafic whole rock samples give 607 ± 19 Ma (Voshage et al., 1987) for the intrusion event. These authors interpret the chronological inconsistency between the mafics/ultramafics and the paragneisses by a multiple intrusion affecting different portions of the lower crust.

After intrusion into the crust the massif was subjected to a second deformation phase, manifested in the dislocation and subgrain microstructure. According to our estimates the temperature during this tectonic event did not exceed 650°C. In the western part of the massif this high stress deformation ($\sigma_{II} \approx 300$ MPa) produced an additional fine-grained dynamical recrystallization superimposed on the porphyroclastic microstructure. The second deformation event may be correlated with the tilting of the Ivrea zone. According to Hunziker and Zingg (1980) and Voshage et al. (1987) this tectonic event may have occurred at Hercynian times (293 ± 13 Ma, Rb-Sr phlogopite whole rock isochron) terminating the granulite facies metamorphism.

Tab 5: Comparison of dynamically recrystallized grain sizes $D_{1,2}$, subgrain sizes d and dislocation densities ρ in olivine of different peridotite massifs. Subgrain sizes in brackets are measured optically on decorated thin sections, others are measured by TEM.

	D_1 [μm]	D_2 [μm]	d [μm]	ρ [$10^8/\text{cm}^2$]	References
Anita Bay (New Zealand)	≈ 100	≈ 20	5 - 10	5	B, ML & H, 1971 B, 1977 D, D, FG, C & P, 1984
Mt. Albert (Canada)	≈ 1000	≈ 50	10	2	G & K, 1973
Kittelfjäll (Sweden)	200 - 1000	≤ 5	1.5 - 3 (4.5 ± 1)	1 - 10	BT & B, 1976 BT, 1977 R, 1979
Alpe Arami (Switzerland)	30 - 300		0.6 - 6	1.5 - 5	BT & B, 1976 BT, 1976
Finero (Switzerland)	50 - 200	3 - 75	0.9 - 3	2 - 8	BT & B, 1976 K & V, 1979
Balmuccia (Italy)	≈ 1000	30	2.5 (7 - 12)	7 - 8	this study

A comparison of the results on the olivine microstructure of Balmuccia with those for other peridotite complexes is given in Table 5. Two notable features are the general existence of a bimodal grain size distribution ($D_1 \approx 0.1 - 1 \text{ mm}$, $D_2 \approx 10 - 50 \mu\text{m}$) and an agreement of the subgrain size and the dislocation density within an order of magnitude ($d \approx 1 - 10 \mu\text{m}$ and $\rho \approx (10^8 - 10^9)/\text{cm}^2$). Moreover in the other massifs listed dislocations also have Burgers vectors of $[100]$ and $[001]$. $[100]$ dislocations are mainly arranged in low angle tilt boundaries while $[001]$ dislocations are found in screw orientation. The agreement with the results on the microstructure in the Balmuccia massif suggests a similar two stage tectonic history as described above.

VII CONCLUSIONS

Due to the invention of ion-millers TEM has become applicable to minerals and rocks in the early seventies. Since then this method has been successfully applied in geoscience. In the present work the power of TEM in analysing deformation-induced microstructures has been demonstrated clearly. However, inspite of these successes it should not be overlooked that we are still at the beginning of TEM research in the broad and complex field of minerals and rocks. In the following the major problems to be solved by TEM in future research in structural geology will be summarized.

- While dislocations and low angle grain boundaries have been investigated in various minerals, the structure of large angle boundaries is still unknown. Conventional as well as high resolution TEM on well-chosen natural or synthetic polycrystals in combination with energy dispersive micro-analysis might contribute to the basic understanding of the grain boundary structure in minerals and its correlation with grain boundary sliding and migration as well as grain boundary diffusion and segregation processes.
- To unravel the thermo-mechanical history of rocks microstructural parameters have to be compared with those calibrated experimentally. So far most of the experiments have been carried out on single crystals. However, to interpret microstructures in rocks it is necessary to do deformation experiments over a wide range of test conditions on polycrystalline and finally on polyphase materials of varying compositions. This would greatly increase the constraints on the thermo-mechanical history of a particular geologic deformation event. Several stages of the event could be assessed if the kinetics of recovery and deformation differ for the various minerals present. Therefore more laboratory studies of the recovery kinetics in minerals are needed. At present, it is difficult to estimate the times and temperatures required to alter a deformation-induced microstructure. In a similar manner, investigations of the development of steady state microstructures are important. In the best of situations, it may prove possible to determine paleostress levels from partially recovered dislocation structures. For example, Götze and Kohlstedt (1973) have demonstrated that during static annealing of naturally deformed olivine the dislocations rearrange into low angle boundaries the total density given by free and bound dislocations remaining constant. The starting dislocation structure can be worked out by noting that the ratio of free dislocations to those in subgrain boundaries after large strains is approximately 2:1.
- There is also a strong need of TEM studies on naturally deformed rocks of macroscopically well characterized settings. Here, too, the microstructure should

be investigated in different rock-forming minerals. It would be also helpful if temperatures could be estimated from metamorphic reactions related to the deformation events. These temperatures may be compared with those derived from the microstructure. If the age of metamorphism can be determined then dating of the tectonic event is also given.

Finally I hope that the present work will encourage some young geoscientists to enter the promising field of microstructural analyses by TEM.

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APPENDIX

Nomenclature of ultrabasic rocks

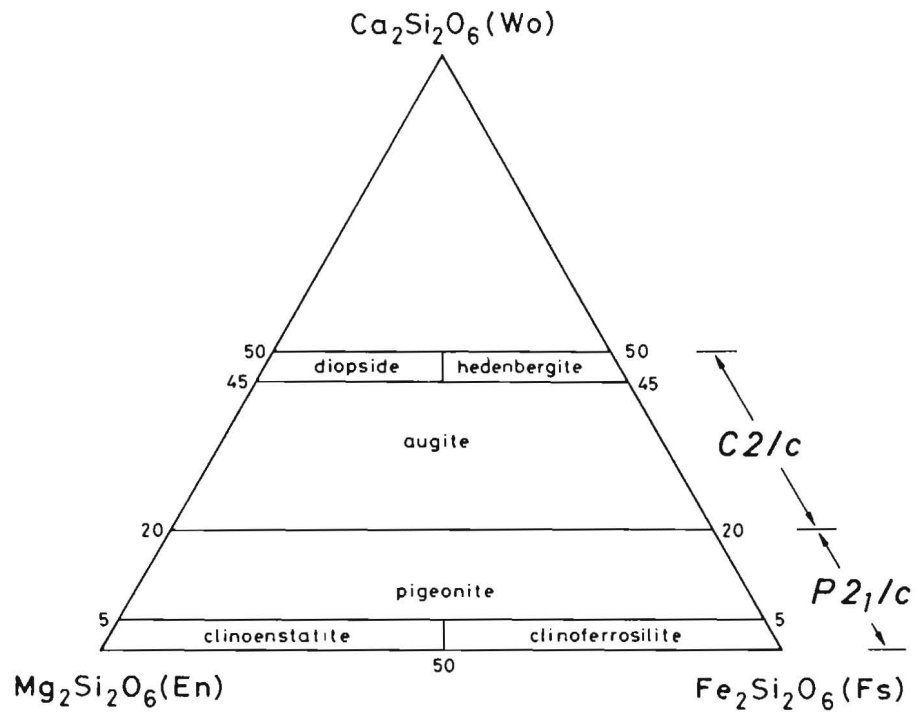


Fig. A1: Composition ranges of Ca-Mg-Fe clinopyroxenes according to the nomenclature of Morimoto (1988). Generally the compositions of the pyroxenes are given by the components Wo = wollastonite, En = enstatite and Fs = ferrosillite.

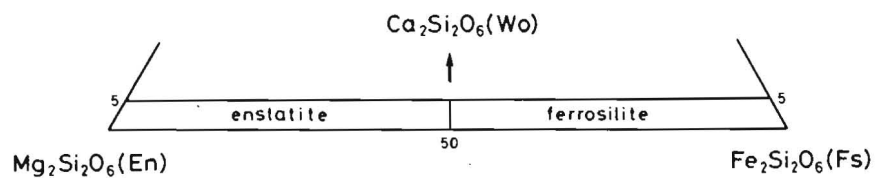


Fig. A2: Composition ranges of orthopyroxenes according to the nomenclature of Morimoto (1988).

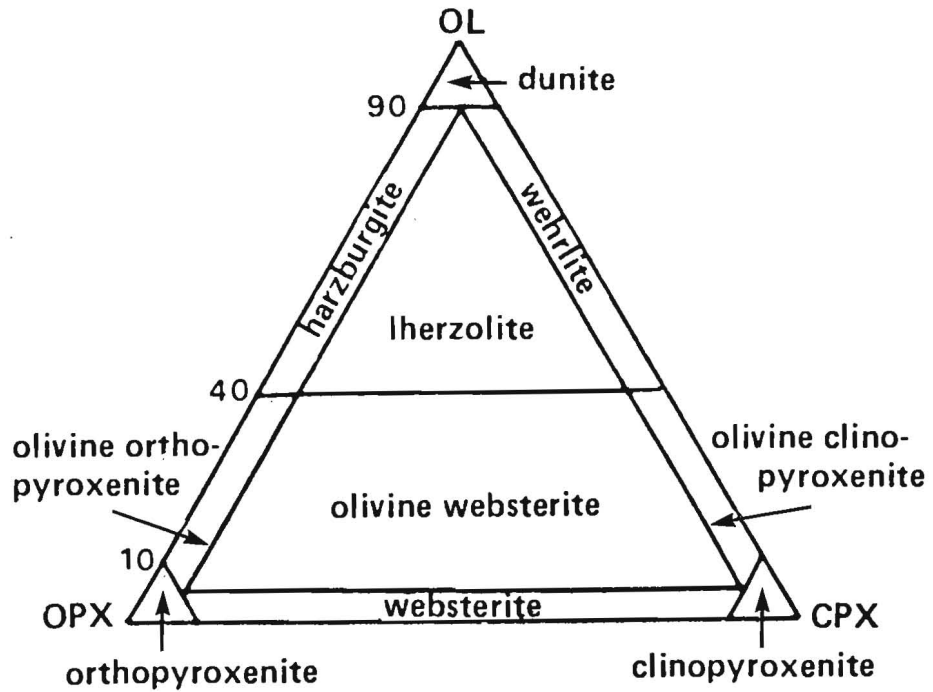


Fig. A3: Nomenclature of ultrabasic rocks consisting of olivine (OL), orthopyroxene (OPX) and clinopyroxene (CPX). Olivine ($(\text{Mg}, \text{Fe})_2\text{SiO}_4$ (orthorhombic) is a solid solution of forsterite Mg_2SiO_4 and fayalite Fe_2SiO_4 . The pyroxenes are characterized in Figs. A1 and A2.

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