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Neutron Diffraction Texture Analysis in Extruded Al-Pb Composites

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Dedicated to the memory of Professor Günter Wassermann

The textures of the two phases of Al–Pb composite samples of various compositions extruded 96% at room temperature were determined by neutron diffraction. In both phases the degree of texture decreases with an increase in amount of the other phase. In the case of the harder Al-phase, this is due to the decreasing internal deformation degree of the hard Al-particles in the soft Pb-matrix. In the case of the softer Pb-phase, the decrease must be related to increasing turbulence of flow in the soft phase. Furthermore, this phase was recrystallized after extrusion.

KEY WORDS: Neutron diffraction, inverse pole figure, composites, aluminium, lead, internal deformation degree.

INTRODUCTION

Following earlier work by G. Wassermann and his coworkers (Merz and Wassermann, 1965; Neuß and Wassermann, 1973) texture studies in highly deformed composites provide a good means to obtain information about the deformation behaviour in two-phase materials. The deformation behavior of a single crystal is to be described by the yield locus which relates the strains to the applied stresses. If an individual grain in a polycrystalline material is considered then stress and strain are local quantities which may be variable even within one grain. In this case, the complete nine-dimensional yield locus as a function of crystal orientation has to be considered for each grain. This is much too complex a problem to be comprehensively dealt with. Hence, current theories start with the assumption of strain being homogeneous within the whole material (Taylor, 1938) which seems to be already a good approximation in many cases. In improved theories this assumption has been relaxed thus allowing for different strains in different grains (see e.g. van Houtte, 1984).

In two-phase or poly-phase materials the homogeneous strain assumption is generally not even a first order approximation especially if the phases have different deformation resistance. If a mixture of a hard and a soft phase is being deformed, the soft phase may flow around the hard one such that the overall deformation of the hard phase is much smaller than the deformation of the whole sample. In the extreme case, the hard phase may not deform at all. On the other hand the overall deformation of the soft phase is higher than that of the sample and it is more turbulent since the soft material has to flow around the hard particles. Furthermore, it has been found that deformation of the hard phase may deviate from that of the whole sample not even with respect to the degree but also with respect to the shape of deformation (Wassermann, Bergmann, Frommeyer, 1978). Harder iron particles in a softer copper matrix may exhibit nearly plane-strain deformation whereas the whole sample is axially symmetrically deformed e.g. by extrusion. Hence, in order to obtain the same degree of approximation as in a single phase materials the deformation behaviour of multiphase materials is at least to be considered with respect to the overall "internal" deformation of each phase separately.

In the above-mentioned theories, applied to single phase materials, texture changes are related to the overall deformation of the material and thus texture studies provide a good means to study the deformation behaviours in such materials. In the same way texture studies in poly-phase materials may give information about the deformation behaviour if they are being referred to the internal deformation of each phase separately.

Texture studies in single phase materials are normally being carried out by x-ray pole figure measurement followed by pole figure inversion (ODF analysis). Pole figure inversion methods are now available which start from incomplete pole figures. Hence, it is usually possible to measure only back-reflaction pole figure thus avoiding the more difficult preparation of transmission samples.

In the case of two-phase materials the preparation of tranmission samples is even more difficult, if not impossible, in many cases. Furthermore in this case x-ray texture measurements in back-reflection samples may be influenced by the effect of anisotropic absorption (Bunge, 1986). This effect occurs if the particles of the phase have an anistropic form such as lamellae or fibres and if their smallest diameter is smaller and their greatest diameter is greater than the penetration depth of the used x-rays. In unfortunate cases this effect can modify the reflected intensity by an order of magnitude (Hanneforth, 1986) hence rendering x-ray texture measurements completely impossible.

The effect of anistropic absorption does not occur in neutron diffraction. Because of the much smaller absorptions coefficients of the neutrons compared with x-rays, (see e.g. Bacon, 1975) the sample can usually be chosen smaller than the penetration depth (Kleinstück *et al.*, 1976). Hence, the above mentioned condition for the occurrence of anisotropic absorption is never fulfilled. Thus, neutron diffraction is the only reliable method to study textures in highly deformed two-phase composites.

EXPERIMENTAL PROCEDURE

Aluminium–lead composite samples were prepared from powders of the two materials which were sieved to mean grain sizes of 50–70 μm. The powders were mixed in a turbula mixer for one hour. They were then compacted by uniaxial pressure of 10^8 Pa. After that they were extruded at room temperature to 96% reduction in area. For lubrication purposes a lead coating was used. Two samples of the pure metals and four composite samples with 20, 40, 60, 80 volume percent of aluminium were thus produced with a final rod diameter of 6 mm.

In order to measure the internal deformation degree of the two phases, scanning electron microscopy was used. Longitudinal and transverse sections were prepared and investigated using the microscope Cambridge S600 at magnifications of about 100–1000. Photographs were taken and quantitatively evaluated using a graphical tablet MOP 1 connected to a computer PSI 80. The deformation degree of the harder Al-phase was determined by measuring the mean cross-sectional area of the Al-fibres in the transverse section.

The neutron texture analysis was performed at the GKSS-Research Centre at Geesthacht using the texture diffractometer TEX 1 at the reactor FRG 1. The layout of this instrument is shown in Figure 1. Pole figure measurements were carried out using the Eulerian cradle shown in Figure 2. The maximum diameter of the primary beam was 45 mm. The usable cross-section is, however, limited in the present state by the diameter of 25 mm of the cylindrical ^{3}He counter. In order to fully exploit the available

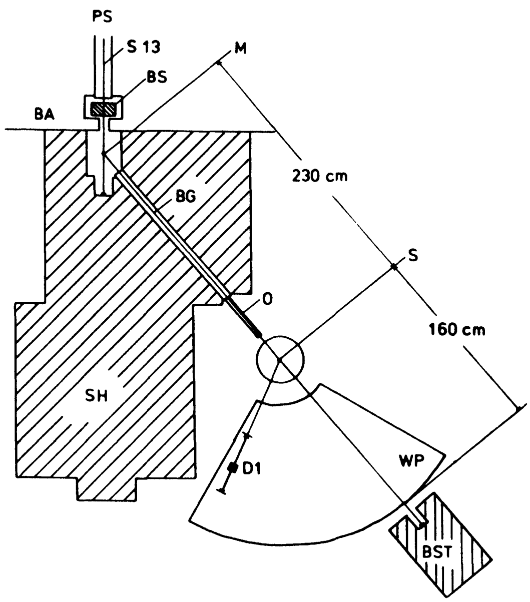

Figure 1 Layout of the texture diffractometer TEX 1 at the reactor FRG 1 at Geesthacht

PS	Primary beam	BA	Biological shielding
S13	Beam guide 13	BS	Beam shutter
M	Monochromator	BG	Beam guide
O	Optical bench	S	Sample
D1	Detector	WP	Detector bank
BST	Beam stop	SH	Shielding

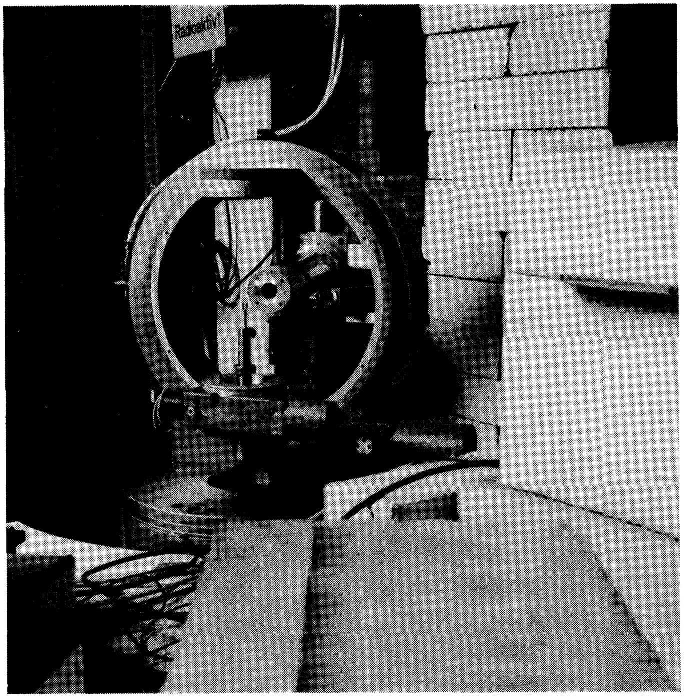

Figure 2 The Eulerian cradle with sample mounted.

cross-section and thus to reduce the measuring time composite samples of 12 mm width and 12 mm length were prepared as shown in Figure 3. With respect to absorption a compound sample of this kind is equivalent to the ideal spherical shape (Tobisch and Bunge, 1972).

Because of the axial symmetry of the extruded samples the pole figures are axially symmetric, too. Hence, only one-dimensional α-scans need to be measured. They were taken in 5°-steps for the (111) (200) (220) pole figures of both aluminium and lead. The counting time per step depends on the volume fraction of the corresponding phase. The wavelength used was 1.618 Å.

The pole figures were used to calculate the ODF which is, in this case, independent of the angle φ_1 and hence is equivalent with the inverse pole figure of the axial direction. The calculations were

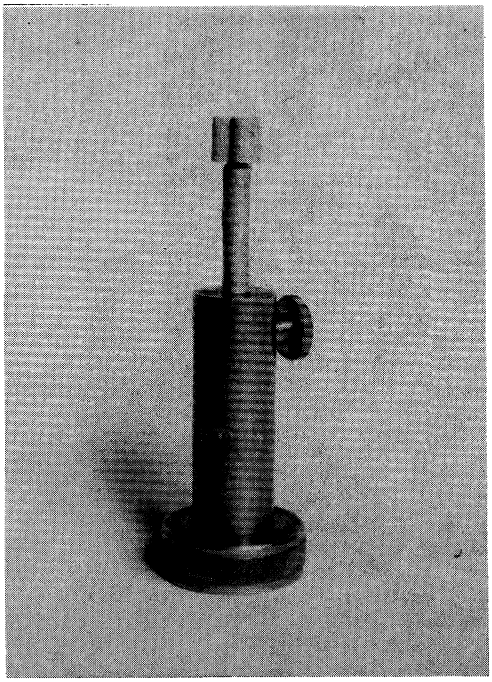

Figure 3 Composite sample mounted on the sample holder.

carried out with a series expansion up to $l = 22$ for even l-values (see e.g. Bunge, 1982).

RESULTS

Examples of a longitudinal and a transverse cross-section of an Al60-Pb40 sample are shown in Figure 4. The hard aluminium phase (black) is immersed in the softer Pb-phase which forms the matrix. The internal deformation degree of the Al-fibres defined by the mean cross-section of the fibres compared with the mean cross-section of the starting powder particles is shown in Figure 5. It is seen that the degree of deformation of the hard Al-phase is the higher the higher its volume fraction. In the pure Al-sample it equals the macroscopic deformation degree η of the whole sample.

a)

b)

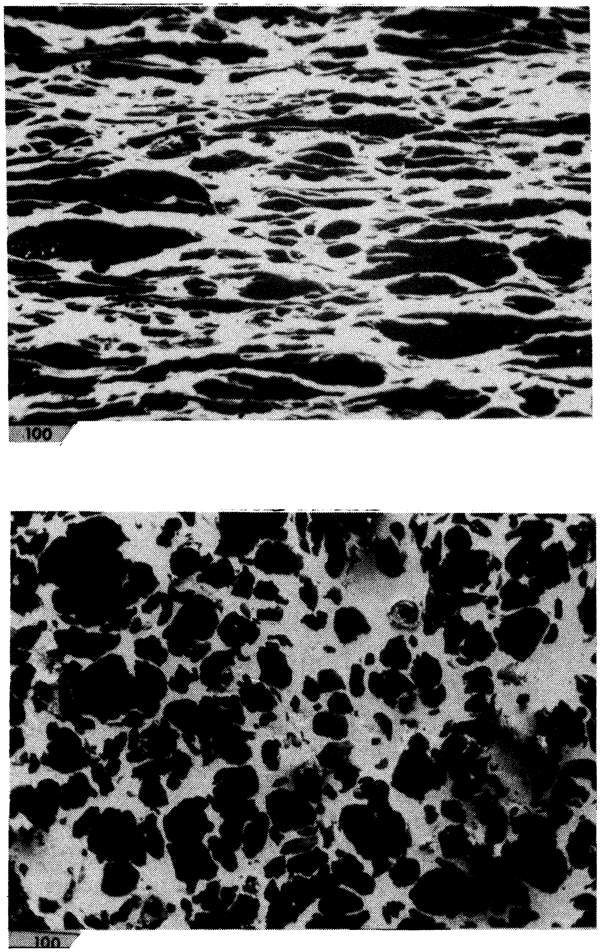

Figure 4 Scanning electron micrographs of an Al60-Pb40 sample. a) longitudinal section, b) transverse section.

If there are only few particles, e.g. 20% then their internal deformation degree is only 30% compared with the external degree of 96%. The hard Al-particles are merely floating in the soft lead matrix, in this case.

Two measured pole figures of the pure metals are shown in Figure 6. Inverse pole figures, calculated from a total of three pole

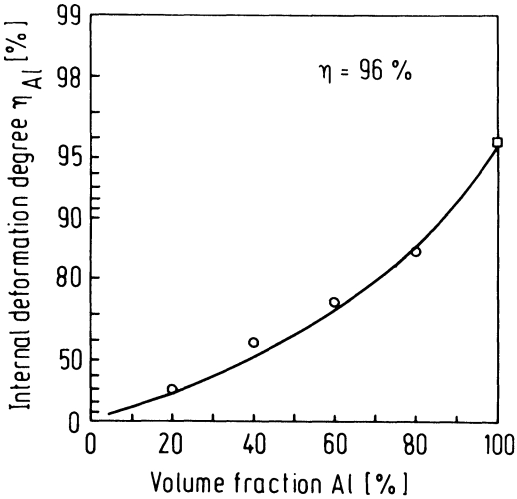

Figure 5 The internal deformation degree η_{Al} of the Al fibres in the composite as a function of the volume fraction. (Scale proportional to $\log 1/(1-\eta_{Al})$).

figures are given in Figure 7. It is seen that the pure Al-sample develops a strong $\langle 111\rangle$-deformation texture with only a minor $\langle 100\rangle$-component whereas the pure Pb-sample shows a strong $\langle 100\rangle$-recrystallization texture. The textures of the composites are much weaker but contain the same two texture components.

The textures of the pure metals are rather sharp. This means that with a series truncation at $l = 22$ a truncation error occurs additionally to the error due to the omission of the odd part. Hence, the total error in the inverse pole figures amounts to about 20%. In the specific case of $\langle 111\rangle$ and $\langle 100\rangle$ fibre textures, the main features of these two texture components can be estimated by the maximum orientation densities at the corresponding points of the inverse pole figures which are identical with the normalized pole densities of the (111) and (200) pole figures at $\alpha = 0$ respectively. These latter intensities, however, are free of truncation errors. Hence, the normalized pole densities of the Al-(111) and Pb-(200) pole figure at $\alpha = 0$ are plotted in Figure 8. It is seen that both textures decrease with decreasing volume fraction in the sample. The reason for this decrease is, however, completely different in the

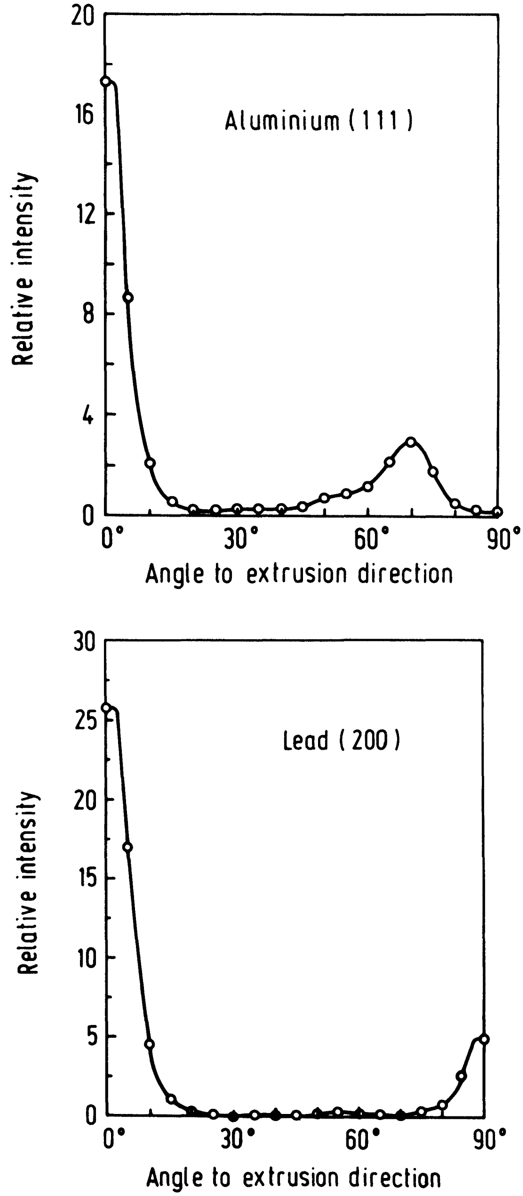

Figure 6 Normalized $(111)_{Al}$ and $(200)_{Pb}$ pole figures of the two single phase samples.

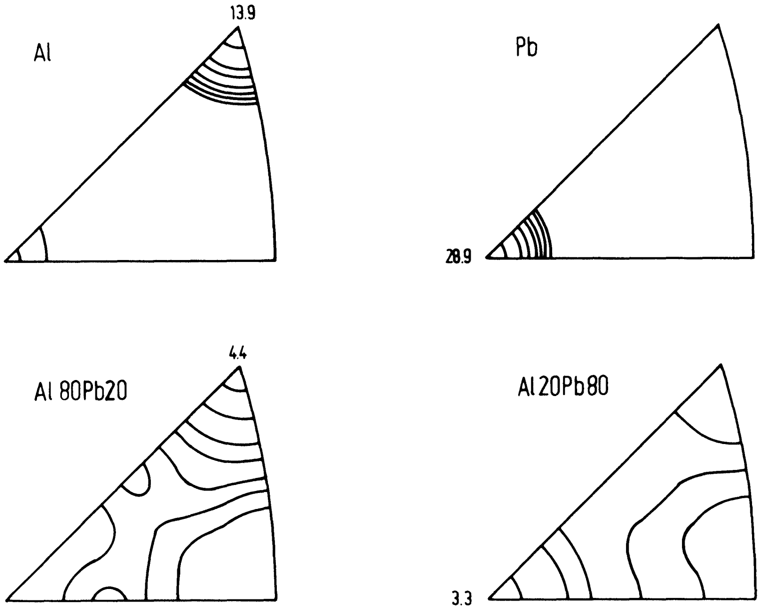

Figure 7 Inverse pole figures of the pure metals and of the Al and Pb-phase of Al-Pb composites deformed 96% by extrusion at room temperature.

two components. In Figure 9 the orientation density of the ⟨111⟩ comonent of the Al-phase is plotted (in logarithmic scale) against the internal deformation degree of this phase taken from Figure 5. Hence, the "degree of texture" increases with increasing internal deformation degree of this phase. On the other hand, the deformation degree of the soft Pb-phase remains the same or even increases with increasing amount of hard Al-particles (depending on how it is defined in the case of increasing turbulence of flow). At the same time, however, the degree of texture decreases as seen in Figure 8.

CONCLUSIONS

As is seen in Figure 4 the originally spherical Al-particles become elongated in the extrusion direction thus assuming shapes which may be idealized as elongated rotational ellipsoides. Assuming this

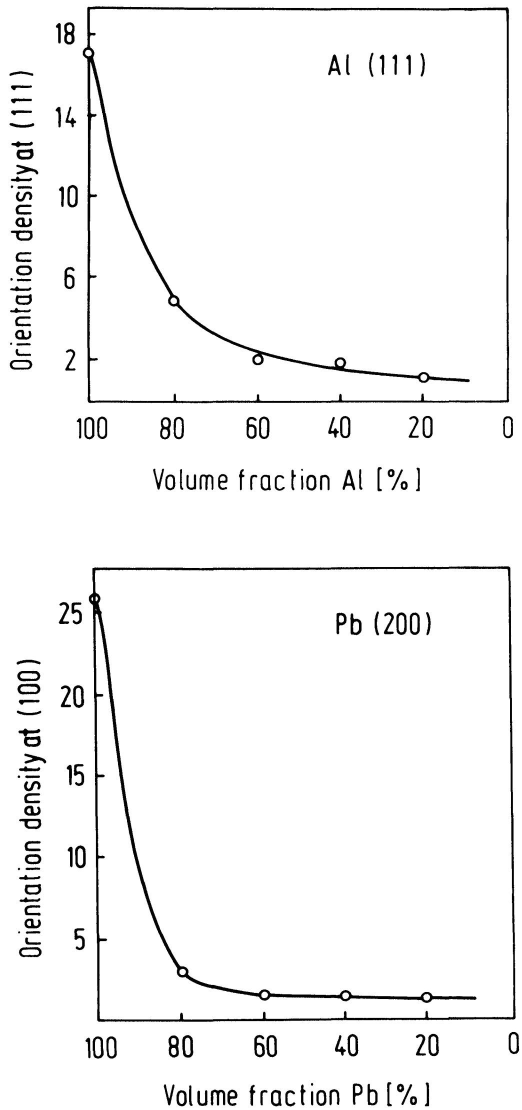

Figure 8 Normalized pole densities at $\alpha = 0°$ of the pole figures $(111)_{Al}$ and $(200)_{Pb}$ respectively as a function of the corresponding volume fractions.

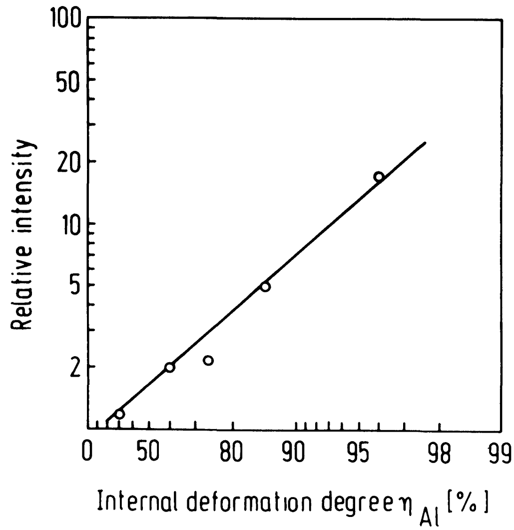

Figure 9 Normalized pole density values of Figure 8a plotted versus internal deformation degree η_{Al} of the Al-phase (The scales are proportional to log I and log $1/(1 - \eta_{Al})$ respectively).

ideal shape, the internal deformation degree of the hard Al-phase can be determined as is shown in Figure 5. It increases with increasing volume fraction of Al in the composite. This is easily understood qualitatively. With increasing volume fraction of Al the diameter of the Pb-ranges decreases. Hencé, stronger compatibility strains near the Al-particles are necessary which lead to stronger hardening. Hence, the Pb-matrix can transfer increasing stresses into the hard Al-phase which in turn deforms increasingly. On the other hand, the idealized circular cross-section of the Al-fibres is only an approximation. The real cross-sections become increasingly disturbed as a consequence of the near neighbourhood of other hard Al-particles. Nevertheless, the idealized rotational ellipsoid seems to be a reasonable approximation which corresponds quite well with the development of texture in the Al-phase as is shown in Figure 9. Hence, in this phase a similar situation may be assumed as in a single phase material if only one considers the internal deformation degree instead of the external one. The situation in the soft Pb-phase is quite different from at least two reasons. Firstly, with increasing volume fraction of Al the flow in the soft Pb-matrix becomes increasingly turbulent. Hence, despite of a constant or even higher degree of deformation in this phase the texture becomes weaker. Secondly, the lead phase was recrystallized after extrusion. Hence, its texture was a recrystallization texture rather than a deformation texture. Because of the turbulent flow of the lead phase near the Al-particles the local deformation texture in these regions must be assumed to be nearly random. Furthermore, the local deformation degree in the turbulent regions is higher. Thus, nucleation will preferredly take place in these regions leading to a nearly random recrystallization texture.

As was mentioned in the introduction the internal deformation of the hard phase may deviate from the external deformation not only by the degree but also by the deformation geometry. In the present example i.e. the system Al-Pb, this effect seems to be rather small. Nevertheless, it may be strong enough, also in this case, to be detectable in the textures. Hence, more detailed texture investigations are presently being carried out to answer this question more precisely.

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References

Bacon, G. E. (1975). *Neutron Diffraction.* Clarendon Press, Oxford.

Bunge, H. J. (1982). *Texture Analysis in Materials Science.* Butterworths Publ. London.

Bunge, H. J. (1986). In: *Experimental Methods of Texture Analysis,* p. 395–402. DGM Informationsgesellschaft Oberursel Ed. H. J. Bunge.

Hanneforth, R. (1986). Diploma work TU Clausthal.

Kleinstück, K., Tobisch, J., Betzl, M., Schläfer, D. and Schläfer, U. (1976). *Kristall u. Techn.* **11,** 409–429.

Merz, D., Wassermann, G. (1965). *Z. Metallkde* **56,** 516–522.

Neuss, R. and Wassermann, G. (1973). *Z. Metallkde* **64,** 696–704.

Taylor, G. I. (1938). *J. Inst. Metals* **62,** 307–324.

Tobisch, J. and Bunge, H. J. (1972). Texture **1,** 125–127.

Van Houtte, (1984). Proceedings ICOTOM 7 Noordwijkerhout 7-23.

Wassermann, G., Bergmann, H. W., Frommeyer, G. (1978). *Textures of Materials* (ICOTOM 5). Springer-Verlag Berlin Ed. G. Gottstein and K. Lücke. p. 37–46.