JOURNAL OF MATERIALS SCIENCE 15 (1980) 131–138

X-ray microbeam and recrystallization studies of plastic deformation around fatigue cracks

ELMAR TSCHEGG
Technische Universität Wien, Institut für Angewandte Physik, A-1040 Wien, Karlsplatz 13, Austria.

CHRISTIAN FALTIN, STEFANIE STANZL
Universität Wien, Institut für Festkörperphysik, A-1090 Wien, Boltzmanngasse 5, Austria.

X-ray microbeam technique and recrystallization method were used to measure plastic zones around 20 kHz fatigue cracks in mild steel. Both methods render approximately the same extension of a plastic zone. In order to obtain the amount of deformation within a plastic zone measurements of integral line breadth and recrystallization temperature were calibrated by specimens deformed with known per cent static tension. But since static and cyclic loading cause different dislocation structures (especially the dislocation density is much more inhomogeneous in fatigued specimens) quantitative comparison is difficult. Furthermore the two methods display different values of the actual amount of deformation. X-ray line broadening indicates an average dislocation density, whereas recrystallization displays a maximum amount of deformation, which corresponds to the local dislocation concentrations.

1. Introduction

The size and shape of the plastic zone, as well as the amount and distribution of plastic deformation around a fatigue crack play a dominant role in crack growth. Several theories have been proposed, which are based on the existence of a small plastically deformed region around the crack tip (the cyclic or reversed plastic zone) within a larger area of less plastically deformed material (the monotonic plastic zone) [1–8].

Various experimental techniques have been used to analyse these zones [9], but this knowledge is still incomplete. Therefore, an additional supplementary technique is proposed in this paper which involves a combination of the X-ray method developed by Wood and co-workers [10–24] and a grain recrystallization method [9, 25–28].

2. Experimental procedure

A mild steel was used for this investigation (0.036 wt % C, 0.01 wt % Si, 0.08 wt % Mn, 0.012 wt % P, 0.008 wt % S, 0.022 wt % Al). This was cold drawn to 3 mm diameter rods, then cold rolled to 1 mm x 3.8 mm x 125 mm bands, recrystallized at 700° C for 4 h and furnace cooled *in vacuo*. The grain size was determined metallographically as $\sim 15\,\mu m$, and the material had a yield stress of $270\,MN\,m^{-2}$ and a fracture stress of $340\,MN\,m^{-2}$. No decarburization of the edges could be recognized. Fatigue experiments were performed with 20 kHz ultrasound [25].

The length of the samples was chosen to be equal to the half-wave length of ultrasound in iron in order to obtain resonance. Thus the push–pull maximum stress arises in the centre of the sample. The displacement amplitude was measured by an electrodynamic converter, the signal of which was used for an automatic amplitude-stabilization. The stress amplitude was calculated by differentiation of the wave equation and by means of Hooke's law (controlled by small strain gauges).

To prevent self heating of the samples during high-frequency stressing, the load has been interrupted by periodic pauses in addition to cooling the samples by water or oil.

0022–2461/80/010131–08$02.80/0

3. Recrystallization tests and results

3.1. Recrystallization method

The recrystallization method makes use of the fact that the grain size after annealing at a certain temperature depends on the previous amount of deformation. Another recrystallization method uses the fact that the recrystallization temperature, i.e. the temperature at which recrystallization just takes place, also depends on the previous amount of deformation. This dependence of grain size or recrystallization temperature on the amount of deformation may be used for measuring an unknown per cent deformation. For this purpose the method has to be calibrated, i.e. one has to find the "best" recrystallization condition.

The "best" condition in the case of method one, is the temperature and time which causes recrystallization of all amounts of deformation, but which does not produce significant grain growth. One has to measure grain sizes depending on known amounts of deformation, e.g. by tensile testing or by rolling. This recrystallization diagram then serves as calibration curve for unknown deformations. In the case of bi or triaxial-deformation modes one obtains equivalent amounts of deformation. This means that the microstructural changes in the grains are similar to that obtained by uni-axial deformation with the equivalent amounts of deformation.

When the second method [9] is used, the samples first are annealed at a lower temperature, which only causes recrystallization of the most highly deformed areas. At a somewhat higher temperature, the less deformed areas recrystallize, etc.

The mild steel used in this work is appropriate for recrystallization tests in many respects, as it was relatively "clean" (see composition above), which would favour recrystallization [29], and it had a small initial grain size $\sim 15\,\mu m$. As has been shown by Stanzl and Faltin [30] the annealing temperature *before* deformation is important not only in producing the initial grain size but also in controlling the driving energy for recrystallization *after* deformation. This temperature and annealing time were carefully selected, so that grain-size differences for the various per cent deformations became as large as possible (e.g. grain size $\sim 20\,\mu m$ after 55% strain and $\sim 500\,\mu m$ after 7% strain). Attention was paid to the following difficulties: as stated above, at 7% strain a grain size of about $500\,\mu m$ could be observed. However, it was possible that the grains have continued to grow into a less deformed area, and in order to preclude the possibility, our tests were performed in such a way that this was avoided. (The 7% boundary, i.e. Lüders-strain, produced by static load, was marked before annealing. Then the annealing temperature was chosen to be just high enough to complete recrystallization but to avoid continued growth of the as-recrystallized grains.)

The second difficulty is the minimum size of a plastic zone, i.e. it should not be less than the diameter of the newly formed grains [29]. This implies that very small plastic zones, as might occur around the fatigue cracks, do not cause recrystallization [30]. Very small amounts of deformation, i.e. under about 2%, cannot be measured by the recrystallization method [29].

A third difficulty could arise with steel possessing higher carbon contents, in which annealing may produce decarburization of the surface layers, resulting in larger grains. This effect was not observed in the mild steel used for this investigation; on the contrary, it was found, that the recrystallized areas at the surface were smaller than in the interior of the specimens.

3.2. Recrystallization results of 20 kHz fatigued mild steel

Bands of 1 mm thickness were used as specimens. Consequently the plastic zone is nearly the same in the centre of the samples as at the surface. Therefore, no difficulties are expected if metallographic observations of the crystalline structure and X-ray results are compared. The specimens were broken by an ultra-sonic stress amplitude of $130\,MN\,m^{-2}$ at a temperature of 20° C. The broken specimens were examined by the X-ray method as described in the following section. Afterwards the same specimens were recrystallized and polished, after removing a surface layer of about 0.3 mm.

Fig. 1 illustrates a sample which has been step-annealed [9]. Annealing was started at a comparatively low temperature, i.e. 580° C. Only the areas with the highest amount of deformation recrystallize, this being shown by the 55% line in Fig. 1. At 600° C deformations up to 40% recrystallize; at 650° C, 20%; and at 800° C areas of 7% strain become visible. The lines in Fig. 1 show the boundaries of these zones of deformation produced by fatique loading until the crack reached about 60% of sample width (the resonance limits for these specimen). Furthermore,

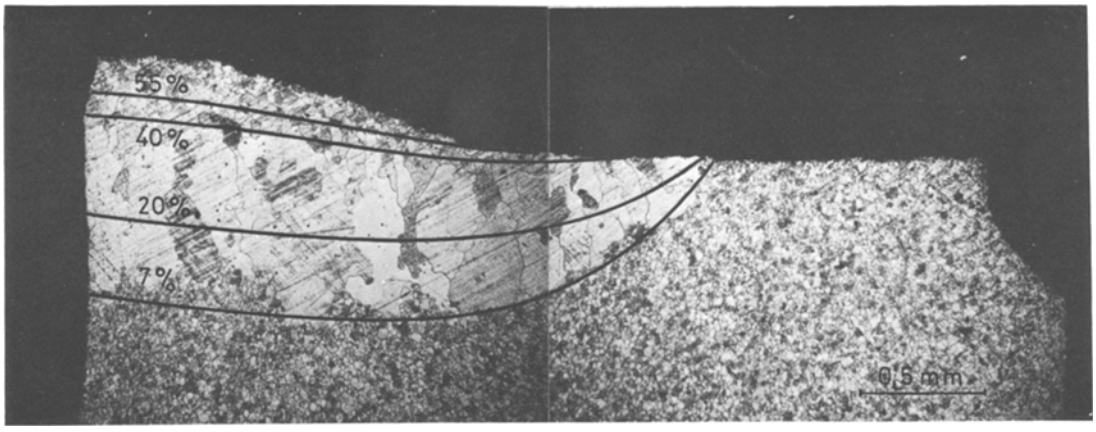


Figure 1 Recrystallization of the plastic zone round a 20 kHz fatigue crack of mild steel. Crack initiated at the notch (right-hand side). Lines indicate amounts of plastic deformation obtained by the recrystallization method with different temperatures, × 35.

Fig. 1 indicates that recrystallization does not begin directly at the notch at some distance away along the crack. This effect has already been treated in more detail [26].

4. X-ray microbeam tests and results

4.1. X-ray microbeam method

An X-ray tube with Cr-anode (wavelength $K\alpha = 2.29$ Å), operating at 30 kV and 30 mA was employed using the (2 1 1) reflection and the back-reflection technique. A collimator tube was used with an inner diameter of 250 μm and a length of 60 mm. The distance between specimen surface and X-ray film was 43 mm and the irradiated area on the specimen surface was about 300 μm diameter. The specimen was not rotated, and the exposure time was 30 min.

4.2. X-ray microbeam tests

The X-ray method was applied to check the results obtained by the recrystallization method. Therefore, the samples were first measured by the X-ray technique and later by the recrystallization method. In Fig. 2 the size and site of irradiated spots are indicated by circles. Measurements are arranged perpendicular to the crack edge. Each row is characterized by a letter. In Figs. 3A to F the sectors of each row are arranged in a circle. Within one circle the distance from the fracture surface is varied. Each sector is assigned a number, i.e. zero is taken directly at the fracture surface, one is the first spot, being adjacent to the fracture edge, and so on.

In Fig. 4 the diffraction pattern of the undeformed material is shown, the spotty ring pattern indicating the presence of defect-free grains. In addition, some Laue reflections appear in this pattern, which are caused by the presence of single larger grains.

In the deformed material two effects are expected:

(1) the sharp single reflexions are replaced by Debye–Scherrer (DS) rings;

(2) the Debye–Scherrer arcs are broadened.

Usually these effects occur simultaneously. For example, the effect of tensile deformation is shown in Fig. 5, in which both effects become more pronced with increasing amounts of deformation.

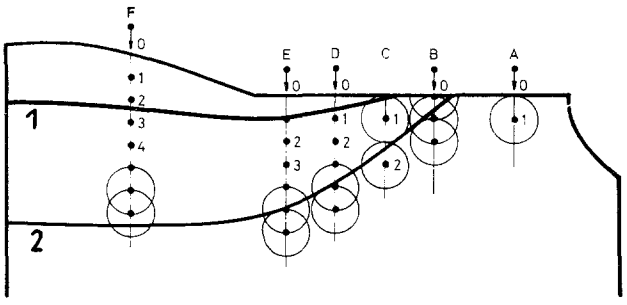


Figure 2 X-ray diffraction test of the same sample as in Fig. 1. Circles indicate irradiated spots. Line 1: boundary of integral line broadening. Line 2: boundary for transition of reflexions to a Debye–Scherrer ring (boundary of the plastic zone.)

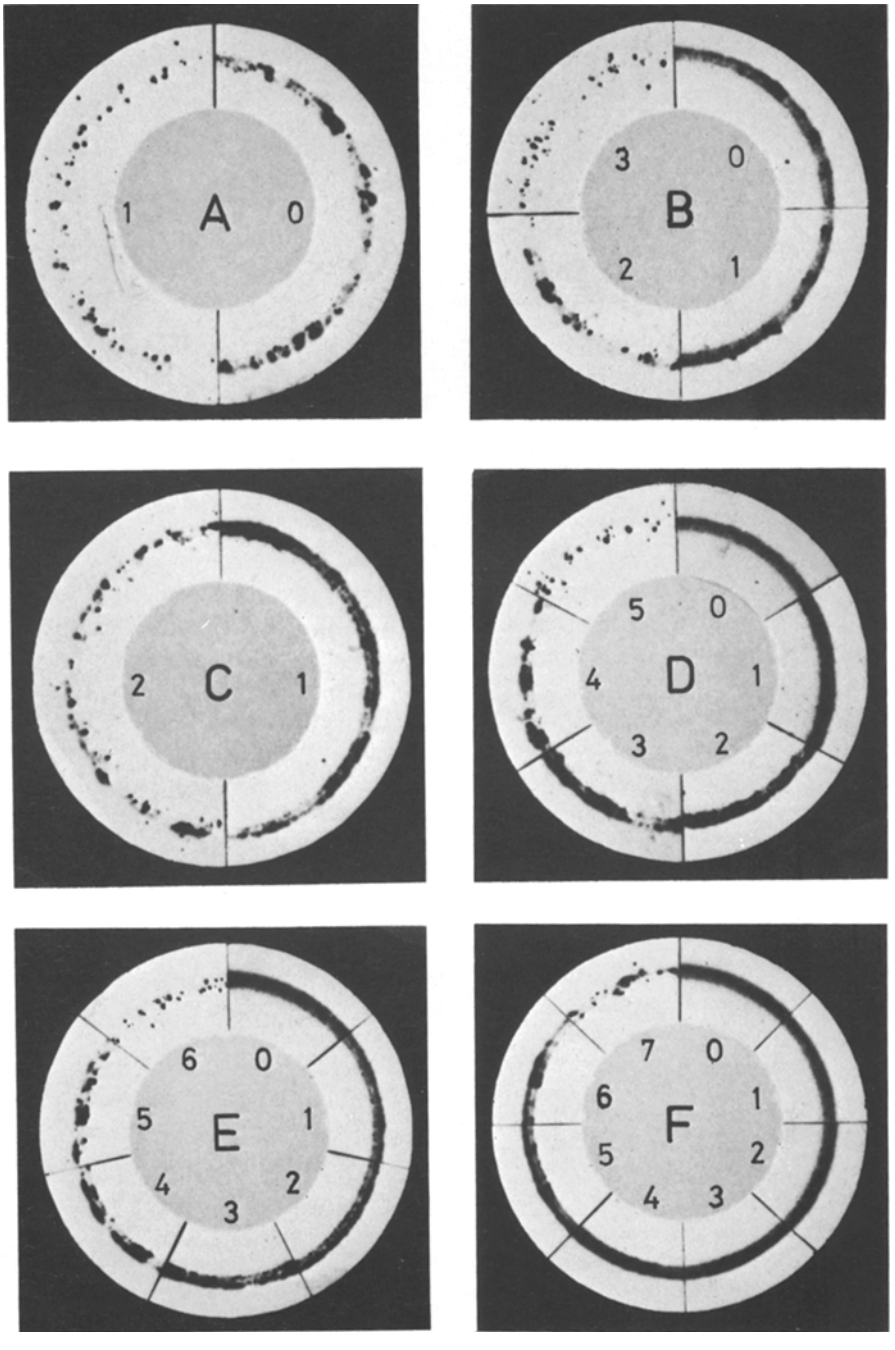


Figure 3 X-ray diffraction patterns around the fracture of 20 kHz fatigued mild steel. Letters A, B, C, . . . , indicate the distances from crack initiation; numbers 0, 1, 2, 3, . . . , show within one vertical row the distance from fracture edge as drawn in Fig. 2. 0 : corresponds to the fracture surface; 1 : the neighbouring point in each vertical row to 0; 2 : the neighbouring points to 1; 3, 4, . . . , the next ones.

By measuring the integral line breath of samples with known amounts of deformation it is possible to calibrate the method. But this calibration is not quantitatively valid for cyclic strains. As Fig. 3A to F indicates, effect 1 changes more than effect 2; i.e. continuous Debye–Scherrer rings are established while no broadening of the rings or reflexions can be recognized (using a photometer).

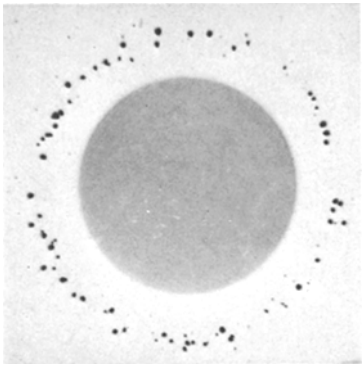
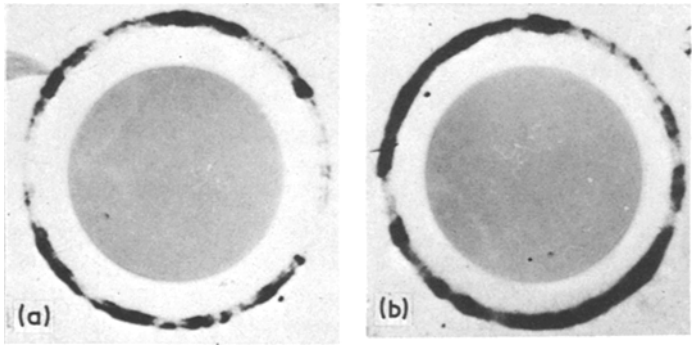


Figure 4 Diffraction patterns of undeformed mild steel.

by static loading, results in a smaller amount of deformation than the recrystallization method. All areas still further away from the crack surface cannot be compared with statically deformed specimens, because their integral line breadth is too small.

Attempts were made to resolve degrees of deformation within this boundary (i.e. amounts of deformation between 7 and 30%) but as may be seen by comparison of Fig. 3 D0, E0, F0, F1, F2, F3 the differences are too small. Thus we may conclude X-ray line breadth measurement is far less sensitive for the measurement of large amounts of deformation than the recrystallization method.



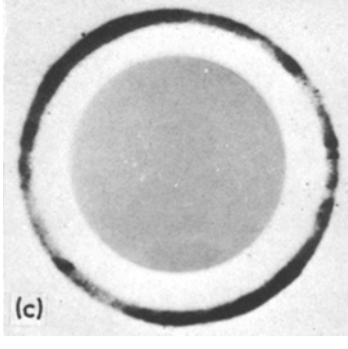


Figure 5 Diffraction patterns of tensile tested mild steel. (a) Tensile deformation 7% (Lüders strain); (b) tensile deformation 11%; (c) tensile deformation 20%.

4.3. X-ray microbeam results

Taking into account these difficulties, the following results may be discussed. The evaluation of the diffraction patterns (Fig. 3A to F) is summarized in Fig. 2, distinguishing three areas with different amounts of deformation. Only diffraction patterns D0, E0, F0, F1, F2, F3 show integral breaths of DS rings, which are greater than in Fig. 5a, i.e. 7% (Lüders strain). If the limit of this zone is indicated (line 1 in Fig. 2) and compared with Fig. 1, it is evident that it corresponds with an amount of deformation of about 30% as determined by recrystallization. The X-ray method, calibrated

Line 2 in Fig. 2 gives the boundary of the plastic zone around the fatigue crack outside of which no difference whatsoever could be detected compared with the undeformed material (Fig. 4, B3, C2, D5, E6).

If this boundary is compared with that determined by recrystallization it is evident that the extension of the plastic zone obtained by the X-ray method is about 100 μm greater than that found by recrystallization. Just inside line 2, continuous DS rings occur (effect 2) but with no measurable line broadening (effect 1).

In addition, as shown in Fig. 3 A1, B1 and B2, the size of the plastic zone at the beginning of the crack growth (i.e. at low cyclic stress intensity) is approximately zero. This is in accordance with recrystallization results. Only the fracture surface shows a change of the diffraction pattern, i.e. a slight broadening of some grain reflexions even at lowest stress intensities (A0). That means that the thickness of the plastic zone is $\leqslant 10\,\mu$m, i.e. the penetration of X-rays.

5. Discussion

Two questions arise from these experimental results;

(1) which changes in a deformed material cause the two effects: (a) the transition of spotty diffraction pattern to continuous Debye–Scherrer rings, and (b) the introduction of line broadening?

(2) why is there a difference in the amounts of deformation as determined by the X-ray and the recrystallization methods, respectively?

To explain the first question some older work which was mainly concerned with high frequency fatigue tests is reviewed [10–24].

Wood and co-workers who used the back-reflection technique (incident beam diameter approximately 1 mm), found for mild steel [10] and for brass [11] "a dispersal of the grains into widely oriented crystallites by static or slow cyclic stress: but not at high frequency cyclic stress" Wever *et al.* [12] found that the use of an alternating stress below the fatigue limit did not cause any change of X-ray patterns, whereas stressing above this limit faded the reflexions. Lihl [13] pointed out by means of X-ray studies, that mild steel hardened inhomogeneously by alternating stresses and he spoke of a "mosaic structure" being influenced by dynamic stress. Later on, Wood and co-workers [14, 15] mentioned that the dislocation theory could account for formation of "sub-boundaries" and "fragmentation" of grains by condensation of dislocations into grain-boundary-like structures. More detailed evidence was obtained by electron microscopy and X-ray diffraction by Hartmann and Macherauch [16] and Wood *et al.* comparing b c c and f c c metals [17]. Karashima *et al.* [18] found for fatigue fracture in f c c metals a remarkable spread of diffraction spots caused by "diffuse substructures formed near the fracture surface". Awatani and Katagiri [19] obtained diffraction patterns of an irradiated area with 1 mm cross-section and found that iron, steel and copper specimens fatigued with ultrasonic frequency, showed "no marked reduction of grains to disoriented elements, although it occurs in conventional low frequency fatigue". Yokobori and co-workers [20, 21] could differentiate with their X-ray microbeam studies (elliptically radiated area with 150 and 80 μm axis) between a macroscopic and a microscopic plastic zone in fatigued low-carbon steel. They measured the "misorientation of the grains and the subgrain size near the crack tip". Similar tests were performed by Latiere [22]. Hempel *et al.* measured integral intensities depending on the number of cycles at alternating bending tests of an unalloyed steel [23]. Nagao and Weiss [24] also used X-ray measurements for explaining microstructural variations induced by different ratios of cycles. It appears reasonable to substitute the old expressions like "fragmentation", "mosaic structure", etc. by "dislocation cell substructure". It is well known that alternating loads produce more pronounced cell structures than static loads. This would explain why one diffraction spot is replaced by a great number of smaller spots, thus increasing the number of spots on a particular diffraction ring.

The interior of the cells is virtually defect-free. Therefore, broadening of diffraction lines is less pronounced than for statically loaded material with a similar amount of deformation in which the interiors of cells contain a large number of dislocations and other lattice defects.

However, this explanation is not completely satisfactory. In samples fatigued with ultrasound at room temperature, transmission electron microscopy has shown that scattered tangles with high local dislocation densities and higher dislocation densities in grain boundaries exist rather than dislocation cells [31]. From this we may conclude that each effect, which subdivides the original grains into zones of high and low dislocation density and which causes little misorientation of these new areas, leads to a split-up of the original spots.

In the case of higher temperature (about 200° C) ultrasonic stressing of mild steel a pronounced dislocation cell structure is obtained [31]. X-ray diffraction patterns of this material display a clear difference compared with room temperature fatigued material (see Fig. 6a and b). More and smaller reflexions form a distinct DS ring. In addition, its integral breadth is even smaller than that of the sample, fatigued at room temperature (Fig. 6a).

From this we can conclude that broadening of reflexions or DS rings is mainly caused by an increase of the average dislocation density. The transition from spotty to continuous DS rings may be understood as the result of discrete subdivision of one grain into several subgrains (areas with very low dislocation density). In most cases these areas are dislocation cells, but less well-established dislocation arrangements may produce similar effects.

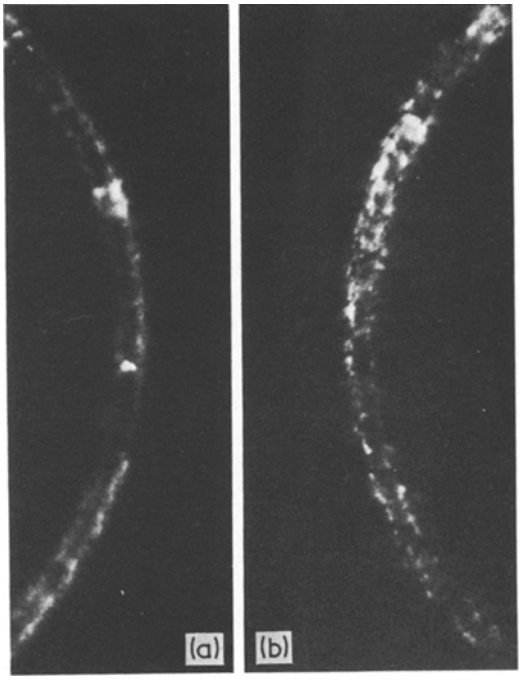


Figure 6 Comparison of X-ray diffraction patterns of 20 kHz fatigued mild steel, broken (a) at room temperature, (b) at 200° C.

The second question is, why the X-ray line broadening method indicates much smaller amounts of "deformation" than the recrystallization method. Recrystallization depends on the formation of nuclei. If their density is high because of a high dislocation density, small recrystallization grains are formed or a relatively low recrystallization temperature is necessary, thus indicating "large amounts of deformation". If zones with very high and very low dislocation densities are closely spaced (as in the case of a fatigued material), the recrystallization process will be initiated by the zones of high dislocation density and therefore indicate "high amounts of deformation". Recrystallization grains which were nucleated inside these higher deformed zones will then start to grow into the areas of lower deformation.

The recrystallization method thus emphasizes areas of large "deformation" of an inhomogeneously strained material.

On the other hand, the X-ray line broadening method is based on a different mechanism. Line broadening of an X-ray beam is determined by the average dislocation density in the irradiated zone. Therefore, the X-ray line broadening method indicates an average amount of "deformation". The second effect, noticed in the X-ray method, i.e. splitting up of spots into a great number of spots, has not been evaluated quantitatively in this work. But it is another measure of the intensity of deformation and indicates a similar extension of the plastically deformed area to the recrystallization method, as can be seen by line 2 in Fig. 2.

Summarizing the results, the X-ray diffraction and recrystallization results may be correlated in the following manner.

Within line 1. Line broadening indicates a large mean dislocation density. Local high concentrations of dislocations cause a great number of recrystallization nuclei (substantially more than in statically strained material, which possesses the same linebreadth).

Between lines 1 and 2. The number of recrystallization nuclei is very small with no significant line-broadening, however, the high density of spots on the DS rings indicate a dislocation structure. This is, however, unable to form new nuclei, but allows growth of the grains, nucleated within line 1. Therefore, the recrystallization boundary lies about 100 μm inside line 2.

In other words, the two methods (recrystallization and X-ray line broadening) determine a valid "amount of deformation". Recrystallization exhibits the local concentration of plastic deformation and X-ray line broadening the average deformation. These two values will be different for samples with inhomogeneous plastic deformation, as is the case for cyclically stressed material.

If we wish to know the *maximum* amount of plastic deformation which has occurred, the recrystallization method is ideal, because it selects the maximum deformed area and is very sensitive to large amounts of deformation.

If we wish to determine the average deformation, then the X-ray broadening method is preferred. Effect 2 of the X-ray microbeam method, i.e. increase of Debye–Scherrer spots, is very sensitive for measuring *small* amounts of deformation, i.e. small dislocation densities. This is the amount of deformation where grain growth becomes effective instead of recrystallization nucleation. It also indicates localized strained grains in an unstrained matrix. Therefore, it is an ideal method for determining the boundary of the plastic zones around a fatigue crack.

6. Conclusions

The X-ray microbeam technique (with 300 μm diameter irradiated areas) has been applied to measure the amount of the deformation and the shape of a plastic zone ahead of cracks. The results are compared with those obtained by the recrystallization method. It is shown, that it is difficult to define an amount of "deformation", especially in the case of cyclic loading which produces inhomogeneous plastic deformation. Two effects are used for evaluation, i.e. line broadening and formation of continuous DS rings. The difficulties which arise with integral line breadth measurements of cyclic plastic zones are discussed.

Measurements have shown that the X-ray line broadening method renders an average amount of plastic deformation, whereas the recrystallization method is sensitive to the maximum amounts of deformation in the same plastic zone.

Acknowledgements

The authors are indebted to Professor Dr E. Hornbogen and Professor Dr H. Ebel for some helpful discussions. We thank Professor Dr P. Skalicky and Professor Dr K. Lintner for making this work possible.

References

1. D. S. DUGDALE, *J. Mech. Phys. Solids* **8** (1960) 100.
2. A. K. HEAD, *Phil. Mag.* **44** (1953) 925.
3. F. A. MC. CLINTOCK, On the plasticity of the Growth of Fatique Cracks, in "Fracture of Solids", (Wiley, New York, 1963) p. 65.
4. B. TOMKINS, *Phil. Mag.* **18** (1968) 1056.
5. B. A. BILBY and K. H. SWINDEN, *Proc. Roy. Soc. London* **285A** (1965) 22.
6. W. ELBER, *Materialprüf.* **12** (1970) 189.
7. C. BATHIAS and R. M. PELLOUX, *Met. Trans.* **4** (1973) 1265.
8. G. T. HAHN and A. R. ROSENFIELD, *Acta. Met.* **13** (1965) 293.
9. E. M. MINUTH and E. HORNBOGEN, *Prakt. Metallogr.* **14** (1977) 14.
10. H. J. GOUGH and W. A. WOOD, *Proc. Roy. Soc. Lond. A* **154** (1936) 510.
11. W. A. WOOD and P. L. THORPE, *ibid* **174** (1939) 310.
12. F. WEVER, M. HEMPEL and H. MÖLLER, *Arch. Eisenhüttenw.* **11** (1938) 315.
13. F. LIHL, *Metall.* **23/24** (1948) 391.
14. W. A. WOOD and R. B. DAVIES, *Proc. Roy. Soc. Lond. A* **220** (1953) 255.
15. W. A. WOOD and R. L. SEGALL, *ibid* **242** (1957) 180.
16. R. J. HARTMANN and E. MACHERAUCH, *Z. Metall.* **54** (1963) 161, 197, 282.
17. W. A. WOOD, W. H. REIMANN and K. R. SARGANT, *Trans. Met. Soc. AIME* **230** (1964) 511.
18. S. KARASHIMA, H. OIKAWA and T. OGURA, *Trans. J.I.M.* **9** (1968) 205.
19. J. AWATANI and K. KATAGIRI, *Bull. JSME* **12** (1969) 1320.
20. T. YOKOBORI, K. TACHIKAWA and A. KAMAI, *Rep. Res. Inst. Strength and Fract. Mat. Tohoku Univ.* **6** (1970) 1.
21. T. YOKOBORI, K. SATO and Y. YAMAGUCHI, *Rep. Res. Inst. Strength and Fract. Mat. Tohoku Univ.* **6** (1970) 49.
22. H. J. LATIERE, *Eng. Fract. Mech.* **8** (1976) 691.
23. M. HEMPEL, H. KUDIELKA and Y. YAZAKI, *Arch. Eisenhüttenw.* **42** (1971) 785.
24. M. NAGAO and V. WEISS, *Trans. ASME* **99** (1977) 110.
25. S. STANZL and R. MITSCHE, *Arch. Eisenhüttenw.* **42** (1971) 411.
26. R. MITSCHE, S. STANZL and F. MATZER, *Arch. Eisenhüttenw.* **45** (1974) 179.
27. G. GIEDENBACHER and F. STURM, *Prakt. Metallogr.* **15** (1978) 3.
28. Y. IINO, *Metal Sci. J.* **12** (1978) 207.
29. E. HORNBOGEN, E. M. MINUTH and S. STANZL, *Prakt. Metallogr.* **16** (1979) 171.
30. S. STANZL and C. FALTIN, *ibid.* 186.
31. S. STANZL, R. MITSCHE and B. WEISS, *Arch. Eisenhüttenw.* **41** (1970) 867.

Received 26 April and accepted 28 June 1979.