Degradation Assessment of Fatigue by X-Ray Diffraction Technique

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Abstract X-ray diffraction technique (XRD) proved useful as a tool for the assessment of material degradation extent after a long-time service. Framework 5 project „XPECTION" [1], [2], [3] was dealing with this task with respect to high temperature creep degradation of boiler tube steels. In this paper, the X-ray diffraction technique was applied to reveal subtle details of the microstructural changes induced by fatigue. This study of micromechanics of cyclic deformation by means of X-ray diffraction technique made it possible us to gather results unattainable by other methods. Some new ideas and results presented in this paper are based on experimental programme results published and discussed in [5].

1 INTRODUCTION AND BACKGROUND

In many industrial applications materials are subjected to degradation of mechanical properties as a result of real service conditions. The assessment of the remnant lifetime of components and structures is commonly based on correlated procedures including numerous destructive, non-destructive and mathematical techniques that should guarantee reasonably precise assessment of the current damage extent of materials in question and the remnant lifetime evaluation of the component under consideration.

Fatigue, the form of failure that occurs in structures subjected to dynamic and fluctuating stresses, is important inasmuch as it is the single largest cause of failure of structural components, estimated to compromise approximately 90% of all metallic failures. Although there exists a vast amount of published information on fatigue of materials, the general understanding of this subject is far from to be complete.

The material damage (D) as a result of cyclic loading, high (creep) temperatures etc. is frequently described in a phenomenological manner by a monotonically increasing function possessing boundary conditions D = 0, at time t = 0 (before loading) and D = D_F = 1 at fracture, i.e. t = t_F . If the damage function is known or suggested, this results in a unique deterministic correlation between the time of exploatation and the extent of material damage, what can be finally used for the remaining lifetime assessment.

Such a unique mathematical function was suggested and used within the EU XPECTION project, [2], [3], for the evaluation of the creep damage extent by means of X-ray diffraction technique, and elsewhere in case of the cyclicly loaded steel and Al-alloy test specimens, [4].

The assessment of the component remnant lifetime by X-ray diffraction technique is based on the fact that mechanical properties of the most materials depend strongly on crystallite size and orientation of structural components, which are the crucial parameters for the determination of the ageing state and the prediction of residual lifetime of the components.

The original crystalline grains can decay into a number of fragments as a consequence of mechanical or thermal loads or phase transitions and frequently this fragmentation is not apparent by a microscope. X-ray diffraction method however, detects the real structure, because it characterises the crystallographic structure of the particles. Therefore, the X-ray diffraction can become a powerful tool during the remnant lifetime assessment of in-service degraded components and structrures, base metals and weldments.

There are two ways, in which the real structure can be studied by XRD. In case of crystallites smaller than 100 nm, the feature of the real structure influences the broadening of the diffraction lines. In case of crystallites greater than 10 μm, the whole diffraction line in a two-dimensional shape, the so called azimuthal profile, can be considered. Diffracted X-ray beams, which fulfil Bragg´s law are no single „lines“ , but cones with a two-dimensional information. In the case of fine crystallites the diffraction lines are continuous with homogeneous azimuthal intensity distribution. In the case of coarse crystallites greater than 10 μm however, the diffraction lines become spotty and the information on the size, shape, orientation and various structural defects of crystallites can be derived from the distribution of diffraction intensity throughout the corresponding spots (azimutal or lateral diffraction line profile) by so called grain-by-grain (GBG) method. The 2D „lateral, azimuthal“ profile was recognised as a valuable source of the basic information as far as the extent of material degradation degree due to permanent plastic deformation is concerned.

2 CYCLIC STRAIN AND FATIGUE STRENGTH

Cyclic deformation, unlike unidirectional deformation, does not spread uniformly through a grain but tends instead to concentrate cycle after cycle in preferred zones, which finally turn into microcracks. These zones differ according to how large are the amplitudes of the imposed cyclic stress [12].

Large amplitudes reduce a grain to a disoriented mosaic cell structure and concentrate deformation at the cell boundaries. The dislocations too tend to line up in cell formations, some of which may coincide with the structural cells and some with separate mobile-dislocation cells. Thus to concentrate deformation at cell boundaries the grains must behave as if cycle after cycle they buckled in and out always at much the same boundaries. In the course of time, the cyclic buckling disintegrates the cells thereby increasing their surface energy. This energy serves then as the driving force for recovery and recrystallization of disintegrated cells.

At medium amplitudes, the deformation concentrates along regularly aligned slip zones; few structural cell boundaries form which might provide alternative sites for concentrated deformation. The reduced disorientation provides dislocations with a longer free path along their slip zones. This allows the dislocations to persist in those zones for many cycles decreasing at the same time the buckling the grains which seems to be the main reason for disintegrating coherent scattering regions. So the zones grow and the number of their growth defects (paracrystalline distortions) increases up to a moment when they break down the zones into smaller fragments. And, once more, the energy stored in this way in the disintegrated regions starts later on their restoring to the original condition.

At small stress amplitudes, the deformation again persists in slip zones but now only to a limited degree. Deformation, before starting slip-zone microcracks or significant notch-peak effects in one zone, moves on to other zones; it progressively disperses. The dispersion can be accounted for by the reasonable assumption that in any slip zone where deformation persists two opposing processes occur. One is a strain-hardening of the zone, which may be significant even though hardening averaged over a whole specimen is small. This hardening will tend to make the cyclic deformation leave one zone and start another elsewhere. The second process is crack nucleation. Since this process must weaken a zone it will induce deformation to continue in the zone more and more. The to-and-fro movement of dislocations due to small amplitude cyclic strain sweeps the dislocations from various small volumes softening in this way cold-worked metals. The regions depleted from dislocations become strain-free nuclei which grow at the expense of their disordered surroundings. And, after a

prolonged growth period, they again disintegrate as a consequence of accumulated paracrystalline distortions [6],[7].

3 MONITORING MICROSTRUCTURAL CHANGES BY X-RAY DIFFRACTION

Generation and multiplication of dislocations, their mutual intersections and various reactions which result in arrangement of dislocations into miscelaneous structures affect azimuthal profile of x-ray diffraction lines [8], [9]. The azimuthal profile (χ-scan) of an x-ray diffraction line, i.e. the distribution of diffracted intensity along the „Debye ring“ of diffraction (Fig.1) is in fact a superposition of diffraction spots from individual coherent scattering regions (CSRs) – mosaic blocks (cells). In fact, the diffraction spots are reflections of the incident x-ray beam from the corresponding CSRs which – in case that they are suitably oriented – diffract x-rays similarly as a mirror reflects the rays of sun [10]. If the CSRs are small (<10 μm), their number in the irradiated volume is large and their reflections overlap, forming thus a continuous background of the diffraction line. In case that the CSRs are greater than 10 μm, their reflections do not overlap any more and rise above the background constituted from overlapping reflections of small CSRs [11] – see Fig.2 (diffraction pattern) and Fig.3 (azimuthal profile).

For achieving an assessment of the material properties from the two-dimensional X-ray diffraction patterns an evaluation procedure has been developed taking into account particular features like the statistic and crystallographic underground separation as well as the isolation of the coarse grain figures in the measured patterns.

In case of crystallites greater than 10 μm, the whole diffraction line in its two-dimensional shape, the so called azimuthal profile χ, has to be considered. Diffracted X-ray beams, which fulfil Bragg´s law are no single “lines”, but cones with two-dimensional information (Debye cones). When a polycrystalline sample with random orientation of the crystallites is placed into the X-ray beam, the Debye cones are detected, and any linear scan through that cone will give an accurate powder pattern. When the sample consits of few coarse crystallites, not all orientations are represented and the diffracted beams are focused on single spots allocated on the Debye cone. Cut by a film or a two-dimensional detector, circles are measured in their whole shape or partially. In this way, each X-ray diffraction reflex has characteristic 2θ – position, that allows the substance identification and a characteristic azimuthal profile (Fig. 3). The latter is used for the real structure analysis.

In the case of fine crystallites the diffraction lines are continuous with homogeneous azimuthal intensity distribution like in Fig. 3. In the case of coarse crystallites greater than 10 μm however, the diffraction lines become spotty and the information on the size, shape, orientation and various structural defects of crystallites can be derived from the distribution of diffraction intensity throughout the corresponding spots (azimuthal or lateral diffraction line profile) by the so called grain-by-grain (GBG) method (Fig. 4). And even in case of fine-grained material, narrow and parallel beam makes the diffraction line spotty and enables the application of GBG method [1] - [3].

However, real structure investigation by GBG method requires special detection techniques. While conventional powder diffraction uses zero or one dimensional detectors, measuring the azimuthal diffraction line profile requires two dimensional detectors (global area detectors). Such detectors record X-ray diffraction patterns efficiently with exposure times in the range of ten seconds. This opens the door for the field or on site XRD for real structure investigation of critical components using modern digital technologies.

4 EXPERIMENTAL MATERIALS AND TEST RESULTS

A mild carbon steel was used for this investigation (0.1%C, 0.4%Mn, 0.2%Si). Test specimens were fatigue loaded with 2000, 4000, 6000, 8000, 10000, 12000, 14000, 16000, 18000, 20000 and 22000 cycles at constant strain of 0.25% in tension without fracture. Azimuthal profiles (χ-scans) of the ferrite (211) diffraction line of some of these samples are given in Figs.3 – 8. For each χ-scan, the total area P_1 of all solitary peaks corresponding to mosaic blocks greater than 10 μm as well as the area P_2 under the curve of the continuous background made up from overlapping reflections of the mosaic blocks smaller than 10 μm has been determined (Fig.9). Then, the proportion of the large mosaic blocks quantified in terms of the value of $y = \log_{10}[100.P_1/(P_1 + P_2)]$ is displayed as a function of the number of cycles n in Fig.10.

From this plot it is apparent that cyclic stress results in alternating dispersal and repeated assembly of dislocations. This leads to a rotating disintegration and growth of mosaic blocks – CSRs, the boundaries of which are formed by the piled up dislocations. The size of the structural cells (mosaic blocks) peaked after 6000, 10000 and 22000 cycles, these summits being interlaced by valleys after 8000 and 16000 cycles.

5 DISCUSSION OF RESULTS

The structure of materials subjected to cyclic strain is told to be in a "cyclic state". This cyclic character is expressed at two levels. On the one hand, with the period of the cyclic load (usually about seconds), on the other, with the period of thousands and tens of thousands of cycles, which portions the development of the material's structure in cyclic strain out into a number of stages. On going through these stages, initial microcracks are nucleated by pile-up and rearrangement of dislocations, growing and, at the end, causing the structure to collaps locally where they congregate in sufficient numbers. The crack propagation being a complex process which includes both strain and relaxation, the rotation of which paves the way to the final failure. This process is cyclic, indeed: the cyclic stress brings about decay and disorientation of structurally coherent cells (mosaic blocks), activating at the same time the recrystallization (relaxation) processes. But, in course of recrystallization, paracrystalline distortions arise and accumulate, which finally breaks up the growing (crypto-defective) crystallites. The structure, disordered, disintegrated in this way will be regularized, recrystallized by further cyclic loading again etc., etc. Monitoring this cyclic process by x-ray diffraction enables to estimate the residual service life of pertinent components (constructions).

6 CONCLUSIONS

The results of X-ray diffraction investigation of steel and Al-alloy fatigue loaded test specimens proved that the X-ray diffraction technique can efficiently qualitatively and quantitatively describe the changes that occured within the microstructure of test samples. This technique thus can be used for description of material degradation in service and asessment of the remnant life of components and structures.

The experimental data presented in this paper reveal the possibility of cyclic material response during harmonic fatigue loading. The idea of a monotonic damage function need not be generally valid, especially in the early stages of fatigue process before an extensive cracks are initiated. This fact can thus complicate the remaining lifetime evaluation because of the non-unique correlation between diffraction patterns (in this case, or another experimental technique) and the extent of material damage.

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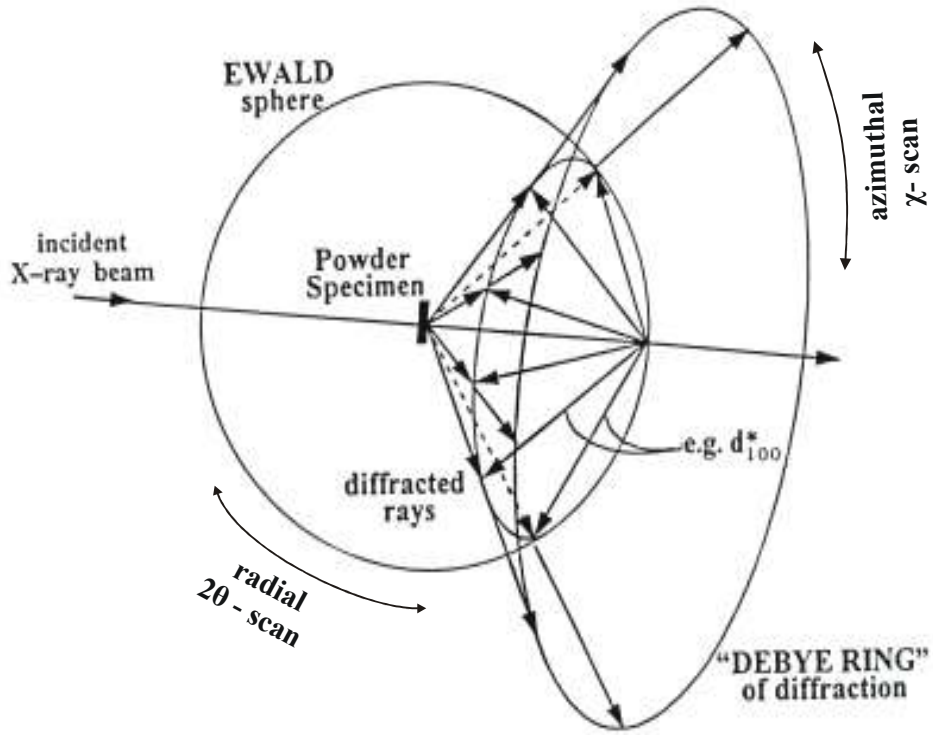

Fig.1. The distribution of diffracted intensity along the „Debye ring" makes up the azimuthal profile (χ-scan) of the diffraction line.

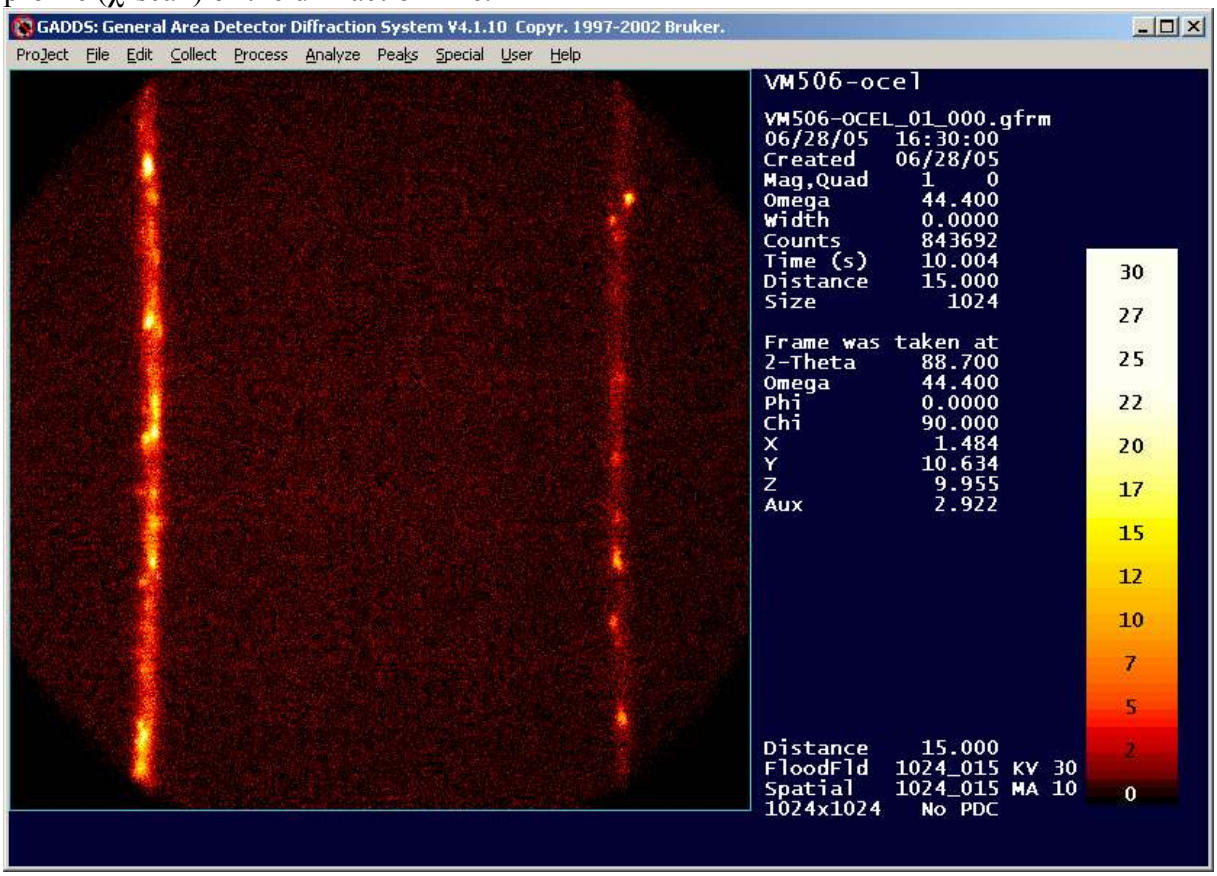

Fig.2. X-ray diffraction pattern of the steel under study sample – virgin state (before loading) composed of ferrite diffraction lines (211), left, and (200), right

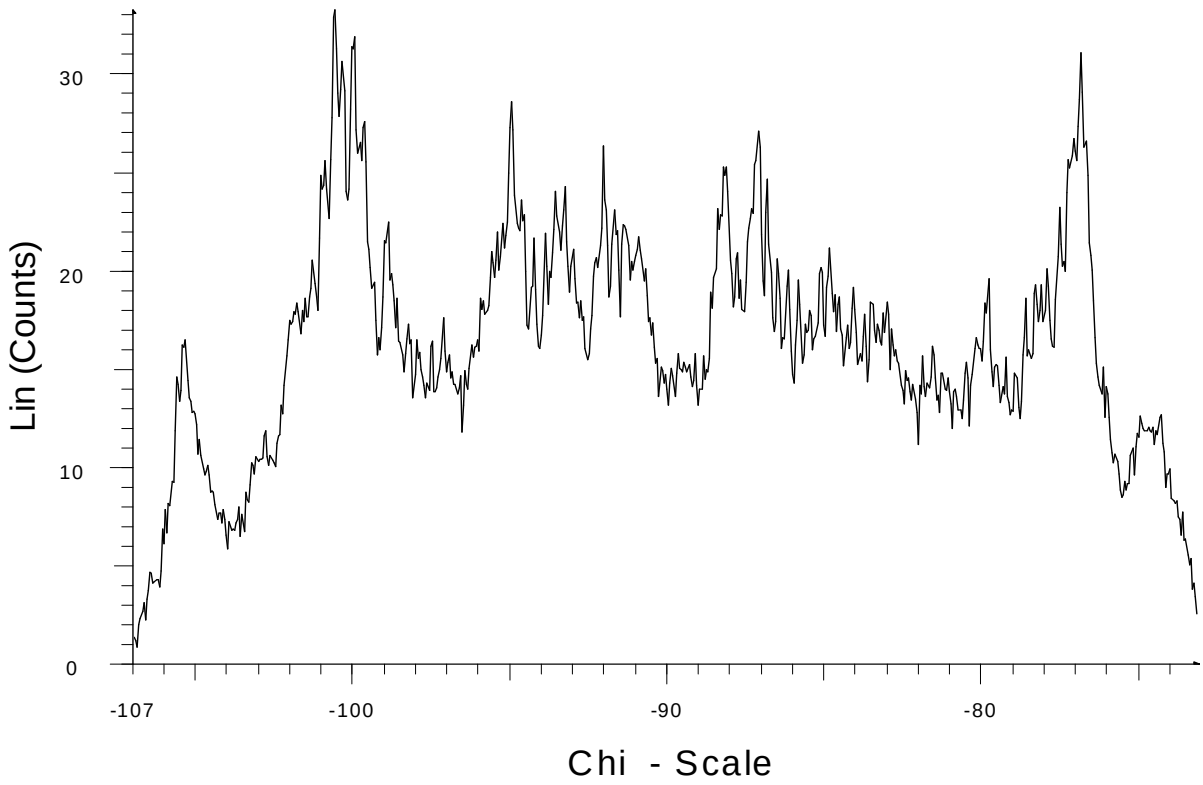

Fig.3. Azimuthal profile (χ-scan) of the ferrite (211) diffraction line situated on the left hand side of the diffraction pattern given in Fig.2: virgin state (before loading)

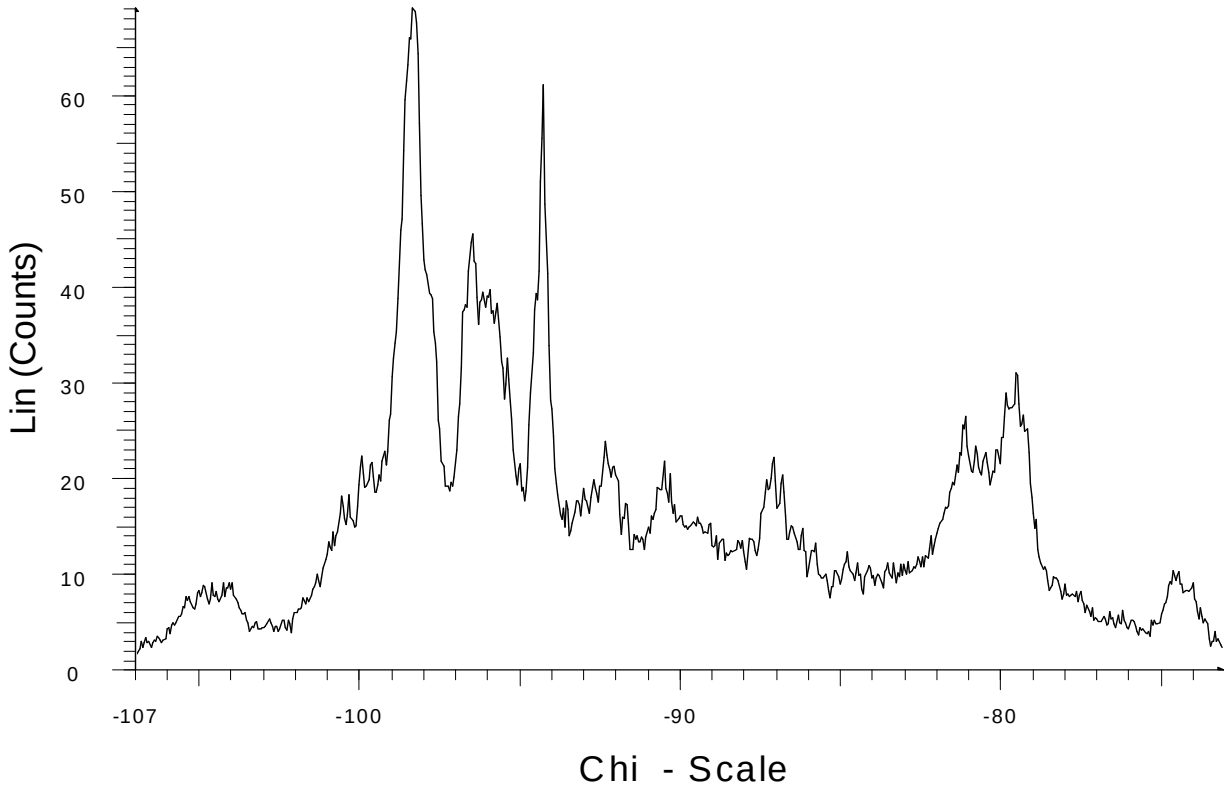

Fig.4. Azimuthal profile (χ-scan) of the ferrite (211) diffraction line: after 6000 cycles

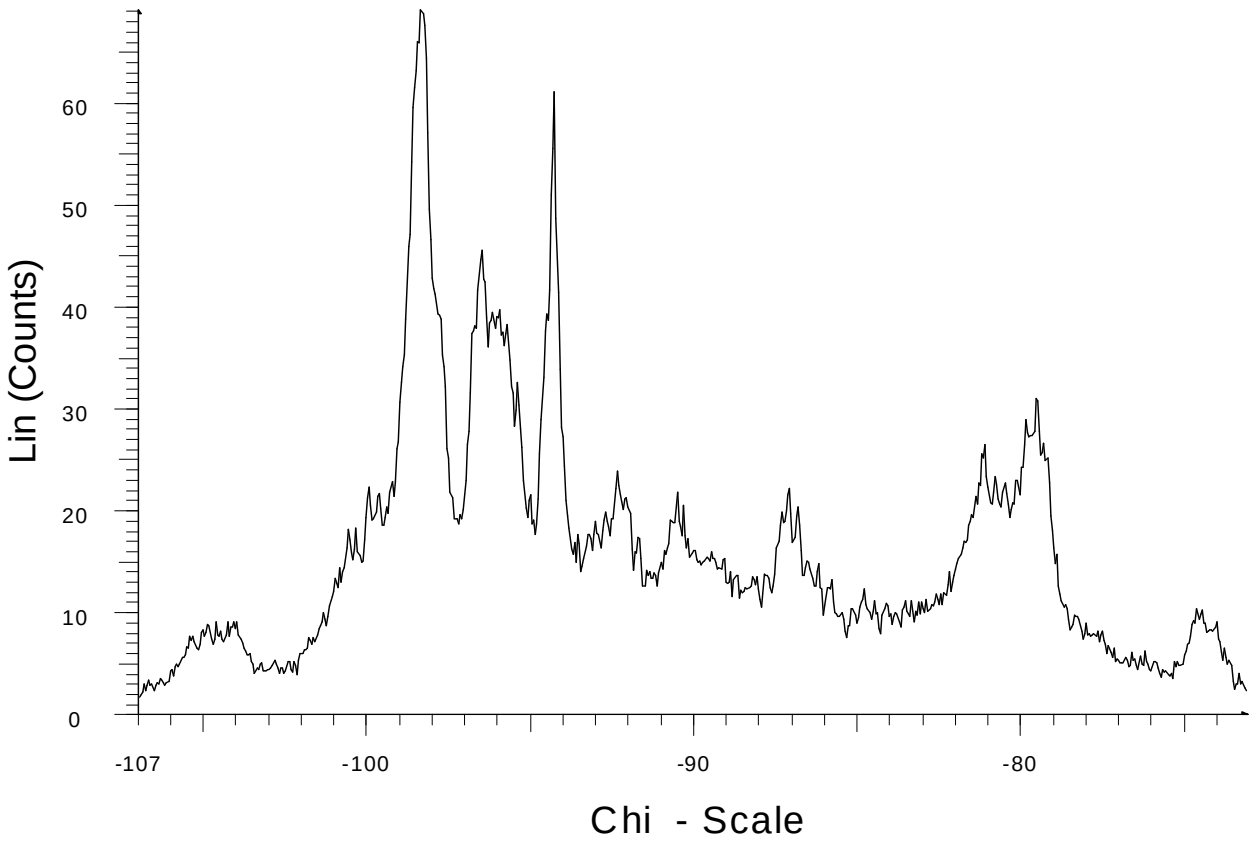

Fig.5. Azimuthal profile (χ-scan) of the ferrite (211) diffraction line: after 8000 cycles

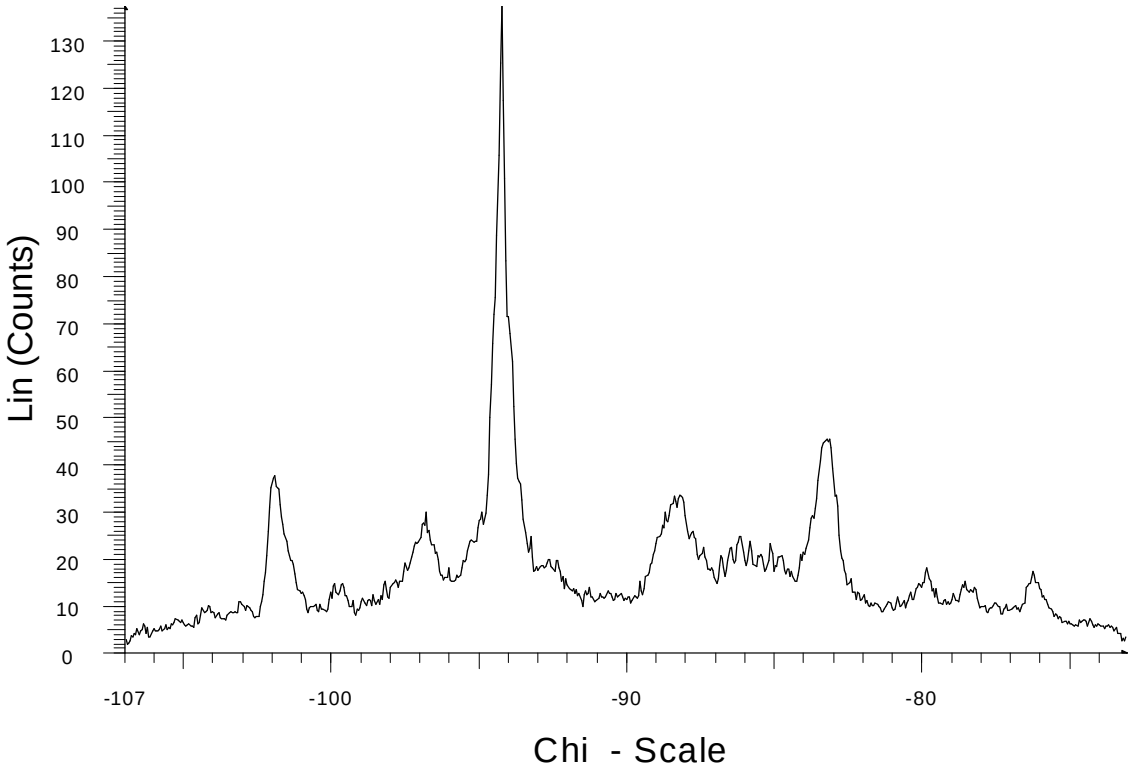

Fig.6. Azimuthal profile (χ-scan) of the ferrite (211) diffraction line: after 10000 cycles

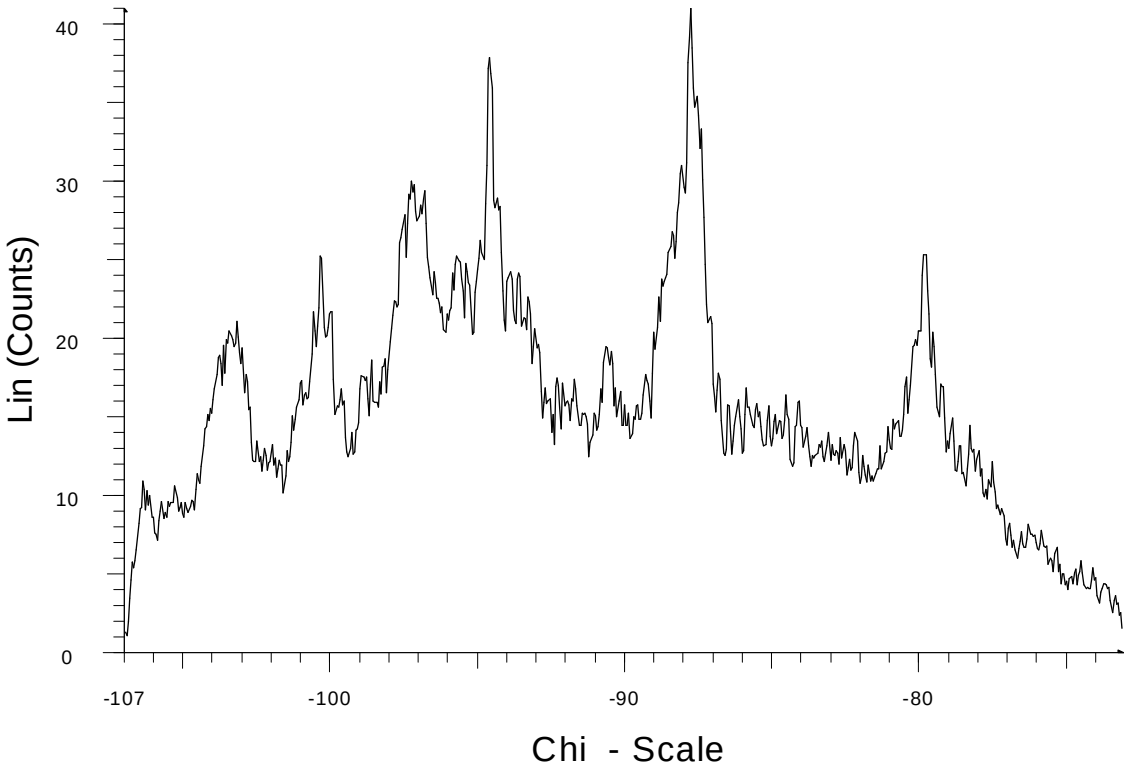

Fig.7. Azimuthal profile (χ-scan) of the ferrite (211) diffraction line: after 16000 cycles

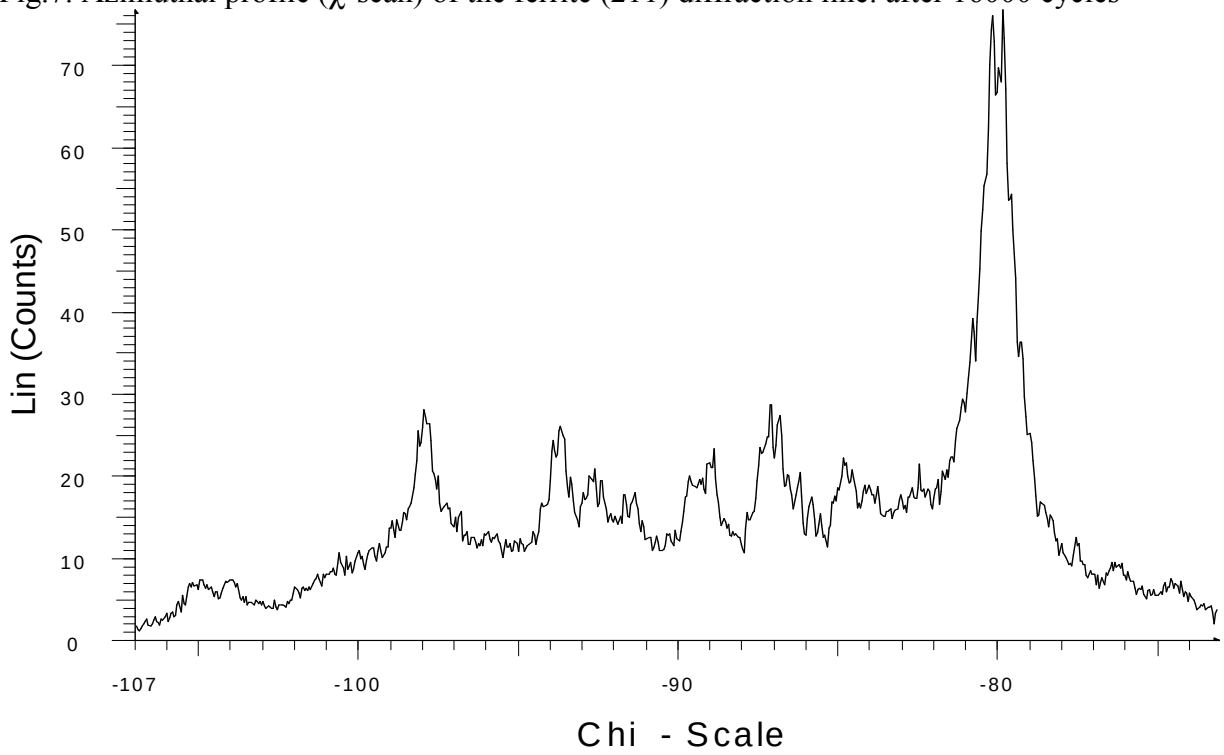

Fig.8. Azimuthal profile (χ-scan) of the ferrite (211) diffraction line: after 22000 cycles

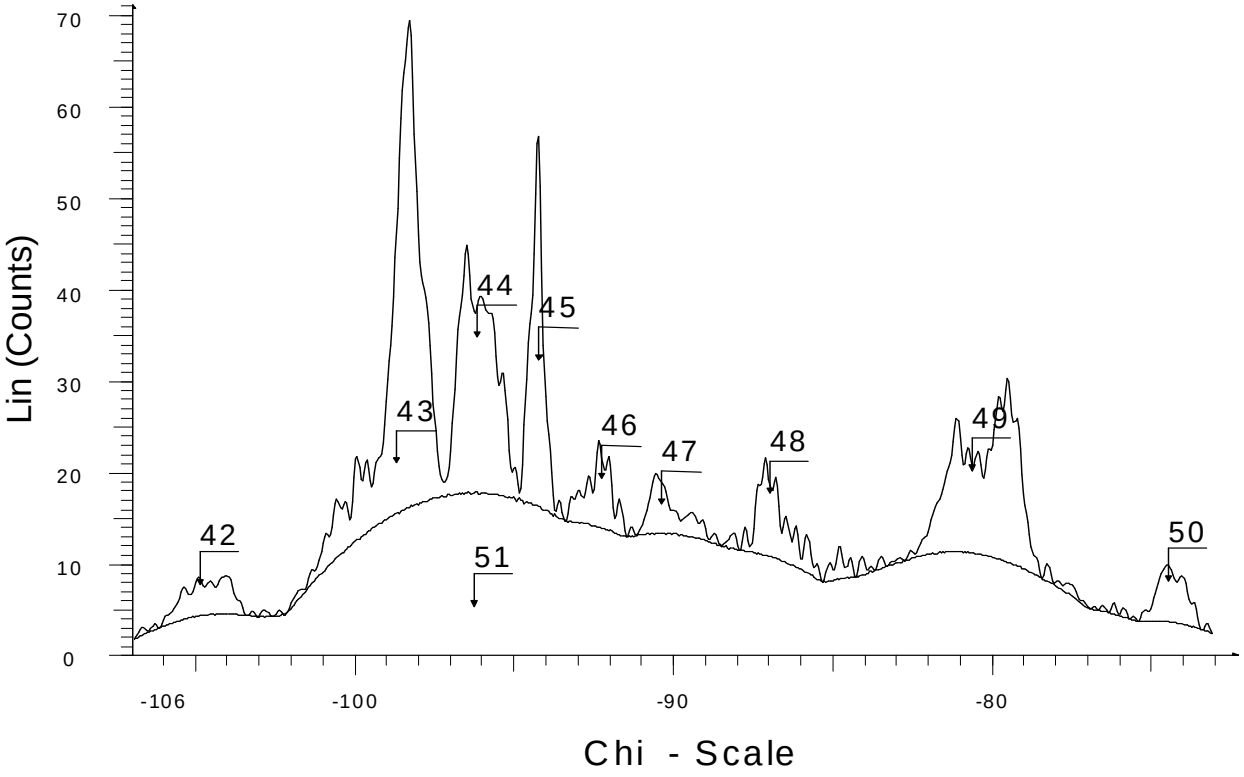

Fig.9. Determining the values of the quantities P_1 and P_2 in the formula $y=\log_{10}[100.P_1/(P_1+P_2)]$ (see text): P_1 = the sum of areas under the solitary peaks rising up above the background = P(42) + P(43) + … + P(50) = 121.2 ; P_2 = the area under the continuous background = P(51) = 186.2 ; y = 1.59. Here, e.g., P(43) means the area under the peak designated “43”, etc.

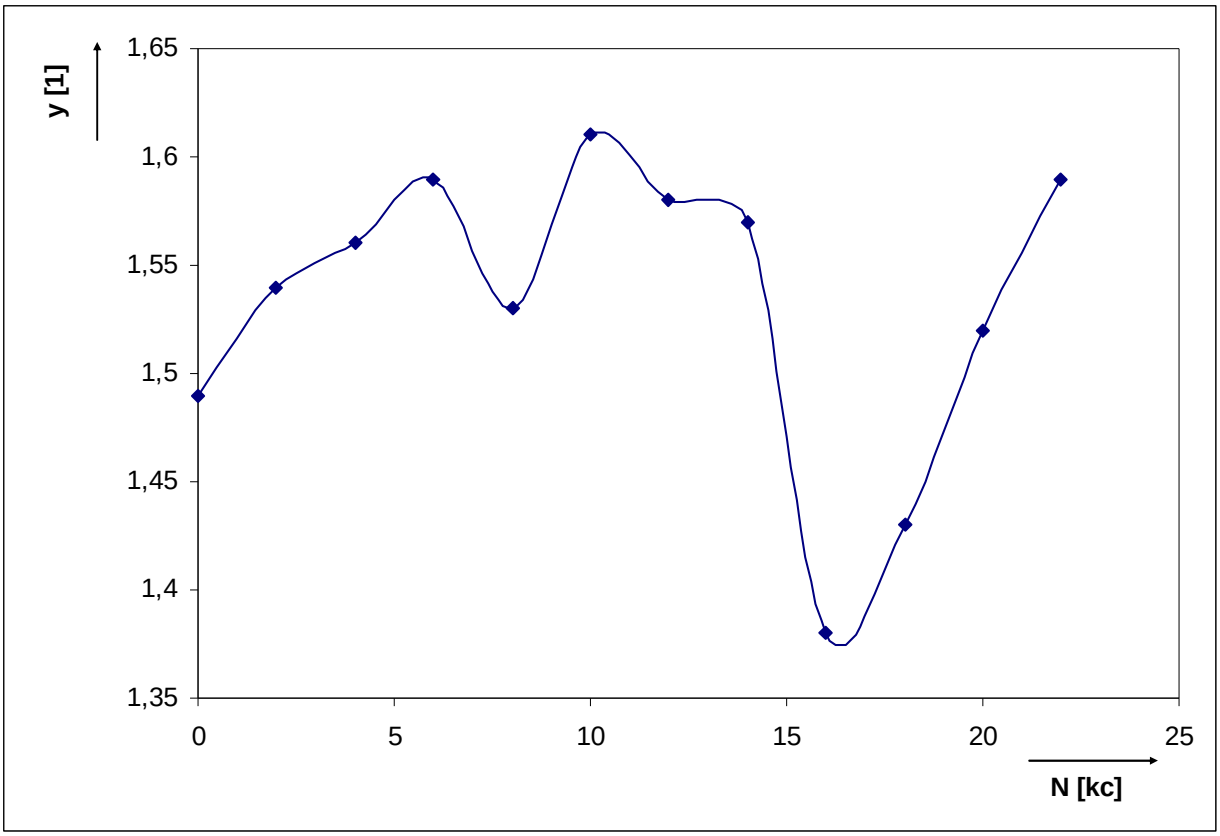

Fig.10 Variation of the coarse fraction (>10 μm) of mosaic blocks (expressed in terms of the quantity $y = \log_{10}[100.P_1 / (P_1+P_2)]$ quoted in the text) with the number of load cycles N (kilocycles).