I-RAY INVESTIGATION ON FATIGUE OF METALS

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Abstract

Profile of X-ray diffraction lines is very sensitive to the change in structure of metals. The X-ray diffraction technique is employed for studying the mechanism of fatigue of engineering metallic materials. The half-value breadth of diffraction lines is taken as the measure of change in structure that has occurred in the metals subjecting to fatigue stress. The behavior of recidual stress during fatigue process, being measured by X-ray technique, is also studied for obtaining additional informations. The finding in the change of half-value breadth during fatigue is interpreted by the mechanism of fatigue that is derived by submicroscopic study of fatigued material by means of the X-ray beam. The contents is a proposition of interrelating the macroscopic phenomena with the findings in submicroscopic scope.

1. Introduction

There are several ways of approach in the studies of problems concerned with fatigue of metals. As the basic study of fatigue phenomenon, process of fatigue of metallic materials is discussed in the scope of order or disorder of atoms composing the materials. In order of eliminating the factors influencing to complex character of fatigue, single crystals or specimens of extremely coarse grains of pure metals or metals of simple chemical composition are used as the test materials in this line of works. We are supplied ample knowledges of fundamental behaviour of atomic structure in metals during fatigue process.

A great number of studies, on the other hand, have been made for the purpose of application to engineering problems. In this line of work, materials subjecting to the investigation are mainly engineering metals of rather complex composition and fracturing of materials is observed or interpreted from macroscopic or submacroscopic viewpoint. This side of approach is, of course, of immense importance to machine designer and metallurgists.

In practice, various criterions of fatigue lives of materials have been proposed so far to predict the lifetime of structures subjecting to fatigue stressing. These criterions are in general serived basing on probable models of fatigue mechanism that are introduced from the finding in fundamental study in atomic order. Although some of the criterions of fatigue fracture are useful for the prediction of lifetime of materials, we are seldom convinced of the fracture mechanism, because of lack of vivid proof of the fatigue mechanism on the engineering materials under consideration. In the model of fracture mechanism, the change of micro-structure is assumed to occur during fatigue process, while the fatigue criterions deal with macroscopic character of fatigue. In this meaning, the authors believe that there still remains a gap to be fulfilled between the fatigue studies in engineering field and those in the physical aspect. It is therefore desirable to do some afforts for search of practical experimental means to find the change in micro-structure of engineering metals, enabling us to recognize propriety of the fracture model.

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The technique of X-ray diffraction is not new as the tool of experimental study, but has often been adopted for direct and non-destructive observation of change in minute structure of crystalline materials at fundamental research. It is known that many valuable findings are derived from the observation of micro-structure by means of X-ray diffraction in the field of fundamental study of materials. 1)-5) In the present study, the authors intend to apply the X-ray diffraction technique to elucidate the minute variation of micro-structure that has occurred in engineering metallic materials during fatigue process.

The authors have conducted so far a series of X-ray studies on fatigue on engineering metals, where the half-value breadth of X-ray diffraction lines was adopted as the measure of the change in structure. The relation of half-value breadth and number of cycles of fatigue stresses was studied in detail for various sorts of engineering metallic materials. As the results of a number of experiments, it has been found that the variation in half-value breadth showed very regular relation with number of cycles. In the earlier half of the present paper is presented a brief survey of the finding in this line of investigation. It would be noteworthy to mention that the finding in this investigation promises an optimistic expectation of non-destructive detection of fatigue damage and also of prediction of lifetime of materials in fatigue.

The technique of X-ray diffraction that has been employed in the early studies was, however, of usual back-reflection, being conventional means for application to fatigue study in engineering aspect, and so the finding obtained were not sufficient for microscopic interpretation of fatigue fracture. Observation of fine structure by means of electron-microscope and of change in residual stress that inevitably occur during fatigue process have been adopted as the supplementary means for the better understanding of the finding in the change of half-value breadth.

Because of insufficient resolving power of ordinary back-reflection technique for the study of fatigue of engineering metallic materials, X-ray microbeam technique has recently been introduced. Thus it is expected that engineering metallic materials will reveal more clearly the feature of variation in micro-structure during fatigue by means of new technique and the understanding of the finding in the early studies would be supported by the observation of micro-structure.

2. Variation of Half-value Breadth in Fatigue of Metals

I-ray investigation of fatigue in metals have been made by many researchers. Almost all of them aimed to observe the variation of the form of spots on film reflected from annealed coarse grained materials before and after fatigue stressing. Although valuable findings on fatigue failure have been reported by these authors, there remains some question whether the findings would reveal average feature of whole grains in fatigue area. In this respect, the authors adopted a little different technique to attack the fatigue problem. The continuous interferential rings gives the average of the change in microstructure of a great number of minute crystal grains that contribute to the interference. The half-value breadth read from the interference line is taken as the characteristic measure of the average state of micro-structure. In the present study I-ray pattern were taken by the back reflection method, where X-ray was collimated by double pin hole of 0.5 to 1.0 mm in diameter. Both of microphotometry of photographic pattern and automatic recording by use of the Geiger counter unit were employed to obtain the interference profile from which the half-value breadth was read. Attention was paied for obtaining

continuous interference rings even from the annealed coarse grained materials, by scanning fatigued area by X-ray spot. Special equipment have been developed for this purpose.

Specimens were subjected mainly to rotary bending stress. The I-ray pattern were taken at various stages of the fatigue process until fracture occurs. In this manner, the variation of half-value breadth in fatigue process has been studied, and fatigue process is discussed in view of the variation in half-value breadth.

inumber of experiments have been made on various sorts of engineering metals, carbon steels of various carbon content, light metal and its alloys, and copper and its alloys. It has been found that the mode of change in half-value breadth during fatigue is quite regular and has a similar trend for annealed material, for work-hardened material and for heat-treated material, separately, independent of the sort of materials. In the followings the case of a carbon steel is presented as the example of annealed, work-hardened and heat-treated materials.

A 0.78 % carbon steel of 3 mm dia. was prepared for test specimen. The chemical composition and the mechanical properties are shown in Tablesl and 2. The specimens were prepared in three different ways:

- i) full annealed at 8500 in vacuum
- ii) 50 % cold drawn by drawing machine
- iii) quenched from 8500 in machine oil and tempered at 5000 for 30 min.

A 0.42 % carbon steel wire that was quenched from 8500 in machine oil without subsequent tempering was used as specimens for comparison with quenched material with subsequent tempering.

X-ray interferential rings from (310) atomic planes by Co-ka radiation were taken on film, where the distance between the films and surface of specimen was kept constant for all cases. The interferential rings for annealed materials are rather spotty as is shown in Fig. 1-a and so does not give reliable microphotometer curve. The specimens were therefore rotated around the axis once per 2 minutes in order to obtain continuous interferential ring (Fig. 1-b). Exposure time was 15 minutes for annealed or heat-treated materials and 40 minutes for cold-worked materials under the condition of 8 mm in tube current and 35 kV in secondary voltage.

(a) Annealed Material

Alternating bending stress of 25.5, 21.7, 18.9 and 15.0 kg/mm² were applied to specimens, where the endurance limit of the material was 18.0 kg/mm². Fig. 2 shows the relation between the half-value breadth and the number of stress cycles obtained as the result of experiment.

It is found that the half-value breadth increases remarkedly during the early cycles (the first stage of fatigue) and then increases gradually (the second stage of fatigue), then in the subsequent stage (the third stage of fatigue) the lines are greatly broadened until fracture occurs. It is worthy to note that the breadth reach a presumably definite value before fracturing. Repeating the stress under the endurance limit causes the variation of half-value breadth that is composed of the first and the second stages, missing the third stage, and the breadth reaches a limiting value that depends on the

magnitude of alternating stress in long run of testing.

(b) Cold-worked Materials

The experimental results on cold-worked material is shown in Fig. 3. Cold-worked material has a considerable amount of internal strain at initial condition, and the initial value of half-value breadth is very high. When the material is subjected to repeated stresses, the half-value breadth decreases markedly in very early period (during about 10th cycles) and then diminishes slowly. Near the number of cycles when macroscopic crack is suspected to appear the breadth begins to decrease notably, reaching a definite value just before fracture for all stress amplitude applied. Thus, in this case also, the process of diminution of half-value breadth is composed of three stages. In the case of diminution of breadth is missing and the breadth tend to reach a steady value dependent to the level of stress amplitude.

(c) Heated and Quenched materials

Two sorts of heat-treatment were applied to high carbon steels. 0.78 % carbon steel was quenched from 850°C in oil accompanied by subsequent tempering at 500°C for half an hour, and 0.42 % carbon steel was quenched without subsequent tempering. The results of experiments for both materials are exhibited in Figs. 4 and 5, respectively. In this series of study the Cr-Ka radiation was used, the (211) atomic plane being relevant to interference.

It may be found from the figures that the mode of variation of half-value breadth in the case of quenched and tempered material is similar to the case of cold-worked materials, being composed of three stages, although the diminution of breadth in the first stage is not so steep as compared with the latter case. In the case of quenched material without tempering, the mode of diminution in half-value breadth is a little different from the case with tempering. The characteristic feature is exaggeratedly exhibited in the case of stress amplitude below the endurance limit of the material. It is likely that the influence of some other factors affecting to the change of half-value breadth is superposed to the variation of breadth shown in Fig. 4. These results can be interpreted by taking account of the fact that the hardened steels generally contain a small amount of retained austenite and it is transformed to martensite by subjection to cyclic stresses.

The mode of variation of half-value breadth in fatigue process are characterized by the cases presented in the examples. It is probable that any sort of material has a character of half-value breadth versus number of cycles relation similar to any one of the preceding examples, as far as the authors have experienced.

3. Variation of Macroscopic Residual Stress and Hardness During Fatigue

Since the fatigue is the process of change in micro-structure caused by the application of cyclic stresses, it would be said that the fatigue process is that of variation of microscopic stress on strain. Summing up of micro-scopic stress in a area is measured as a macroscopic residual stress. In this meaning, it is probable that macroscopic residual stress that is present in a material would be varied by cyclic application of stresses. On the contrary information on the variation in macroscopic residual stress during fatigue might offer some instruction for the interpretation of the change in half-value breadth during fatigue.

I-ray diffraction is applicable for measuring the macroscopic residual stress. Recently, the reliability of the I-ray stress measurement has been very much improved by adopting newly developed instrument and introducing fine technique. Because of advantage of I-ray method in measuring residual stress non-destructively, the variation of macroscopic residual stress during fatigue was studied by I-ray method.

Two sorts of specimens were used for the study, the one is annealed 0.07 % carbon steel and the other cold-worked 0.07 % carbon steel. A rotating bending fatigue testing machine was used for the fatigue test (1700 cycles per minute). The fatigue limit for the annealed material and the cold-worked material were 17.5 and 21.0 kg/mm².

Residual stress was measured from (310) diffraction lines according to two exposure method. Specimens were demounted from the testing machine at several stages of the number of cycles and set in the diffractometer.

The results of the experiments are shown in Figs. 6 and 7, for annealed and cold-worked materials, respectively. In these figures, the variation of macroscopic residual stress is shown by solid lines and that of half-value breadth is shown by dotted lines for comparison. It is found from Fig. 6 on annealed material that the compressive residual stress appears at very early period of stress repetition and it increases until the maximum value is reached at about 2 X 10⁵. It is interesting to note that, looking at the mode of variation of half-value breadth in the same figure, the number of cycles at maximum value of residual stress is close to the transition from the first stage to the second stage of the variation of half-value breadth. After this the residual stress decreases steeply and thereafter its diminution proceeds gradually with number of cycles until fracture occurs. Under the stress amplitude below the fatigue limit, both changes of residual stress and the half-value breadth show similar appearance, that is, the residual stress tends to reach a value that is far lower than the maximum value.

In the case of cold-worked material (Fig. 7), the variation of residual stress has the trend similar to that of half-value breadth. Residual stress decreases notably in the early period of stress repetition and then diminishes gradually until fracture occurs. Thus the process of diminution of residual stress is composed of the first and the second stage, being coincident to those of variation in half-value breadth.

For reference to the observation on the variation of half-value breadth and that of residual stress during fatigue, variation of hardness was measured on the same material. The result is shown in Fig. 8, an annealed material, for an example. Notable increase of hardness in early period of stress cycling has a good correlation with the change of half-value breadth and residual stress in the first stage. Slight decrease in hardness at the early second stage and the steady hardness value thereafter is likely to give suggestion for the understanding of the mechanism of the second stage variation of half-value breadth.

4. Crystal Plane Dependence of the Change in Half-walue Breadth

In Section 2, the variation of half-value breadth during fatigue process was presented by taking the case of measurement on the diffraction from (310) atomic planes. Since fatigue is a process of change in microstructure caused by alternation of minute slip in crystals, the structure change would not be isotropic in crystals, because preferential slip would occur according to

relative orientation of slip plane to the direction of stress applied. As far as the X-ray diffraction is concerned with preferential plane relevant to the condition of diffraction, the qualitative as well as the quantitative variation of half-value variation process during fatigue would be different according to the atomic plane that reflects X-ray. In this connection, experiments were carried out to study the atomic plane dependence of the change in half-value breadth using 0.07 % carbon steel annealed plates, to which alternate bending stress (1700 cycles per minute) was applied.

X-ray diffractometer charged by the tube of chromium or cobalt target was employed. The (310), (220), (211) and (200) atomic planes were selected as the subject of study. The specimen was applied by alternating stress of 22kg/mm2 where the endurance limit of the materials was 18.5 kg/mm². The results obtained are exhibited in Fig. 9. It is found from the figure that the trend of variation of the half-value breadth for (310), (220) and (211) is more exaggerated than the other cases, while the half-value breadth of (110) atomic planes shows very little variation with number of cycles.

The dependence of the change in half-value breadth to crystallographic plane, that diffracts I-ray, during fatigue as shown in Fig. 8 would be interpreted by taking account of the nature of preferential slip of crystal in polycrystalline aggregate in relation with its orientation to applied stress.

The possibility of preferential slip of any crystallographic plane is estimated by the magnitude of the orientation factor μ defined as

where φ is the angle between the normal to slip plane and the stress axis, and ψ the angle between the slip direction and the stress axis. Let us consider the case of a crystal of which an atomic plane that is taken to diffract X-ray is parallel to the surface. Possibility of real slip of the crystal by a slip system would be estimated by taking the mean value of the orientation factor of active slip plane that belongs to the slip system, with respect to probable orientation of the crystal. Denoting the angle of the stress direction to the orientation of crystal as λ , it is given by

$$\bar{\mu} = \frac{1}{2\pi} \sum_{i=1}^{n} \left[\int_{\lambda_{i-1}}^{\lambda_{i}} \mu_{i} \, d\lambda \right],$$

where $\lambda_0 = 0$ and $\lambda_n = 2$.

We can calculate $\bar{\mu}$ of crystals with respect to one of the three slip systems [110], [211] and [321] when any one of atomic planes (200), (310), (211) and (110) is diffraction plane. Taking an atomic plane as the diffraction plane, $\bar{\mu}$ of crystal is different for different slip system. The value of $\bar{\mu}$ of crystals for the case of different diffraction planes with respect to any slip system is tabulated in Table 3. In the last column, the maximum of $\bar{\mu}$ for crystals with each diffraction plane is given. The order for the case of respective atomic plane as diffraction plane. It is interesting to note that $\bar{\mu}$ of crystals in the case of (200) as the diffraction plane is the largest and that in the case of (100) as diffraction plane is the smallest. (310), (211) and (110) that reflect X-ray have good correlation with the trend of variation in half-value breadth shown in Fig. 8.

5. Interpretation of the Variation of Half-value Breadth

In the preceding section, the interesting feature of half-value breadth during fatigue has been described. Since the half-value breadth of I-ray diffraction simply presents the change in crystal structure, its variation has to be interpreted by the change in crystal structure. A series of work has been done by the authors on the observation of micro-structure of fatigued materials in relation with the variation of half-value breadth and an understanding on the change of half-value breadth has been given as followings.

In the annealed materials, it is free from internal stress and so sharp interferential pattern of X-ray is given from this state of materials. When annealed materials are subjected to alternating stresses, work-hardening occurs in the crystal lattice, as a result of plastic deformation at an early stage of stress cycles. Since stress of a definite magnitude is applied repeatedly in a steady direction, the work-hardening saturates after a certain number of cycles. Steep increase of the half-value breadth of the first stage is interpreted in this way.

In the course of subsequent fatigue process, nucleation and growth of fatigue damage, sub-microscopic cracks, proceeds. Around the damaged material, micro-stress concentration are produced by repeating stressing. Micro-stress concentration supply the energy for growth of the fatigue damage. This process is taken as the second stage of fatigue. Thus the steady increase of half-value breadth in the second stage implies the combined influence of micro-stress and fatigue damage. When sub-microscopic crack develops into visible crack, the steady variation of breadth ceases and the rate of increase of half-value breadth increases notably. It is the third stage of variation.

In the case of cold-worked materials, fragmentation, internal strain and misorientation exist in crystal grain. The micro-stress in random direction would be in high potentiality. Combined effect of these factors gives the large value of initial half-value breadth. When fatigue stress is applied to these materials, relaxation of certain grains specified by the relative direction with alternating stress, the fatigue mechanism described in the case of annealed materials would take plane. This mechanism accompanies the relaxation of internal strain in other direction. Relaxation of internal strain yield in sharpening of half-value breadth.

Thus, in the case of fatigue of cold-worked materials, two opposite processes of variation in half-value breadth coexist. Since it is considered that the influence of the relaxation of micro-stress in random direction is predominant over that of the accumulation of fatigue damage in a specific direction, general trend of variation in half-value breadth appears as sharpening with cycling of stress. Thus the observed trend of the change in half-value breadth during fatigue in the case of work-hardened materials is interpreted. Similar consideration would be valid for the case of heat-treated materials.

The mechanism of fatigue in relation with the variation of half-value breadth is supported by the observation of micro-structure by optical and electron microscope in a series of experimentation, slthough detailed description is saved here.

Although the observation of structure by microscopes give us a number of information for understanding of the mechanism of fatigue, they are limited within qualitative instruction. In order to establish better understanding of mechanism of fatigue of engineering materials in conjunction with that of vari-

ation of half-value breadth, the fine technique of X-ray diffraction by 50 microns X-ray micro-beam has been employed. Diffraction of X-ray by 50 microns micro-beam offers the resolving power of 1 micron or less and we are able to observe the change of structure of individual grain in polycrystalline aggregates. Since improved X-ray source of micro-focus unit and the X-ray micro-camera that facilitates us to point out a very local spot on the surface of specimen are available, a 0.06 % carbon steel specimen was studied by means of the X-ray micro-beam technique on fatigue mechanism.

As far as crystal structure is not severely deformed, the diffraction of crystal by 50 micra micro-beam appears on film as small spots and we are able to have qualitative as well as quantitative information on the mechanism of deformation of crystals by observing the shape of spots and accounting the number of divided spots on film. Thus, the technique has the advantage to other method in giving the information on the size of fragmented crystalline, the order of misorientation, the magnitude of lattice strain and dislocation

In order to limit the area of fatigue, notched specimen of 0.47 mm in notch radius has been prepared and X-ray spot was directed on the local area of 0.07 mm distant from the notch root. The X-ray pattern is shown in Figs. 10-a and b, before and after fatigue, respectively. The enlarged photograph of a spot in the process of fatigue is exhibited in Fig. 11. We can see the mode of deformation of the shape of spots in relation with the deformation

According to the formulae that are originally presented by P. B. Hirsch 14), we can calculate misorientation $oldsymbol{\beta}$, lattice strained/d by measuring the change ast and as in the length of spot in the direction of tangent and normal of interferential ring on film, by using the formulae;

$$\rho = \frac{|\cos 2\theta|}{2 \sin \theta} \frac{\text{Ast}}{R_0} \text{ and } \frac{\text{Ad}}{d} = \frac{\cos^2 2\theta}{\tan \theta} \frac{\text{Asr}}{2R_0}$$

where θ is diffraction angle and R_o is the distance between film and the surface of specimen. Also we can know the number of subdevided cell structure

The result is presented in Fig. 12 in relation with the number of stress cycles. In the figure, observation on three spots appeared in Fig. 11 is presented. Looking at the figure, it is found that the mode of change in misorientation, lattice strain and number of subdevided cell is similar to the change in half-value breadth with number of cycles. Although it is not easy at present situation of the study to get quantitative correlation of finding on the change in micro-structure read from the micro-beam technique with that of half-value breadth by ordinary X-ray diffraction technique, the meaning of the change in half-value breadth is interpreted by the change in

6. Summary

Fatigue of engineering metallic materials are studied by X-ray diffraction by measuring the half-value breadth of interferential profile taken at several stages of stress cyclings. The results are presented on annealed, workhardened and heat-treated materials, taking the examples of carbon steels. It is found that the variation of the half-value breadth has a very regular

relation with number of stress cycles for each material; the profile of diffraction from annealed materials, being initially sharp, is broadened by fatigue stressing and that of work-hardened or heat-treated materials, being broad before stressing, is sharpened during fatigue. Both modes of variation of the half-value breadth has three subsequent stages. The variation of half-value breadth is interpreted in correlation with the change in micro-structure during fatigue, based on the information from observation by optical and electron microscope.

I-ray micro-beam technique is applied for the fatigue study of engineering metals to obtain further information of the change in micro-structure of crystal in polycrystalline aggregate. It was intended to support the understanding of fatigue mechanism by the information given by the X-ray fine technique. It is found that the trend of the change in half-value breadth has a good correlation with the finding in the micro-beam study.

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Table 1 Chemical Composition (%)

C	Mn	Si.	P	S	Sn	Cr	Fe
0.78	0.56	0.21	0.014	0.006	0.04	0.22	Bal.

Table 2 Mechanical Properties (kg/mm²)

	tensile strength	fatigue limit
cold-worked specimen	126 ~ 127	39.5
annealed specimen	led specimen — 18.	
heat treated specimen		22.5

Table 3 Values of Orientation Factor $\overline{\mu}$

diffraction	s	lip sys	max. value of		
plane	[110]	[211]	[321]	the three	
(200)	0.464	0.468	0.482	0.482	
(310)	0.416	0.469	0.358	0.469	
(211)	0.363	0.444	0.460	0.460	
(110)	0.401	0.433	0.435	0.435	

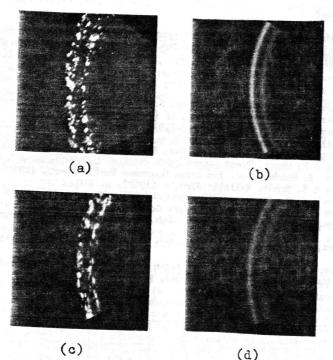


Fig.1 X-ray patterns of annealed 0.13%C steel specimen.

(a) Before fatigue test,

(c) After fatigue test (0=20 kg/mm², N=10⁵)

(d) , specimen was rotated.

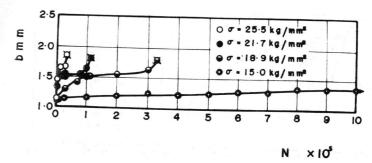


Fig.2 Relation between half-value breadth and number of stress cycles of annealed 0.78 % C steel specimens (6 w = 18.0 kg/mm²)

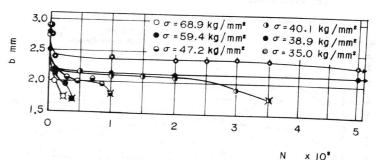


Fig.3 Relation between half-value breadth and number of stress cycles of cold-worked 0.78%C steel specimens (0 = 39.5 kg/mm²)

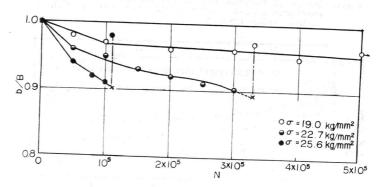


Fig.4 Relation between ratio of half-value breadth b/B and number of stress cycles N of 0.78 % C steel specimens quenched from 850°C in machine oil and tempered at 500°C for 1 hr (6w=22.0 kg/mm²)

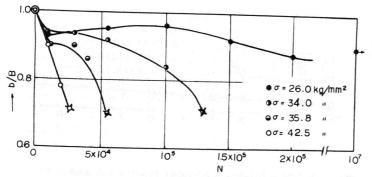


Fig.5 Relation between ratio of half-value breadth b/B and number of stress cycles N of 0.42 % C steel specimens quenched from 850°C in machine oil, without tempering (0 = 32.0 kg/nm²)

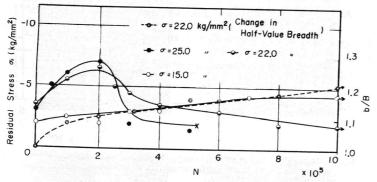


Fig. 6 Change in residual stress for annealed 0.07%C steel specimens (%=17.5 kg/mm²), comparing with the change in half-value breadth.

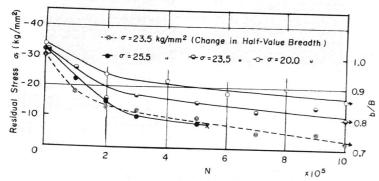


Fig.7 Change in residual stress for cold-worked 0.07%C steel specimens (δ_w =21.0 kg/mm²), comparing with the change in half-value breadth.

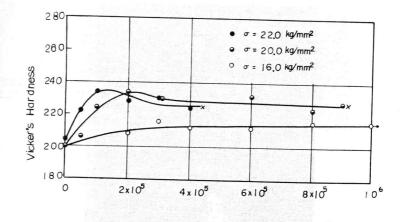


Fig.8 Change in Vicker's hardness for annealed 0.07 % C steel specimens (Cw = 18.5 kg/mm²)

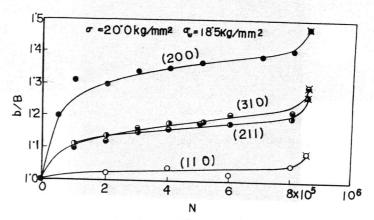
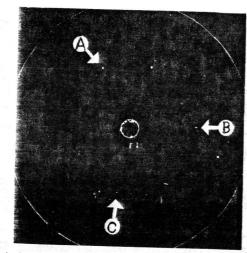
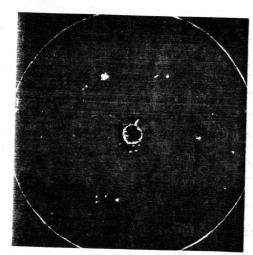


Fig. 9 Diffraction plane dependence of the change in halfvalue breadth with stress repetitions for annealed 0.07 % C steel specimen (% =18.5 kg/mm²).



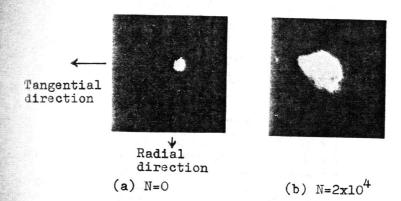
(a) N=0



(b) N=3x10⁵ (When a macroscopic crack was initiated)

Fig.10

Micro-beam X-ray diffraction patterns before (a) and after (b) fatigue testing (0.06 % C annealed steel). Diameter of X-ray beam : 50 \mu, Characteristic X-ray: Crka



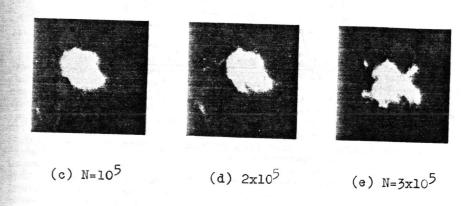


Fig.11 Change of diffraction spot A during fatigue process(enlarged).

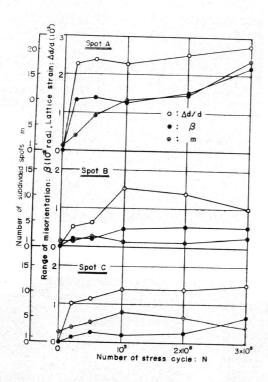


Fig.12 Changes in $\Delta d/d$, β and m (0.06%C annealed steel)