X-ray Investigation on the Fatigue Damage of Metals Containing α - and β -Phases

(On the Changes in Half-Value Breadth and Residual Stress of 6-4 Brass due to Stress Cycles)

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In the field of mechanical engineering, the need for a simple but effective way of evaluating the fatigue strength and lifetime of structural materials is an important problem with which the design engineers have faced. Accordingly, a considerable amount of investigations have been made in this field. However, the basic nature of fatigue damage and the conditions which lead to the initiation and propagation of fatigue cracks are not sufficiently understood. Nor any satisfactory method of assessing the exact state of fatigue damage has yet been found.

X-ray diffraction technique is not a new as the method of experimental study, but has often been adopted for direct and non-destructive observations of change in the local structures of crystalline materials at fundamental research. X-ray technique is one of the most powerful means to investigate the changes of microscopic-structure due to external forces, considering the fatigue phenomena occur on the surface of materials.

Accordingly, one of the authors have studied the relations of half-value breadth of X-ray diffraction lines, residual stresses and hardness and number of cycles of fatigue stresses in detail for various sorts of engineering metallic materials. In 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 stress cycles, the authors have reported that the lifetime of materials in fatigue could be predicted.

Consequently, it needs to investigate whether or not this predicting method is fitted for alloy containg α and β phase, moreover, it is interesting to make clear the fatigue mechanism of such materials which are present two phases having the different yield stress and type of crystal structure.

§1. Introduction

For the study of strength of materials, analysis and experiment must aim for the two different objectives. The analytical approach uses the laws of statics, dynamics and the theory of strength to establish the basis for calculation. The experiment determines the behavior of the material under various loads. It the case under consideration, the relevant processes within the cyclically stressed material cannot be fully explored through fatigue tests. Changes within the material, detectable by mechanical or magnetic means, are predominantly the effect of fatigue stress. X-ray and microscopic investigations indicate crystalline and structural changes, and process of fatigue of metal is discussed in the scope of order or disorder of atoms composing the materials. Namely, there are two quite different lines of attacking fatigue problems; the phenomenological and the metallographical or physical.

A great number of investigations have been made for the these lines of works. In practice, various criterions of materials have been proposed so far to predict the lifetime of structures subjecting to fatigue stressing. These criterions are in general derived basing on probable models of fatigue mechanism that are introduced from the finding in fundamental study in microscopic 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 microstructure 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 efforts in search of practical experimental means to find the change in micro-structure of engineering metals, enabling us to recognize properties of the fracture model.

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 observations of change in minute structure of crystalline materials at fundamental reseach. The authors considered that X-ray investigation is a powerful means to study the effect of repeated stressing on the fatigue damage of materials. Up to date numerous investigations have utilized it to study the extent of fatigue damage and also to search for the basic mechanism of fatigue, and many significant results of study are reported.

The authors have conducted so far a series of X-ray studies on fatigue with engineering metals, where the half-value breadth of X-ray diffraction lines was adopted as the measure of the changes in micro-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 numder of experiments, it has been found that the variation in half-value breadth showed very regular relation with number of stress cycles. It would be noteworthy to mention that the finding in this investigation promises an optimistic expectation of non-destructive detection of fatigue damage in principle and also of prediction of lifetime of materials in fatigue. In the other series of investigation, the authors have made discussion on the changes in half-value breadth in relation to the residual stress measured by X-ray technique or the basic mechanism of fatigue and suggested noticeable information to attack the fatigue problems in the future.

However, at present, it is clear that the values of half value breadth and residual stress are affected by the type of internal stress state seriously¹⁾⁻³⁾. For example, in steel, an iron carbide (Fe₃ C) lamella in ferrite may be in equilibrium with the matrix under a certain condition, that

is, when it is formed or annealed, but upon cooling room temperature or plastic deformation extremly high locked stresses develop in the surrounding matrix because of the difference in some elastic and plastic properties or thermal expansion coefficient. Really, the presence of so-called "Gefügespannungen" has been pointed out^{4), 5)}, and it is considered to be due to the difference in the yield stress of various phases coexisting in an alloy. Therefore, if we consider that the Gefügespannungen is caused by the progress of fatigue slip and the growth of microcracks in certain grains of the α -phase, they may contribute to the behaviour of the line broadening and the state of residual stress in the fatigue process.

For the reasons, in the present paper, the authors attempted to investigate the fatigue mechanism of metals containing α -and β -phases (6-4 brass) in relation to the Gefügespannungen and carried out some experiments on the changes in half-value breadth and residual stress due to stress repetitions using $(\alpha + \beta)$ brass.

§ 2. On the Changes in Half-Value Breadth and Its Crystal Plane Dependency in the α -and β -Phases due to Stress Repetitions

The specimens used in the series of experiments were annealed and electro-polished 6-4 $(\alpha + \beta)$ brass. The Wöhler type rotating bending fatigue testing machine was used for the fatigue test, and the speed of stress repetitions was about 2,000 cycles per minute. The fatigue limit which was decided at about 10^7 cycles of stress Dimentions of the specimen, 12.7 kg/mm^2 . chemical compositions and its mechanical properties are shown in Fig. 1, Table I and Table The half-value breadth was measured from II. (420) α -phase, (331) α -phase, (321) β -phase, (400) α -phase and (310) β -phase diffraction lines using $CuK\alpha$ and $Cok\alpha$ radiations, adopting the ordinary X-ray back reflection method at various stress cycles. The specimens were demounted from the fatigue testing machine at several stages of the number of the cycles, and set on the X-ray camera for the measurement of half-



Fig. 1 Dimensions of the specimen

Cu	Zn	Fe
60.07	39.89	0.04

Table I Chemical compositions

Yield Point	Tensile strength	Elongation
(kg/mm²)	(kg/mm²)	(%)
I 8. O	30.6	42.0

Table I Mechanical properties



Fig. 2 (a) Example of microphotometer curves using CoKα radiation (Measurement of half-value breadth)

value breadth and then its half-value breadth was measured. To obtain the X-ray diffraction intensity curve from the X-ray film, a new type of automatic microphotometer was used which

was specially designed for the stress measurement by X-ray. Fig. 2 (a), (b) and (c) show some example of the X-ray diffraction patterns and microphotometer curves in those experiments.

Experiments were performed to investigate the changes in half-value breadth diffracted the several atomic planes during the fatigue process in relation to the effects of the presence of two phases which held different yield strength, elastic constants and crystal structures. Several results of the experiments are shown in Figs. 3, 4,



Fig. 2 (b) Example of microphotometer curves using $CoK\alpha$ radiation

(Measurement of residual stress, $\psi = 0^{\circ}$)



Fig. 2 (c) Example of microphotometer curves using $CoK\alpha$ radiation (Measurement of residual stress, $\psi=25^{\circ}$)

5 and 6. In Figs. 3 and 4, the ratio of values in half-value breadth b/B, where B and b were the initial and current values of half-value breadth, during the stress cycles measured from various





ig.6 Change of half-value breadth vs. number of stress cycles

diffraction lines of α and β -phases using $CuK\alpha$ radiations are plotted against the number of stress cycles N at two different stress levels of 18.6 kg/mm² and 14.0 kg/mm² respectively. Figs. 5 and 6 show the results obtained by the same procedure as above mentioned using $CoK\alpha$ radiation. It is found from these figures that the value of half-value breadths of $(4.0)_{\alpha}$. phase, (331) a-phase, (321) B-phase (400) a-phase and (310) β -phase diffraction lines increase obviously in the early period of stress alternations, and in the subsequent stage they increase gradually until fracture occurs as was found in the previous investigations⁶⁾⁻⁹⁾. It is noticed from these results that the changes in half-value breadth diffracted from (321) β -phase and (310) β -phase atomic planes in β -phase are enormously greater than those of (420) α -phase, (331) α -phase and (400) α -phase atomic planes in α -phase which are very slight in the fatigue process. This is an interesting fact considered from the presence of Gefügespannungen as descrived in the preceding section. The b/B-log N plots were made in order to obtain the characteristic feature of the b/B-N curves. Figs. 7 and 8 show the results of this plots. From the relations between b/B and log N shown in Figs. 7 and 8, it can be seen that there is an approximately linear relationship. In addition, the dependency of diffraction lines which diffracted from crystal planes in α -phase of $(\alpha + \beta)$ brass is observed, but that of the changes in β -phase is not observed at all.

§ 3. On the Changes in Residual Stress due to Stress Repetitions

A number of investigations have been reported concerning the fundamental nature of residual stress measurement by Xrays in view of physical metallurgy. However, it is not thought that the results of these physical investigations are applicable to the fatigue fracture of industrial alloys containing two phases under service conditions. The authors have made a series of studies to find how Gefügespannungen might contribute to the fatigue fracture of alloys containing α and β -phases. Investigations have been made that object in view of the relation between the behaviours of residual stress and half-value breadth in the α -and β -phase under a cyclic stress.

The material used in these experiments was $(\alpha + \beta)$ brass as shown in Tables I and II. The specimens were annealed at 400°C for 2 hours and cooled in the air. The fatigue tests were carried out in the way as stated in the previous section, using Wöhler type fatigue testing machine. The residual stress was measured from (400) a-phase and diffraction lines (310) *a-phase* by $CoK\alpha$ radiation according to the $\sin^2\psi$ method of X-ray

stress measurement. For the determination of the peaks of diffraction intensity the automatic recording type microphotomater was employed. The X-ray diffraction patterns and microphotomater curves thus obtained are shown in Fig. 2 (a), (b) and (c). The results of the change in residual stress are shown in Figs. 9 and 10. As mentioned in the previous series of investigations it is observed that the fatigue process basically consists of three stages in the changes of half-value breadth. In the first stage of fatigue the compressive residual stress appeared in the α -phase, but in the β -phase



Fig. 8 Relation between b/B and log N

the tensile residual stress increased in this period, while in the second stage of fatigue, both the residual stresses in α -and β -phases respectively decreased steeply. The diminution proceeded gradually thereafter and finally the residual stresses remained until fracture occurred. It is very interesting to note, in the case of $(\alpha + \beta)$ brass, the tensile residual stress appeared in the β -phase, while the compressive residual stress was generated in the α -phase in the period of the first stage of fatigue process.



§ 4. Microscopic Observation of $(\alpha + \beta)$ Brass in Fatigue process.

A number of investigations have made efforts to examine the basic mechanism of fatigue phenomena from the standpoint of slip mechanism by observing them with optical and electronic microscope. Most of the fundamental works on the fatigue or plastic deformation of metals have been performed with single crystal specimens, so as to e iminate the complicated effects of grain boundaries and the restrains imposed by the neighbouring grains and the second phase particles. In the previous papers the authors investigated the appearance of the slip bands on the surface of the annealed low carbon steel during the stress alternations, and dis-

cussed the change in half-value breadth in order to understand better the physical meaning of the change in half-value breadth. In the present investigatoin the authors have studied the structure of the slip bands that had developed on the surface of the α -and β -grains during the reversed bending by means of an electronic microscope, so as to make certain the relation between behaviours of the residual stress and the half-value breadth in connection with the Gefügespannungen.

The experiments were carried out with the same material as in the case of previous experiments and stressed alternately under a constant stress amplitude $\sigma = 16.5 \text{ kg/mm}^2$ by the cantilever type plate bending fatigue testing machine. The specimens were annealed for 2 hrs. at 400°C before the fatigue test, and then polished electrically in an aqueous solusion of H_3PO_4 (60gr) -CrO₃ (25gr). At the several stages in fatigue process, the slip hand, existence of the deformation marking and microcracks etc. were observed in the regions of interphase of α and β .

Fig. 11 (a) - (f) shows some

examples of the formation of slip traces under the reversed stress above the fatigue limit of this material. Fig. 11 (a), (c) shows the regularly arranged slip lines in the special grains of α phase in the early stage of fatigue. They grow longer and wider with increase in the number of fatigue stress in the second stage of fatigue (Fig. 11 (d), (e)). Attention should be paid to the fact that the fatigue failure runs dominantly along the boundaries of α -and β -phase grains (Fig. 11 (f)). It is considered that the very presence of semi-microcracks on the boundaries proves the contribution of Gefügespannungen to the real fracture of fatigue in two-phase alloy.

The typical electronic micrographs at $N=5 \times 10^5$ are shown in Fig. 12 (a)-(d). These obser-



(c) N= 3.0×10^5

(f) N=1.0×106 (Just before fracture)

Fig. 11 Change of micro-structure $(\sigma = 16.5 \text{ kg/mm}^2, N_f = 1.0 \times 10^6) (\times 300)$

vations show that extrusion and intrution occur in initially annealed $(\alpha + \beta)$ brass in abundance along the slip bands in α -phase of certain grains during the cyclic deformation, and that remarkable fragmentation of crystal are observed on the boundary between α -and β -grains or its neighbourhood (Fig. 12 (a), (b), (d)).

§ 5. Summary and conclusions

Based on the results of these experiments, the following summary and conclusions may be drawn on the changes in half-value breadth and residual stress in the fatigue process.

(1) As described in the previous section, the behaviour in α -phase is metallographically very



(b) (\times 30000) (d) (\times 30000) Fig. 12 Observation of boundary of α -and β -phases by means of electron microscope (σ =16.5 kg/mm², N=5.0 \times 10⁵)

similar to that of the ferrit in carbon steel, and it is found that slip bands are observed in the special plane of certain α -grains in the very early stage of fatigue, and they become more intense with increase in the number of stress cycles. According to an examination by means of an electronic microscope it is clearly observed that these slip bands consist of wavy slip lines accompanied with extrusion and intrusion phenomena, and moreover, in the limited region of α -and β -grain boundaries the heterogeous deformation is observed remarkably. Presumably, it is considered that the boundary regions of β -grains are in a highly stress state enough to generate fatigue cracks. For these reasons, it may be thought that the general trend of changes in half-value breadth come from the increase of micro-stress due to fatigue slip which occurs predominantly in the most

favourable slip plane and direction of face-centered cubic lattice of α -grains, and that the great increase of half-value breadth of diffraction lines in β -phase is occasioned by the occrurence of fragmentations in the boundary regions of β -grains in thier neighbourhood¹¹⁾⁻¹³⁾.

(2) It is well known in general that if one considers on a large scale of the behaviour of a bar subjected to plastic bending, the characteristic pattern of residual stress is obtained on the release of the load, that is, the tensile and the compressive residual stresses appear respectively either side of the bar. The surface crystals have been plastically deformed and they are loaded in a state of macro-residual stress due to the restraint of the elastic portions below the surface.

So far as an alloy consisting of α -and β -phases is concerned, the phases are subject to external

loading like macro-stress pattarns. It must be emphasized that plastic deformation proceeded in α -phase owing to the effect of elastic strength. and the plastically deformed α -phase should be elongated in the same derection as that of the loading. Hence, in the β -phase of the same material, there exists no noticeable plastic deformation except in the boundary regions. and the elastic stress state is preserved in the inner part of β -phase as it was at the time of loading. The surface of the β -grains which were elongated elastically in the longitudinal direction by bending remains in a state of compressive stress when he external load is removed. For this reasons in the β grains there are combined effects of the tensile stress due to compressive residual stress in the α -grains and the contructive stress of β -grains themselves. Cousidering the sign of residual stress in β -phase due to the magnitude of these effects, the tensile residual stress should be present.

When we consider these arguments in relation to the faigue slip mechanism are above mentioned, the appearance of the compressive residual stress in α -phase is reasonable. It causes tensile stress to rise in α -phase.

The residual stress both in α -and β -phases generated in such a way as above-mentioned decreases in the second stage during the fatigue process. The authors interpret these decrements to be due to the relaxation of residual streses owing to the appearance of micro-cracks in α phases.

(3) Concerning the cryatal plane dependency of the changes in diffraction line widths diffracted from α -crystals, it is concluded that the general aspect of the changes in half-value breadth for the (400), (420) and (331) diffraction lines during the stress repetitions is the same as that for the annealed carbon steel as was pointed out in the previous papers. On the other hand, the changes in interference line breadth diffracted from (310) and (321) crystal planes of β phase have shown good agreement during the fatigue process. In other words it is concluded that the crystal plane dependency of the changes in diffraction line widths do not exist in the β -phase. When a face-centered cubic lattice such as α -phase of Cu-Zn alloys is bent, glides take place on the equally stressed pairs of {111} crystal planes and each glide plane contains the direction in common. The observation of slip trace shows that fatigue slip bands produced on the surface of the repeated bending specimens as mentioned in the proceeding paragraph, despite the small number of cycles, have some of the characteristic features (intrusions and extrusions) observed on the surface of the α -grains. As mentioned in the pervious paper, it is suggested that the crystal plane dependency of diffraction line widths in the α -phase is related with such slip mechanism of face-centered cubic metals in the fatigue process.

On the other hand, the non crystal plane dependency on the changes in diffraction line widths of β -phase is considered from the fact that the heterogeneous deformation (fragmentation) and intensified slip hand owing to stress concentration proceeds predomiuantly near the boundary layers of β -grains, and that the inner parts of the β -grains are occupied with elastic deformation exclusively, from the fact that the stress determined by using various diffraction lines of X-ray coincide with the applied mechanical stress in the elastic region.^{14), 15)}Consequently, no crystal plane dependency is expected to appear in β -phase.

(4) Crystal lattice plane dependency in α phase and non dependency in β -bhase are interpreted as above mentioned. However, to make clear the crystal lattice plane dependency in α phase is necessary to investigate fatigue mechanism of $(\alpha + \beta)$ brass. As described in previous paper, the most common mechanism of vielding in crystalline materials is slip, in which two planes of stoms slip part each other, causing one whole section of the crystal to shift relative to another, slip occurs most easily on certain crystallographic planes, depending on the crystal structure. Generally speaking, the slip planes of easy slip are those in which the atoms are the most closely spaced-those having the largest number of atom per unit area. That these planes are also the most widely separated accounts for the comparative ease of slip. In face centercubiced crystal of α -phase in $(\alpha + \beta)$ brass, the slip plane is the {111} plane, the direction of easy slip is <110>.

For the plastic deformation of a crystal, only the component of the shear stress τ , in the slip p'ane along the slip direction is of importance. This shear stress is the result of the relation $\tau = \mu \sigma$, where σ represents the nominal stress and μ is the orientation facter. The facter μ depends only on the position of the activated slip_system relative to the direction of stress and is, therefore, a purely geometric quantity, and factor μ indicate which of the slip systems will be activated with a given orientation of the crystal and how the yield points $\sigma_s = \tau_k/\mu$ depends on the orientation.

In this connection, an interpretation is presented on the diffraction plane dependence of the change in half-value breadth, which is based on the idea that each crystal in the polycrystalline aggregates is particularly fitted for slip according to its orientation. In the discussion, it is assumed that the diffraction planes lie in parallel with the specimen surface according to the experimental condition of the X-ray diffracted by severely slipped crystals have large line broadening, while those by unslipped crystals remain as sharp.

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

$\mu = \cos \phi \times \cos \alpha^{16}$

where ϕ is the angle between the normal to slip plane and the stress axis, and α is the angle between the slip direction and the stress axis (see Fig. 13). Let us consider the case of a crystal of which an stomic 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 taken the mean value of the orien-



 orientation factor μ = cosφx cos a
φ : the angle between slip plane normal and stress axis
a: the angle between slip direction

and stress axis

$$\begin{array}{c} [hkl] & --- [400]: 90^{\circ} \lambda \\ [hkl] & --- [040]: \lambda \\ [hkl] & --- [004]: 90^{\circ} \end{array} \ (hkl] + [sin\lambda, cos\lambda, o] \\ [hkl] & --- [004]: 90^{\circ} \end{array}$$

Fig. 13 The relation between stress, slip plone and slip direction

tation factor of active slip plane that belongs to the slip system, which 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

$$\overline{\mu} = \frac{1}{2\pi} \sum_{i=1}^{n} \left[\int_{\lambda i-1}^{\lambda i} \mu_i d\lambda \right]^{17}$$

where $\lambda_o = 0$ and $\lambda_n = 2\pi$

Using this principle, the authors calculated the values for each (400), (420) and (331) diffraction plane, respectively (see μ column of Table IV).

Comparing with the calculating orientation factors μ and crystal plane dependency of the changes in half-value breadth of each diffraction plane during fatigue process, the authors knew that the crystal plane dependency in diffraction line widths could not be explained by means of μ only.

For the reasons, the authors conidered about the difference between mechanisms of plastic deformation and fatigue damage, and mechanism of crack initiation was introduced. Namely the authors bear a close resemblance the fatigue fracture to the cleavage one, and assume that crack initiates on the most close-packed crystal plane (FCC: (111)), consider the change of halfvalue dreadth of diffraction lines adopting the experimental resalt by Deruyttere, Greenough¹⁸⁾ and a model on crack initiation by Mott, Stroh¹⁹⁾.

Fig. 14 shows the relation between crystal



Fig. 14 Relation between the fracture stress and crystal orientation in Zn single crystal (Deruyttere & Greenough)

(400)

orientation and fracture stress in Zn single crystal. From this result, when the angle between stress axis and the most close-packed crystal plane is 50° fracture stress value is minimum.

diffraction plane

In other words, in this condition the fructure have good chance.

Now, the authors caluculate the angles between stress axis which fit for experimental con-

> dition (Normal to the direction of diffraction plane) of each Xray diffraction planes (400), (420) and (331) in α -phase obtained in this experiment and fracture plane of face-entered cubic crystal (111). As seen in Table III, the authors adopt only low index as to stress axis. From this table, in three diffraction planes the values which are close to 50° are (400): 53°11', (420): 50°46' and (331): 54°44' (see λ column of Table IV).

> Additionally, Fig. 15 shows a crack nucleation model by Mott and Stroh. In this figure, the piled-up dislocations front a obstacle generate a large stress. Mott considers that tensile stress due to piled-up dislocations will nucleate a micro-crack

as shown in Fig. 15 (a), the dislocation rushs into the crack, Δ type crack are built up. A precise calculation by Stroh makes clear that normal stress σ becomes maximum in the case that θ is equal to 70°30' in Fig. 15 (b). Therefore, the authors calculated the angle between each diffraction planes and (111) which is slip plane, these results are shown in the θ column of Table IV.

According to experimental result by Deruyttere et al. and the model by Mott et al., it is consider that diffraction line broadening of (420)



Fig. 15 A group of edge dislocation piled up at on obstacle (Mott and Stroh)

crystal plane is remarkable compare with that of (331) crystal plane.

From those consideration, crystal plane dependency of half-value breadth in diffraction lines in α -phase of $(\alpha + \beta)$ brass under fatigue process is interpreted. That is, its remarkable change



Table IIIAngles between the direction of stressaxis and the cleavage plane



	μ	λ	θ
(400)	0.464	53°11'	54°44'
(420)	0.366	50°46'	39° 14'
(720)	0.566		75° 02'
(331)	0 368	54°44'	21°59' 48°32' 82°23'

μ : orientation factor

- λ : angle between direction of stress axis and cleavage plane
- heta : angle between diffraction plane and (111) plane

Table IV list of orientation factor, angle between direction of stress axis and cleavage plane and angle between diffraction plane and (111) plane in (400) is effected that its diffraction plane has large value of orientation factor. On the other hand, the value of orientation factor in (420) diffraction plane is equal to one of (331) crystal plane. However, (420) crystal plane has good chance as to cracking. Accordingly, it is concluded that the line broadening is extreme in (420) diffraction plane compare with (331).

In the above discussion, the authors interpreted crystal plane dependency of diffraction line broadening in α -phase of $(\alpha + \beta)$ brass in connection with deformation (fracture) mechanism.

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