AN X-RAY STUDY OF LATTICE STRAIN VARIATION IN LOW CARBON STEEL

by

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ABSTRACT

Simultaneous measurements of lattice [elastic] strain by the x-ray line shift method and total strain with an electrical strain gauge have been carried out on SAE 1010 steel with the help of a special tensometer-attachment to the X-ray diffractometer.

The particular features investigated are: The variation of the lattice strain and the total strain with applied stress above the limit of proportionality, the effect [in detail] of the mode of unloading on the lattice strain and the total strain and the contribution of the intergranular stresses and the stacking faults to X-ray line shifts.

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1. INTRODUCTION AND OBJECTIVE

Careful experiments have led to the recognition of residual lattice strains [RLS] in deformed polycrystalline aggregates. The early observations of this effect have been reported by Wever and Pfarr[1] in 1933, Bollenrath et al[2] in 1939, and Smith and Wood[3,4] in 1941-42.

Various explanations[5] have been advanced to explain the lattice strain effects accompanying the plastic deformation of polycrystals. The basic principle common to all these is as follows. It is assumed that different parts of the aggregate have different yield stresses. Assuming that part A yields at much lower applied stress than part B, subsequent to the process of deformation, the elastic strain in A will be less than that in B. On removal of the applied stress, B will tend to contract further than A but will be prevented from doing so by the restraining influence of A. The state of final equilibrium is such that A is in compression and B in tension. The argument can be extended to a large number of different parts. While this reasoning is tacitly accepted in principle, there appears to be a controversy as to the exact nature of parts A and B.

Bollenrath and his co-workers[2] have plastically extended a mild steel specimen and have observed the residual lattice strain at surfaces successively exposed by etching away layers of the metal. They point out that grains in the free surface of a polycrystalline metal are less restrained than those in the interior and that the former on the average should have a lower yield stress than the latter.

This view is supported to some extent by Glocker and Hasenmaier[6], and contradicted by Smith and Wood[7].

Greenough[5][based on an earlier interpretation by Heyn and Masing] has proposed that different grains would have different yield stresses depending on their orientations. They have found that the intergranular stresses [Heyn intergranular stresses] do make a large contribution to the observed residual lattice strains. However, it is presumed that there must be, in addition, some other effect such as a surface macroscopic stress since all the observed strains are more positive than anticipated. Further, while it is agreed that the residual lattice strain in crystals of one orientation would vary as the orientations of the neighbouring crystals varied, it is pointed out that the average strain taken over many grains of the particular orientation would not be zero, but would depend on the difference of the yield stress of the grain with the given orientation and the average yield stress of the aggregate. Experimental evidences in favour of this argument have been given for iron and steel by Garrod[8] and by Finch[9].

Newton and Vacher[10] have studied residual lattice strains in sectioned bars of plastically deformed iron, and their observations seem to be more in line with the suggestions that the highly distorted material at or near crystal boundaries is the harder part rather than the suggestion that the harder part is an adjacent crystal of different orientation. However, it is pointed by Donachie and Norton[11] that residual lattice strains do not arise in any detectable manner from a macroscopic stess system, and they are not solely,

or in major part, the result of an intergranular stress system.

Garrod and Hawkes[12] have investigated the possible contributory processes to the residual lattice strains in iron and mild steel. They do not find any evidence for significant intergranular system of Heyn stresses. In addition, they believe that no single mechanism could be invoked to explain the results. According to them at least three and possibly four processes are significant in the case of mild steel.

A macroscopic surface effect[2] is expected to result from the different yield stresses of the surface and the interior of a polycrystalline metal. A modification of the above concept suggests that in the plastic range, the surface layer will on the average harden less rapidly than the core[13,14]. Whereas, however, the surface effect should produce a surface stress which is independent of the amount of plastic deformation, the stress due to the hardening effect should increase with increasing deformation. If the material contains more than one phase with different yield strengths, a pseudo-macroscopic effect[15] results. For the X-ray measurements on a given reflection from one phase would indicate a macroscopic stress in that phase due to this cause.

Garrod and Hawkes[11] suggested a model based on the hypothesis originally advanced by Smith and Wood[7] in which the residual lattice strains were attributed to the high-strength grain boundaries. After heavy deformation, each grain in the polycrystalline aggregate is composed of a number of polycrystals with mis-orientations of a few degrees between neighbours and with a non-uniform distribution of dislocations. On this model, the boundaries between particles

are not narrow discontinuities in the lattice but wider volumes in which concentrated dislocation pile-ups, associated with intersecting slip bands or walls of screw dislocations, form highly distorted regions which are presumably principal sites of the strain-hardening observed in cold-worked material. It is suggested by Garrod and Hawkes[12] that these regions act as a harder phase than the interior of the particles and produce a pseudo-macroscopic stress system analogous to the inter-phase effect suggested by Hauk[15].

Bragg, Orowan, Smith and Wood[16] have suggested a model based on the possibility that during the process of plastic deformation, the accumulation of dislocations around obstacles to their propagation through the crystal would cause higher stresses in the region of accumulation of dislocations than in the remaining bulk of the crystal. After removal of the applied stress, these regions of accumulated dislocations are left in a state of tensile stress, these stresses being balanced by those in the bulk of the crystal remote from the dislocations.

Auld and Greenough[17] have found that single crystals of iron do not show residual lattice strains after plastic extension. This is interpreted as showing that the residual lattice strains observed in polycrystalline iron are due to an intergranular stress system rather than due to stresses associated with trapped dislocations.

It should be observed that several workers[5] have assumed that the residual lattice strain observed in unloaded specimens is related to the lack of proportionality between lattice strain and applied stress in the region above the yield stress.

While attempting to draw a distinction between the regions of opposing stresses, it has been observed by C. J. Newton[18], and more recently by Cullity[19], that the regions of high density of dislocations could constitute the harder regions as opposed to the softer regions which could as well be the interior of grains. Keh and Weissmann[20] have produced a series of micrographs showing the progressive changes which occur during the plastic deformation of iron. As plastic deformation proceeds, more and more dislocations appear ultimately culminating in a tagled web of dislocations that delineate a cell structure. Cullity identifies the walls of these cells with regions subjected to a large tensile stress and the subgrains having a nearly constant and a very low dislocation density with regions stressed more or less uniformly in compression. A similar proposal has also been made by Garrod and Coyle[21].

Recently, Swaroop and Tangri[22] have performed simultaneous measurements of total strain in the direction of pulling and lattice strain normal to the direction of pulling on polycrystalline nickel with the help of a special tensometer attachment for the X-ray diffractometer. They have found that, during the initial stages of deformation, the rate of increase in lattice strain closely follows the total strain until the plastic strain sets in. From then onwards, the two strains have been found to deviate from each other, the rate of increase in lattice strain eventually decreasing with increase in applied stress. The specimens pulled to a total strain within the proportionality limit have been found to show no residual lattice or total strains. Furthermore, depending on the mode of unloading, both

compressive and tensile strains have been observed up to a deformation of 0.29% strain. Specimens gradually unloaded over a period of time have been found to show residual lattice strains of a compressive nature, while those instantaneously unloaded showed residual lattice strains of a tensile nature which reverted to a compressive nature on aging at room temperature. These results have been explained on the basis of effects of clustering of dislocations and also the production and behavior of point defects during loading and unloading respectively.

While the above review depicts the various attempts that have been made with a view to elucidate the nature and the origin of the residual lattice strain, no clear understanding has developed yet as to the behavior of lattice strain effects above the limit of proportionality. Furthermore, Swaroop and Tangri[22] have drawn attention to the nature of the lattice strain during and after gradual unloading, and also to the variation in the residual lattice strain with time in the specimens that have been instantaneously unloaded subsequent to deformation to various extents of total strain.

It was pointed out that several authors[18,19,21,22] have tried to relate the origin of the residual lattice strain to the dislocation substructures produced during deformation, and these are supposed to constitute regions of stress opposite in sign to the coherently diffracting domains which essentially form the bulk of the material. An investigation of such a mechanism calls for a study of X-ray background intensity with deformation which is due to the incoherently scattering domains such as the grain boundaries or tangles of dislocations.

Under these circumstances, the objectives of the present investigation are:

(a) To ascertain the behavior of the lattice strain up to about 1.35% strain, with particular interest in the behavior above the limit of proportionality, and explain the phenomenon by an appropriate hypothesis.

(b) To know the nature of the lattice strain during the process of gradual unloading.

(c) To know the stress at which the residual lattice strain originates and to study the variation in the residual lattice strain with time subsequent to instantaneous unloading.

(d) To study the variation in the X-ray background intensity, if any, and relate them to (a).

(e) To investigate into the role of stacking faults or the stem system associated with the origin of the residual lattice strain.

2. EXPERIMENTAL PROCEDURES

2.1 Properties of the Material in the As-supplied Condition.

The material for investigation was SAE 1010 steel received in the form of sheets which were aluminium killed, cold rolled and annealed with the following chemical analysis.

TABLE 1.

Chemical Analysis of the Specimen.

Element	Contents %
Carbon	0.09
Manganese	0.37
Phosphorus	0.004
Sulphur	0.043
Silicon	0.14
Copper	traces

Calculations were made to determine the relative amounts of the various phases present in the material and it was found that 90.02% of ferrite and 9.98% of pearlite were present. Of this amount of pearlite, 1.23% constituted cementite. Quantitative metallographic techniques using the method of point counting and the method of lineal analysis were found to yield almost similar values for the various phases present.

2.2 Specimen Preparation

2.2.1. Thermo-mechanical Treatments

The sheet in the as-supplied condition was cut into strips 2 inches long and 0.625 inches wide. These strips were cold-rolled down to 0.017 inch thickness and annealed at 850°C for two hours. Subsequently, an additional cold-rolling was performed, the resulting thickness of the strips being 0.014 inch. The strips were machined to give specimens [fig. 1] of required dimensions, subsequently annealed at 850°C for one hour. All the annealing operations were preceded by encapsulation of the specimens in silica tube evacuated and sealed in argon at a pressure of 1×10^{-5} Torr.

2.2.2. Electropolishing for X-ray Diffraction

Both the surfaces of the specimens were lightly polished by hand to four zeros emery papers to remove any surface blemishes and then cleaned in acetone. The specimens were then electropolished in the following solution at 15°C [to about 0.010" thickness]:

10% Orthophosphoric acid [70% concentration]

90% Glacial acetic acid

The best polishing conditions were obtained at a potential of 10-12 volts D.C. and a current of 0.6 ampere with constant stirring. The final rinsing was very critical, staining was avoided by quickly rinsing in two separate ethyl alcohol baths and preserving in acetone until use.

Five of the annealed specimens taken at random were electropolished and etched in 2% nital. The specimens were found to be fully recrystallized with an average ferritic grain size of 10.45×10^{-4} cm.

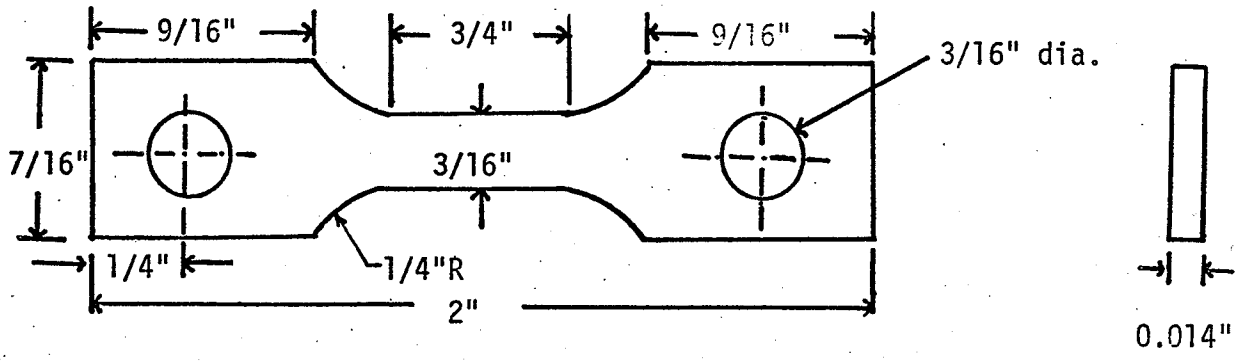

Fig. 1. Tensile Test Specimen.

as determined by planimetric method.

2.3 Tensometer-attachment for the X-ray Diffractometer.

A tensometer-attachment [figs. 2 and 3] was specially designed for the X-ray diffractometer. One of the ends of the specimen is fastened to a fixed grip, while the other is fastened to a movable grip which moves under the influence of a direct load. Thus, the essential advantage of the device is that it facilitates direct uni-axial loading and unloading of the specimen, thereby obviating the requirement of a calibrating device.

The following measurements could be made with this arrangement:

(a) The applied stress by the weights directly added on a pan connected to the movable grip through a cord.

(b) The lattice strain, e_z in a direction perpendicular to the direction of pulling [x-direction] from shifts in the X-ray peak positions [fig. 4].

(c) The total strain, ε_x in the direction of pulling, with the help of an electrical strain gauge [Budd C6-121A] affixed to the back of the specimen directly below the area irradiated by the X-rays. [The electrical strain gauge used was temperature compensated and extreme caution was exercised in the measurement of the total strain as suggested by Carnahan and White[23].]

2.4 X-ray Diffraction Procedures.

2.4.1. Measurement of Lattice Strain.

The calculated values for reflection angles for α-iron

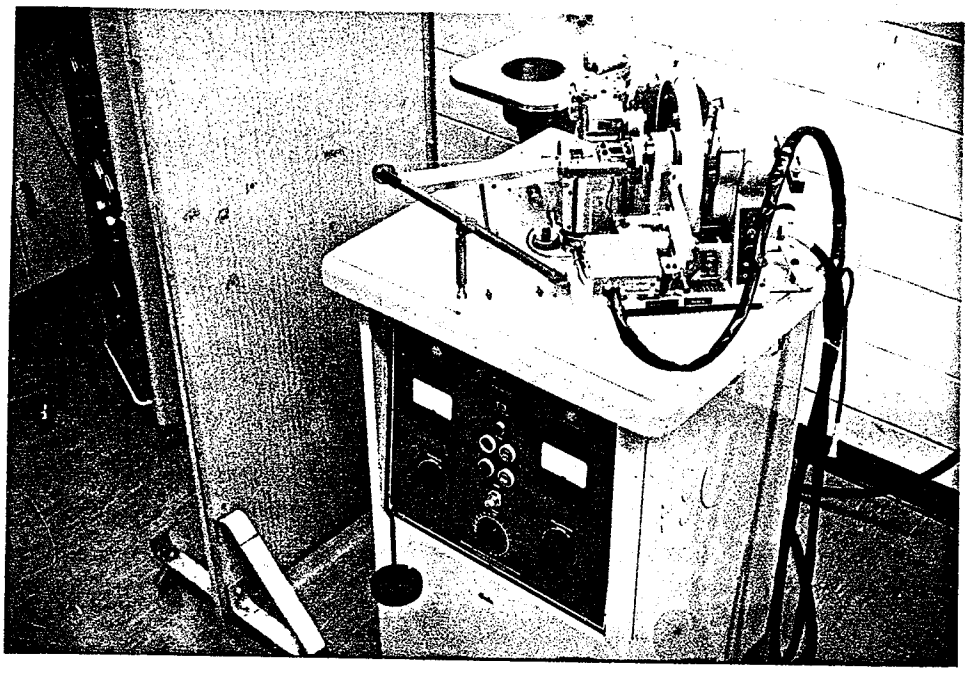

Fig. 2. X-ray Diffractometer with the tensometer-attachment including the loading device.

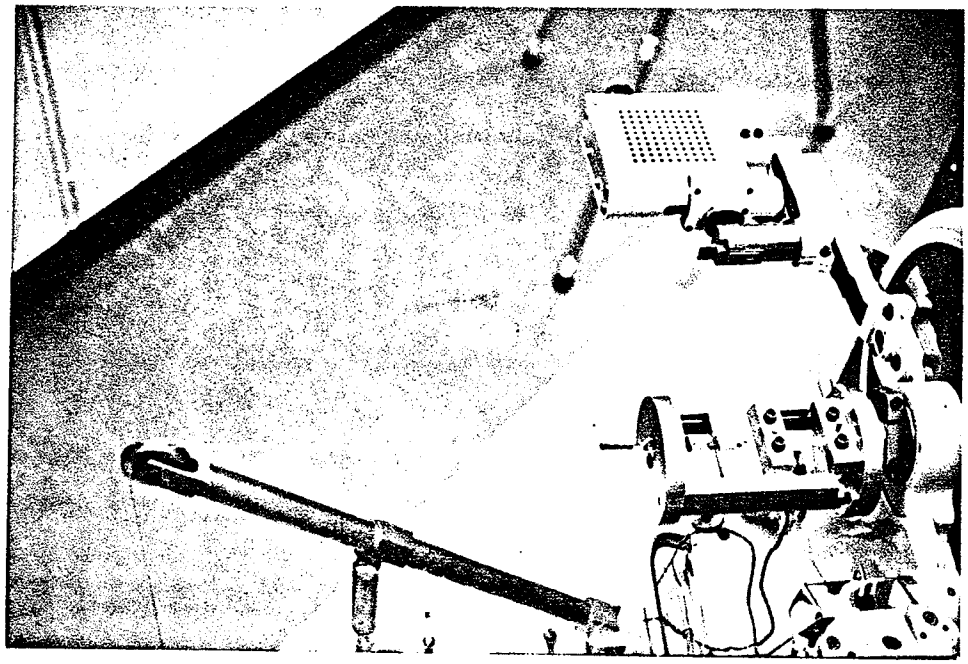

Fig. 3. Tensometer-attachment to the X-ray Diffractometer.

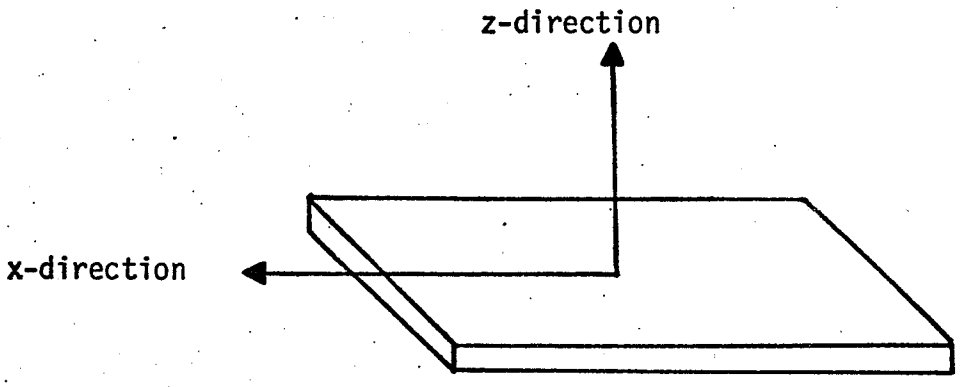

Fig. 4. Directions of measurement of lattice strain and total strain.

ε_x: Total strain measured in x-direction (the direction of the tensile axis).

e_z: Lattice strain measured in z-direction.

e_x: Lattice strain in x-direction (calculated using Poisson's ratio).

with cobalt $K\alpha_1$ and chromium $K\alpha_1$ radiations are given in Table 2.

TABLE 2.

Calculated values for reflection angles.

Reflection (hkl)	Interplanar spacing dA°	Bragg angle, degrees	
		$CoK\alpha_1$	$CrK\alpha_1$
(110)	2.0268	52.4	68.8
(200)	1.4332	77.2	106.0
(211)	1.1702	99.7	156.1
(220)	1.0134	123.9	--
(310)	0.9064	161.32	--
(222)	0.8275	--	--

The (211) and (310) reflections were chosen for investigation with $CrK\alpha_1$ and $CoK\alpha_1$ radiations respectively. The (222) reflection was very weak and therefore was not chosen for experimentation. Inverse-intensity measurements were carried out at known angular increments across the peak in consideration. The reflections from cementite were extremely weak to be of any significance.

In order to determine the interplanar spacings "d" to a precision adequate for the measurement of lattice strain, the 2θ angles of the diffracted rays have to be measured accurately. Several workers[24,25,26] have tried to establish methods for accurate

determination of the peak positions. In Ogilvie's parabola fitting method[25], five data points are obtained at equal 2θ intervals about the diffraction peak, and the parabolic curve is fitted by the method of least squares. Koistinen and Marburger[26,28] greatly simplified the computation procedure and the operation took less time by reducing the number of data points to three. The important limitation of this method, hoever, is that it depends on the line symmetry, and special attention has been drawn to this fact by Werner[5].

If the three points recorded correspond to at least 85% of the maximum intensity and these points straddle the peak of the diffraction curve, some lack of symmetry can be tolerated and the parabola will usually be a good approximation.

A long-range continuous scan of each peak preceded the step-scanning procedure. This was done essentially to establish the range over which the peak was to be scanned step-wise.

The peak positions were determined by parabola fitting following Koristinen and Marburger.[26,28] According to this method, we have :

$$2\theta \text{ vertex} = 2\theta_1 + c\ [(3a + b)/(a + b)]$$

where $a = t_1 - t_2$

$b = t_3 - t_4$

t_1, t_2 and t_3 are the time required to accumulate given number of counts at $2\theta_1$, $2\theta_2$ and $2\theta_3$.

$2\theta_1$, $2\theta_2$ and $2\theta_3$ are the consecutive 2θ positions at which inverse intensities are measured; and $c = 2\theta_2 - 2\theta_1$ or $2\theta_3 - 2\theta_2$.

The limit of accuracy in measurements of the change in