X-ray Diffraction Study of Residual Macrostresses in Shot-peened and Fatigued 4130 Steel

Object of investigation is to evaluate the magnitude of the compressive residual stress relaxation at the surface and the redistribution of residual stresses at subsurface levels after an increasing number of fatigue cycles

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ABSTRACT-A study has been made of the effects of shot peening and fatigue cycling on the residual macrostresses determined by X-ray methods in an austenitized and tempered AISI 4130 steel (150-170 ksi). The results show that the effect of shot peening is to produce a residual compressive macrostress layer 0.014-in. deep. The residual-stress profile (stress vs. depth) exhibits a small negative stress gradient at and near the surface and a large positive stress gradient in the interior. Stress relaxation (due to fatigue cycling) which occurred early in the fatigue history of the specimen was found greater at the surface than in the subsurface layers. Stress gradients of the stress profile increased with continued cycling and varied with depth. A correlation appears to exist between stress relaxation and stress gradients at the surface.

Introduction

The surface condition of a material may be very important in determining its fatigue characteristics. For example, the shot peening of metal surfaces is known to induce compressive stresses at and below the surface¹⁻³ resulting in an enhancement of fatigue life⁴⁻⁶ and fatigue limit⁷⁻⁹ which increases with increasing compressive stress.⁶ It has also been reported that fatigue mechanisms alter the structure of materials at or near the surface. ¹⁰ This observation may be related to reports^{11, 12} that surface, residual, compressive stresses are relieved as the number of fatigue cycles increases, thereby suggesting that, even before a crack is started, fatigue life is continually decreasing from its potential as the number of fatigue cycles increases. The relief of compressive residual stresses by fatigue processes affects the stress-corrosion resistance of the material. Since stress corrosion is known to occur in the presence of a suitable chemical environment and an applied tensile stress,¹³ the conversion of compressive to tensile residual stresses at or near the surface by fatigue cycling can be a critical factor in applications where both cyclic loading conditions and a corrosive environment are present.¹⁴⁻¹⁵

This investigation was undertaken to evaluate the magnitude of the compressive residual-stress relaxation at the surface and the redistribution of residual stresses at subsurface levels after an increasing number of fatigue cycles. Since a nondestructive and reproducible method of measuring surface residual strains is by X-rays, an X-raydiffractometer technique was used in examining the shot-peened and fatigued specimens.

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NOTE : ALL DIMENSIONS IN INCHES Fig. 1—Fatigue-specimen configurations for AISI 4130



B. Ψ=45°

Fig. 2—Schematic diagram showing orientations of the peened surface (of fatigued specimen) relative to the incident X-ray beam at Bragg angle, 2θ and glancing cycles: (A) $\psi = 0$ deg and (B) $\psi = 45$ deg. N and N_{ψ} are normals, respectively, to specimen surface and Bragg plane

Experimental Procedure

Specimen Preparation

The material utilized for this investigation was an AISI 4130 steel heat treated to a tensile strength of 150-170 ksi. The chemical composition of the alloy is shown in Table 1. Fluctuating tension fatigue-test specimens ($8 \times 1.0 \times .125$ -in. thick)) with a gage width of 0.5 in. were machined from a single sheet according to the configuration of Fig. 1. After machining, the specimens were austenitized at 1575° F for 1/2 hr, quenched in oil, tempered at 950° F for 1/2 hr, and cooled in air. Rectangular bend-test specimens $(1.0 \times 0.675 \times 0.125 \text{ in.})$ for determining the X-ray stress factors of 4130 were also prepared from sheet stock. Both fatigue and X-ray stress-factor samples were machined to an RMS 32 finish after austenitizing and tempering treatments.

The gage sections of all the fatigue specimens were peened (on one side only) with cast steel shot (0.033-in. diam) to an intensity of 0.020 A as measured on an Almen No. 2 dial gage. A Peen-amatic machine with a $\frac{3}{6}$ -in. nozzle was used and operated at 95-psi air pressure.

The shot-peened 4130 samples were fatigued in fluctuating tension on a Wiedemann-Baldwin fatigue tester subject to a minimum-to-maximum stress ratio of R = 0.06. To establish an approximate S-N curve, nine shot-peened samples were tested to failure.

To prepare samples for residual-stress analysis by X-ray diffraction, seven shot-peened (0.020 Å intensity) 4130 samples were fatigued in tension-tension under applied stresses of $S_{\min} = 4.38$ ksi and $S_{\max} = 7.30$ ksi. These samples were cycled individually to values of n(number of cycles) = 28, 59, 88, 118, 178, 298 and 475 kc, which correspond respectively, to cycle ratios of $(n/N_f) = 0.05, 0.10, 0.15, 0.20, 0.30, 0.50, 0.80$ assuming N_f as the approximate fatigue life of the sample. These samples are designated, respectively, F1, F2, F3, F4, F5, F6 and F7 (see Table 2). One shot-peened sample (F0) was left uncycled for comparison purposes.

X-ray-diffraction Method

Residual-macrostrain measurements were made on a Siemens Crystalloflex-IV X-ray diffractometer using $\operatorname{CrK}_{\alpha}$ radiation (35 kv, 14 ma) filtered with a vanadium foil. The fatigue specimens were mounted on a variable- Ψ holder of the Siemens stress-attachment unit. A Geiger-Müller (argon filled) detector was used to record the (211) martensite peak. In the two-exposure X-ray-diffractometer method, a high angle ($2\theta > 100 \text{ deg}$) Bragg peak is examined at two angles of incidence (relative to the specimen normal), $\psi = 0 \text{ deg and } \psi = \Psi$ (Fig. 2). For a flat specimen subjected to uniaxial tensile

TABLE 1—CHEMICAL COMPOSITION OF AISI 4130 STEEL

Element	Wt. %
C	0.31
Mn	0.54
Р	0.018
S	0.016
Si	0.31
Cr	0.88
Mo	0.23
Ni	0.09
V	0.006
Cu	0.15
AI	0.023
Sn	0.012

TABLE	2-FATIGUI	E DATA FO	R SHOT-PEEN	ED 4130
STEEL	EXAMINED	BY X-RAY	DIFFRACTION	(CrK _a : (211))

Alloy	AISI 4130 (150-170 ksi)
Applied tensile stress:	
Minimum	S _{Min} = 4.38 ksi
Maximum	S _{Max.} = 73 ksi
Fatigue limit (approxim	ate)S _f = 73 ksi
Fatigue life (approxima	te)N _f = 590,000 cycles
	No of evolor Cycle ratio

Sample No.	n(Kc)	n/Nr
F0	0	
F1	28	0.05
F2	59	0.10
F3	88	0.15
F4	118	0.20
F5	178	0.30
F6	298	0.50
F7	475	0.80

stress, the recorded peak shift, $\Delta 2\theta_{o-45} = 2\theta_o - 2\theta_{\psi}$ is proportional to the strains normal to the specimen surface, and related to the stress in the longitudinal direction (parallel to the specimen length) by:

$$\sigma_L = K(2\theta_o - 2\theta_{\Psi}) \tag{1}$$

where the stress factor, K, a function of Ψ , half the Bragg angle, θ , and the elastic constants, E (Young's modulus), and ν (Poisson's ratio) is given by¹⁶:

$$K = \cot \theta \left[\frac{E}{2(1+\nu)} \cdot \frac{1}{\sin^2 \Psi} \right]$$
(2)

The calculated longitudinal X-ray lattice stress, σ_L , may be an applied stress, σ_L^A , or a residual stress, σ_L^R . Present results are expressed in terms of σ_L^R ($= \sigma_r$).

The stress factor was determined experimentally by recording the peak shifts, $\Delta 2\theta_{o-\Psi}$, resulting from known elastic stresses (calculated from strain-gage measurements) applied to U-bend specimens. By this method, a nearly linear relationship is obtained between the X-ray peak shifts, $\Delta 2\theta_{o-\Psi}$, and the applied (mechanical) macrostress, $S_A = E\epsilon$, where E is Young's modulus, and ϵ is the observed strain from strain-gage measurements.

The peak positions were determined using the three-point-parabola method of Koistinen and Marburger.^{17, 18} This method consists of determining the peak position by calculating the parabola axis from three recorded intensity (or inverse intensity) values. Intensities chosen were 85 percent of maximum intensity and were corrected for Lorentz-polarization effects and for specimen absorption. Reproducibility of the peak shift based on standard deviation measurements was $\Delta 2\theta_{\sigma-45} = \pm 0.03^{\circ}2\theta$, which corresponds to a precision of ± 2600 psi in the X-ray residual-stress measurement.

To examine the residual macrostresses at the peened and fatigued surfaces, X-ray peak shift measurements at $\Psi = 0$ deg and $\Psi = 45$ deg were taken from the center gage section of the control specimen F0 (shot peened, not fatigued) and the seven peened and fatigued specimens designated



Fig. 3—Flow diagram of CDC-6600 computer program for calculation of residual macrostresses from X-ray peak shifts. Final correction of residual stresses for stress relief due to surface removal is done on a separate computer program

F1, F2, F3, F4, F5, F6 and F7. To determine the residual-stress profile (residual stress vs. depth) of the eight specimens, surface layers (at the peened center-gage section) with an area approximately ${}^{3}/{}_{8} \times {}^{15}/{}_{32}$ in. were removed successively in steps of 0.001 in. by etching in an aqueous nitric-hydrochloric acid solution (20 ml HNO₃, 10 ml HCl, 35 ml H₂O). After each layer removal, residual-strain measurements by X-ray diffraction were made.

To take into account the stress relief due to surface removal, the correction (for flat-plate geometry) suggested by Moore and Evans¹⁹ was applied to the calculated residual macrostresses at various specimen depths. The corrected longitudinal X-ray stress σ_L^{Corr} is given by:

$$\sigma_L^{Corr} = \sigma_L^{Obs}(t_i) + 2 \int_{t_i}^T \frac{\sigma_L^{Obs}(t)dt}{t} - \frac{6t_i \int_{t_i}^T \frac{\sigma_L^{Obs}(t)dt}{t^2}}{(3)}$$

where σ_L^{Obs} is the residual stress calculated from strains observed at a depth dt from the top surface of a plate with original thickness, T, varying thickness t, and thickness t_i remaining after removal of the *i*th layer.

A computer program in FORTRAN for the CDC-6600 was drawn up to facilitate the calculation of residual stresses from the observed peak shifts. Its input consists of the chosen intensity values, the corresponding 2θ positions, the Ψ -angles and the X-ray stress factor. The output prints out the 2θ values corrected for absorption and the Lorentzpolarization effect, the peak shift, $\Delta 2\theta_{\sigma-\Psi}$ and the residual stress, σ_r in psi units. The observed (uncorrected) residual stresses were corrected for stress relief due to removed layers by a separate computer program based on eq (3). A flow diagram of the computer operations appears in Fig. 3.

Results and Discussion

X-ray Stress Factor

An important quantity in the calculation of residual macrostresses is the stress factor K in eq (2). Since this quantity varies with alloy composition and heat treatment, 20, 21 it is best that K be determined experimentally, as was done here. The experimental points used in determining the X-ray stress factor for 4130 steel are shown in Fig. 4. These points represent peak shifts due to elastic bending stresses applied to the calibration samples. Deforming the sample beyond the elastic region resulted in a deviation (not shown) from the linear plot in Fig. 4. From the slope of this initial linear portion (drawn by a least-squares fit), the X-ray stress factor of $K_{\alpha-45} = 87 \text{ ksi}/^{\circ}2\theta$ was obtained. This value, which was used in calculating the residual stresses from the peak-shift measurements, is not too far removed from a theoretical value of 86.3 ksi/°2 θ obtained from eq (2) based on values of Young's modulus, $E = 30 \times 10^6$ psi, Poisson's ratio, $\nu = 0.29, \theta (CrK_{\alpha}) = 78 \deg \text{ and } \Psi = 45 \deg.$

Although in the present case there is close agreement between the experimental and theoretical



Fig. 4-X-ray stress factor for 4130 steel (150-170 ksi)

values of the stress factor, K, results from other studies²² show that there can be wide discrepancies between the two values of K. Hence, for the present study, the value of K was determined experimentally. The stress factor in eq (2) is, to a certain extent, also dependent on $\cot \theta$. However, the nearly linear relationship in the elastic region of $\Delta 2\theta$ with applied stress (Fig. 4) indicates that the variation of $\cot \theta$ with applied stress is negligibly small (less than 1/2 percent). Hence, $\cot \theta$ may be considered constant for the particular alloy steel being studied here, although more significant variations have been reported for nonferrous alloys.²³

Fatigue Cycling and Relaxation of Residuai Macrostresses

The curve for applied stress vs. cycles to failure (S-N curve) for shot-peened 4130 steel is shown in Fig. 5. Because of the limited number of specimens cycled to failure, only approximate values can be assigned to the fatigue limit (73 ksi) and the fatigue life (590,000 cycles) of the 4130 samples. Based on these values, seven shot-peened samples were cycled in tension-tension to various fractions of the approximate fatigue life. These cycles are indicated by open circles in Fig. 5 for the seven samples examined for residual stresses by X-ray diffraction.

Figures 6 through 9 show the residual-macrostress profiles (stress vs. depth) of the seven shot-peened and fatigued samples. The stress profile of the control sample, F0 (shot peened but not fatigued) is shown in Fig. 6 as F0' (dotted line) before stressrelief corrections were introduced, and as F0 (solid line) after corrections based on eq (3) were applied to "as-measured" residual-macrostress values.

A major effect of cycling on the specimen is the relaxation or decrease in residual compressive macrostresses at the surface. After cycling sample F1 to 28 kc (Fig. 6) a decrease (relative to F0) of 10 ksi was observed at the surface (d = 0). After



Fig. 5—4130 steel fatigued to failure (solid circles) in fluctuating tension-tension. Samples examined by X-rays were fatigued to number of cycles indicated by open circles



Fig. 6—Variation of residual macrostress with depth (stress profile) in 4130 steel. The effect of correcting for stress relief due to surface removal is shown by FO' (uncorrected curve) and FO (corrected). All curves in subsequent figures have been corrected for stress relief. Curve F1 is the stress profile for a sample shot-peened and fatigued 28,000 cycles. Residual macrostrain measurements were based on shifts of (211) Bragg peak using CrK α radiation



Fig. 7—Variation of residual macrostress with depth in shot-peened 4130 samples fatigued to 59,000 and 88,000 cycles

cycling sample F2 to 59 kc and F3 to 88 kc (Fig. 7), macrostress relaxations at the surface of 20 and 25 ksi, respectively, were noted. This relaxation at the surface increased to 30 ksi for samples F4 and F5 (Fig. 8), which were cycled to 112 and 178 kc,



Fig. 8—Variation of residual macrostress with depth in shot-peened 4130 steel fatigued to 118,000 and 178,000 cycles



Fig. 9—Variation of residual macrostress with depth in shot-peened 4130 steel fatigued to 298,000 and 475,000 cycles

respectively. Continued cycling to 298 and 475 kc resulted in residual-macrostress relaxations at the surface of 25 and 22 ksi for samples F6 and F7, respectively (Fig. 9). Combining the residual-macrostress profiles into one composite curve (Fig. 10) shows that the overall effect of fatigue cycling on the stress profile is a relaxation band of about 25 kis in width.

While the residual macrostresses do change with cycling, the degree of change varies with depth





(Fig. 11). At the surface (solid circles, Fig. 11) the relaxation is quite marked (about 30 ksi after 88 kc), while near the surface (d = 0.001 in.) after 88 kc, the relaxation is 15 ksi. At the profile minimum (d = 0.007 in.) the residual-macrostress decrease is 8 ksi after 88 kc. At the cross-over point (d = 0.013 in.) or depth at which the residual compressive macrostresses become tensile, the decrease is 5 ksi after 88 kc, and at d = 0.015 in. (depth where residual macrostresses are mostly tensile), the decrease is 8 ksi. These results indicate that residual-macrostress relaxation, $\Delta \sigma_{r}$, depends on both the cycling, n, and the specimen depth, d, so that



Fig. 11—Variation of residual macrostresses in 4130 steel with fatigue cycling at various depths: at the surface (solid circles), near the surface (at depth, d = .001 in.), at the stress profile minimum (d = .007), at the cross-over point (d = .013 in.), and at a depth below the cross-over point (d = .013 in.)

 $\Delta \sigma_r$ may be defined relative to the uncycled specimen F0 as:

$$\Delta \sigma_r = |\sigma_r^{FO}(d)| - |\sigma_r^{FN}(d,n)|$$
(4)

where $\sigma_r^{FN}(d,n)$ is the residual stress of the fatigued specimen, FN (with $N = 1, 2, \ldots, 7$, the sample numbers). A plot of $\Delta \sigma_r$, vs. number of cycles, n(Fig. 12) shows that maximum residual-macrostress relaxation occurs at the specimen surface and that this relaxation is from two to three times that at a depth near the surface (d = 0.001 in). Relaxation at other depths appears fairly constant with fatigue cycling, except at the cross-over point (d = 0.013)



Fig. 12—Relaxation of residual macrostresses, $\Delta \delta_r$ (in ksi) at various depths with fatigue cycling



Fig. 13—Changes in the residual stress gradient, $(d\sigma_r/dt)$, in ksi/.001 in, at various depths with fatigue cycling

in.) where the relaxation increases with the number of cycles. Figure 12 also shows that relaxation at the surface occurs early (after 28 kc) in the history of the fatigue process, increases to a maximum at about one-fifth of the approximate fatigue life after which $\Delta \sigma_r$ appears to remain constant or decrease slightly.

These results indicate that after fatiguing in tension-tension, the surface layers lose their protective compressive residual stresses early in the fatigue cycling and more readily than those in the subsurface layers. This behavior may be closely related to observations from transmission electron microscopy that the surface is easily damaged by fatigue cycling²⁴ and that fatigue cracks frequently nucleate at the surface.¹¹

Residual-stress Gradients

The residual-compressive-macrostress profile due to shot peening exhibits two regions with different residual-stress gradients. $d\sigma_r/dt$ (in ksi/0.001 in.) where t (= d) is specimen depth. Region 1 near the surface has a small negative gradient while Region 2, deeper inside the specimen, has a large positive gradient which is about five times the first region gradient (see Fig. 6). The two-gradient profile appears to retain its basic shape even after fatigue cycling (see Fig. 10). By arbitrarily defining the first region gradient as the slope of the profile measured at the surface and the second region gradient as the slope of the curve at the cross-over point (depth at which compressive residual stresses become tensile), a relationship can be found between the stress gradients at these two regions and the number of fatigue cycles.

Figure 13 shows that the negative stress gradients of Region 1 at the surface (solid circles) increase in magnitude with increasing number of cycles to a maximum at 178 kc (F5). The positive stress gradients at Region 2 (unfilled triangles, Fig. 13) also increase with increasing number of cycles to a maximum at 88 kc (F3), after which $d\sigma_r$ dt decreases. At intermediate depths (d = 0.001 and 0.007 in.) and beyond the cross-over point (d =0.015 in.), only slight changes in the stress gradient resulted from the fatigue cycling.

These results indicate that, for a fixed appliedstress amplitude, both $\Delta \sigma_r$ and $d\sigma_r/dt$ are functions of specimen depth, d, and number of cycles, n. At the surface, an increasing negative residual-macrostress gradient is accompanied by an increase in residual-compressive-macrostress relaxation. Near the surface (d = 0.001 in.) and at various depths (d = 0.007, 0.013, 0.015 in.), the relationship between $\Delta \sigma_r$ and $d\sigma_r/dt$ is almost linear. It appears therefore that at the surface, the larger the stress gradient, the more easily the residual stress will be relaxed. This could imply (pending direct experimental evidence) that the smaller the gradient, the more difficult will be the stress relaxation. If this effect were true, then a practical application would be to introduce on the fatigue-specimen surface residual compressive macrostresses with small negative stress gradients, so as to protect the specimen from early fatigue damage.

Other investigators have found that the amount of relaxation occurring in martensitic steels is a function of their heat treatment,^{11, 25, 26} with the amount of relaxation being less pronounced for harder materials. The 4130 specimens used in this study were of an intermediate hardness (<Rc50) so that the observed relaxations are moderate compared to those occurring in lower-hardness steels. The amount of relaxation is also a function of the amplitude of the applied cyclic stress.¹² In this study, since the fatigue limit was not firmly established, it is estimated that the applied stress was between 50 to 70 percent of the fatigue limit, so that the resulting stress relaxation was not as pronounced as might have been observed. Stress amplitudes of over 90 percent of the fatigue limit have been applied to steels of similar hardness and conversion of surface residual stresses from compressive to tensile has been reported.12

Although the precise mechanism of residualmacrostrain relief is not known, relaxation of distorted-lattice planes may account for the observed peak shifts. Since the peaks were not only shifted but also broadened, microstrains associated with dislocation-cell structures and fragmented coherently diffracting domains would also be present. Relief of microstrains arising from dislocation arrays can take place according to various proposed mechanisms such as rearrangement or annihilation of dislocations, annealing by point defects, and relaxation of dislocations with high-energy configurations.27 Jogs produced during fatigue cycling²⁸ are good sources of point defects which may participate in the relaxation process. However, the exact contribution, if any, of microstrain relief to residualmacrostrain relief still requires further study.

Summary

1. The effect of shot peening on AISI 4130 is to produce a residual compressive-stress layer 0.014in. deep. The residual-stress profile exhibits a small (-8 ksi/0.001 in.) negative stress gradient near the surface and a large (+34 ksi/0.001 in.) positive stress gradient in the interior.

2. Fatigue cycling: (1) reduced the magnitude of the residual compressive stresses at the surface induced by the shot peening, and (2) increased the stress gradients at the surface.

3. The observed macrostress relaxation occurred early (after 28 kc) in the fatigue history of the specimen. Both the macrostress relaxation and changes in stress gradients were found to depend on cycling and specimen depth. These quantities were larger at the surface than in the subsurface layers and attained maximum values early in the cycling process, after which the values remained nearly constant with cycling. In the subsurface layers, the macrostress relaxation and stressgradient changes were relatively smaller and remained almost constant with cycling.

4. A direct correlation appears to exist between residual-stress relaxation at the surface and the residual-stress gradient. Such a relationship implies that residual stresses with large stress gradients are easily relaxed.

5. Since fatigue life and stress-corrosion resistance are favored by compressive residual stresses at the surface, the relaxation or conversion of compressive stresses to tensile becomes significant when predicting the service life of applied materials. Although the present method of investigating stress relaxation in cold-worked and cycled specimens has

been applied to steels, its use can be profitably extended to other alloys such as aluminum or titanium, which, in some applications, are protected by shot peening against fatigue and stress-corrosion failures.

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