A Versatile Automated System for X-Ray Stress Measurement*

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A versatile automated system for X-ray stress measurement, using a microcomputer, has been developed on the basis of conventional techniques and the fundamental study on X-ray stress measurement using the Gaussian curve method previously proposed by the author. The simultaneous control of the three stepper motors on the goniometer by three motor controllers allows both rapid measurement and the use of various measuring methods. The most appropriate method for each material is programmable in this system. Residual stress values of a hardened steel were measured by the iso- and side-inclination methods using the fixed time and fixed count methods. They fell within a scatter band of the measured values. The η oscillation method proved to be most useful for the stress measurement of coarse grained steels. The system can also be used for evaluating diffraction line width using the Gaussian curve parameter.

Key Words: Experimental Stress Analysis, Automatic Control, Microcomputer, X-Ray Stress Analyzer, X-Ray Diffraction, Residual Stress, Nondestructive Test, Steels

1. Introduction

X-ray diffraction techniques are widely used for crystal structure analysis and materials evaluation. Kurita et al. have recently proposed new X-ray diffraction techniques for materials evaluation: the Gaussian curve method⁽¹⁾⁻⁽³⁾ which can rapidly measure even residual stress of hardened steels having broad diffraction profiles, the method for hardness measurement of quenched and tempered steels⁽⁴⁾⁽⁵⁾ and plastic strain measurement⁽⁶⁾ with a Gaussian curve parameter (GCP)⁽⁴⁾, and the method for evaluating hardening of quenched steels with GCP⁽⁷⁾.

In applying these methods, however, we encounter the problem that a measurement system for these

methods is not commercially available. Although an automated X-ray stress analyzer with a microcomputer has recently become available on the market, it runs according to a fixed pre-programmed scheme and is not programmable. Since data processing is different for each method, a programmable analyzer is needed.

To automate the measurement using a microcomputer, the control of an X-ray goniometer and a scaler-timer is necessary together with computer data processing; the hardware (control circuits) to do this must be developed. The software to control the system can not be developed without knowing what electric circuits are to be controlled because they are intimately associated. The use of an inexpensive and highly efficient computer and large-scale integration devices (LSI), which have been rapidly developed recently, would allow the development of a system for rapid and precise measurement that has not been available until now. Today, research and development of measurement techniques using X-ray diffraction requires studies from both aspects of software and hardware.

The present study is carried out to develop a

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versatile automated X-ray measurement system that is applicable not only to stress measurement but also to the evaluation of various materials by making use of a goniometer installed in a commercial X-ray stress analyzer. The system is based on conventional X-ray diffraction techniques and the fundamental theoretical study on measurement by X-ray diffraction proposed by the authors^{(1)~(9)}.

In a previous work⁽⁸⁾, the authors designed an automated X-ray stress analyzer with a microcomputer. In the present investigation, however, a more versatile X-ray measurement system has been developed, making use of a highly efficient microcomputer and LSIs, which allow simplification of the hardware to improve the reliability of the system. Applications of this system are also shown.

2. Construction of the Measurement System

Figure 1 shows the configuration of the measurement system which is composed of an X-ray goniometer, X-ray generator, I/O unit, and a microcomputer with its peripheral units. Figure 2 shows a block diagram of the system.

The goniometer used in this study was made by a company different from the one in the previous work⁽⁸⁾. It has three stepper motors (SM) as shown in Fig.2. The 2θ -stepper motor (2θ -SM) drives an X-ray

detector to change the diffraction angle $x (=2\theta)$. The other two stepper motors, ψ 1-SM and ψ 2-SM, revolve the goniometer head around the irradiated area on the specimen surface to change the X-ray incident angle ϕ_0 or angle ψ between the specimen and diffraction plane normals. It ψ 1-SM is used for the iso-inclination method and rotates the goniometer head in the scanning direction of the X-ray detector. The ψ 2-SM, which is installed on the main axis of the goniometer, is used for the side-inclination method and rotates the goniometer head perpendicular to the plane of the direction of scanning of the X-ray detector. Table 1 shows the specifications of the system.

3. I/O Unit

Figure 3 shows a detailed block diagram of the I/O unit shown in Fig.2. This unit includes a scaler-timer to measure X-ray intensity and drivers and controllers of the three stepper motors (SM) to drive the X-ray detector and the goniometer head.

3.1 Scaler-timer

X-Ray pulses from the X-ray detector enter the scaler-timer through the pulse-height analyzer (PHA) as shown in Fig.2.⁽⁸⁾ The scaler-timer was made from two LSIs of a six digit universal counter in order to simplify the circuits and to improve their reliability.

The FTM/FCM selector shown in Fig.3 is the circuit to select the fixed time and fixed count methods. In the fixed time method, X-ray pulses enter counter 2 through the FTM/FCM selector and the number of X-ray pulses is counted. At the same time, clock pulses generated by a clock generator enter counter 1 through the selector and the time is measured. In this case, counters 1 and 2 work as a timer

Table 1 Specifications of automated X-ray measurement system

2θ stepper motor	Minimum step width Scanning speed	0.01° $0.6^\circ/\text{s}$
ψ 1 stepper motor	Minimum step width Scanning speed	0.01° $1.0^\circ/\text{s}$
ψ 2 stepper motor	Minimum step width Scanning speed	0.01° $0.5^\circ/\text{s}$
Scaler and timer	Maximum X-ray counts Maximum X-ray intensity Preset time Preset counts	10^6 counts 10^6 cps 1 to 10^3 s 1 to 10^6 counts

Fig. 1 Configuration of the automated X-ray measurement system

Fig. 2 Block diagram of X-ray measurement system

and a scaler, respectively.

In the fixed count method, on the other hand, X-ray pulses and clock pulses enter counters 1 and 2, respectively, and the time required to count the fixed counts (preset counts) is measured.

Counter 1 starts counting X-ray photons when it has received preset time (p.t.) or preset count (p.c.) data and the FTM/FCM selector has received a FTM/FCM select signal and a count start signal. Having finished the counting, counter 1 issues a count end signal to counter 2 and at the same time a count end interrupt signal to the microcomputer. Upon receiving the signal, the microcomputer drives 2θ -SM to advance the X-ray detector by one step width according to an interrupt service routine and starts counting X-rays again. By repeating this, a diffraction line profile is obtained.

To improve the accuracy of X-ray counting, the scaler-timer is provided with a circuit to establish timing interrelationships between the count start signal and the clock pulses.

3.2 Stepper motor controller and driver

This system has three LSIs of a stepper motor controller in order to control simultaneously the three stepper motors used in this system. The simultaneous control of the three stepper motors allows not only rapid measurement but also measurement by the various methods shown in Fig.4, such as the oscillation method (γ oscillation method) and the fixed ϕ method (SIM-fixed ϕ method)⁽¹⁰⁾ using the side-inclination method (SIM) which are useful for the stress measurement of coarse grained materials.

The stepper motor controllers are isolated electrically from the driver circuits by using photo-couplers

Fig. 3 Block diagram of I/O unit (scaler/timer and stepper motor controllers)

Fig. 4 Methods for X-ray stress measurement practicable with this system

to avoid noise interference from the driver circuits.

4. Computer Program for Stress Measurement

The driving of the stepper motors and the counting of X-ray photons with the scaler-timer are manipulated in the interrupt service routine in order to reduce measurement time. The main program, which carries out data processing, is written in BASIC which is easier to write.

With this system, the peak position of a diffraction line profile can be determined by either the Gaussian curve method⁽¹⁾ or the half-width method⁽⁹⁾. For each method shown in Fig.4, the programs for the counting of X-ray photons and the SM control are standardized and described in a ROM, which can be accessed from the main program written in BASIC. Figure 5 is a flowchart for X-ray stress measurement by the Gaussian curve method, since it is common to all the methods shown in Fig.4, the iso-inclination method only will be explained in detail. The values in parentheses in Fig.5 are for the fixed count method.

The goniometer used in this study is designed to move the X-ray detector in the direction of decreasing diffraction angle when recording a diffraction profile. The goniometer head is tilted by 30° when a specimen is set. At step ① in Fig.5, the measurement system is initialized and the stepper motors (SM) drive the X-ray detector to the maximum diffraction angle x of 170° and the goniometer head to the X-ray incident angle ψ_0 of 30°, where datum points of x and ψ_0 are set. The data of preset time (p.t.) (or preset count p.c.), step width c , and a starting angular point x_0 are input at step ③ in Fig.5.

As soon as an interrupt signal is given between steps ④ and ⑥, the main program is interrupted to

Fig. 5 A flowchart of X-ray stress measurement by the Gaussian curve method

transfer to the interrupt service routine. Having started running the SMs and counting X-rays in the interrupt service routine, the microcomputer returns again to the main program and continues until it is interrupted by a SM stop signal or a count end signal. The use of the interrupt service routine to control the three SMs and to count X-rays with the scaler-timer allows rapid measurement because the microcomputer can process data even during the running of the SMs and the counting of X-rays.

X-ray count y , or time t required to count fixed counts in the case of the fixed count method, are corrected by the LPA (Lorentz-polarization and absorption) factor in step ⑦ to obtain corrected counts z (or u), where $z=y/L$, $u=Lt$ and L is the LPA factor. The measurement of X-ray count y is repeated in the loop of ④ to ⑨ until z becomes less than $0.8z_{\max}$ (or u becomes larger than $1.25u_{\min}$); above this value the diffraction peak can be approximated by a Gaussian curve⁽¹⁾.

When condition ⑨ is fulfilled, the measurement y is completed and the peak position, p , and standard deviation σ_p of p representing the magnitude of scatter arising from X-ray counting statistics, are calculated in step ⑩. At the same time, a starting angular point x_0 for the next ϕ_0 is also determined. Then, the ϕ 1-SM rotates the goniometer head to the next angular position, ϕ_0 , and the measurement of X-ray count y is repeated again in the loop of ④ to ⑨ in Fig. 5. When all the measurements at the four ϕ_0 angles ($0^\circ, 15^\circ, 30^\circ$ and 45°) are finished the microcomputer exits this loop to step ⑬, where the stress value S and its standard deviation σ_s are calculated and the stress measurement is completed.

In the fixed ψ method, which is useful for coarse grained materials, the angle ψ is kept fixed during the scanning of the X-ray detector as in the conventional diffractometer technique, where ψ is the angle between the specimen normal and the diffraction plane normal. This method is achieved by controlling the 2θ -SM and ϕ 1-SM such that they change the 2θ angle twice as much as angle ϕ_0 . The methods of the measuring Gaussian curve parameter (GCP)⁽⁴⁾ and the half-width of the diffraction profile are not described here, because they are the same as those of peak

position determination by the Gaussian curve method⁽¹⁾ and the half-width method⁽⁹⁾, respectively, except for equations for the calculation.

5. Test Procedures

To test the performance and viability of the system, the residual stresses of the three specimens shown in Table 2 were measured. Structural carbon steel JIS type S45C has a broad diffraction profile (half-width of 6.6°) characteristic of a martensitic structure. The specimen of a low-carbon hot rolled steel SS41 with coarse grains of $74 \mu\text{m}$ size was used for residual stress measurement of a coarse grained sample. A small irradiated area ($2 \times 2 \text{ mm}$) was used for this specimen in order to reduce the number of grains diffracted. The accuracy of the stress measurement was checked using an annealed stress-free chromium powder of 200 mesh. Table 3 gives the conditions of the X-ray stress measurement.

6. Test Results

Figures 6 and 7 show the peak position determination of hardened steel specimen S45C by the Gaussian curve method, using the fixed time and fixed count methods respectively. The curve in Fig. 7 for the fixed count method is calculated from data points of $1/u$ using the least squares method. The Gaussian curve method in this system can determine the peak position in less than one third of the time, than in the half-width method, because it uses only several data points around the peak and can minimize the number of measurements not used in calculating the peak position.

Figure 8 shows a $\sin^2\psi$ diagram of the peak positions determined by the method shown in Figs. 6 and 7. The residual stress values obtained by the four methods shown in Fig. 8 range from -603.3 to -621.7 MPa. The standard deviation of the stress value determined by the fixed time method⁽¹⁾⁽²⁾ is 16.6 MPa for the iso-inclination method and 19.4 MPa for the side-inclination method. The stress values determined by the four methods fell within the scatter band of the

Table 2 Dimensions and heat treatment of specimens

Specimen	Dimensions, mm	Heat Treatment
S45C	25 x 25 x 75	Water quenched after being austenitized at 850°C for 30 min
SS41	76 x 15 x 5	Annealed at 1100°C for 10 h after being stretched by 1.0%
Cr Powder		Annealed at 900°C for 30 min

Table 3 Conditions of X-ray residual stress measurement

Characteristic X-rays	Chromium K α
Diffraction planes	(211) planes of α -Fe and Cr
Filter	Vanadium foil
Tube voltage, kV	30
Tube current, mA	8
Irradiated area, mm ²	5x5 (S45C, Cr), 2x2 (SS41)
Preset time, s	8 (S45C, Cr), 10 (SS41)
Preset count, counts	10000

measured values.

Figure 9 shows the results of the stress measurement of the coarse grained specimen SS41 by the Gaussian curve method, using the fixed η and ψ methods with the side-inclination method (SIM), and the η oscillation method. The poor linearity of the

Fig. 6 Peak position determination by the Gaussian curve method using the fixed time method

Fig. 7 Peak position determination by the Gaussian curve method using the fixed count method

diagram by the fixed η method shows the low accuracy of the measurement. Use of the SIM-fixed ψ method and η oscillation improves the linearity of the

Fig. 8 $\sin^2\psi$ diagram for hardened carbon steel S45C by the fixed time and fixed count methods

Fig. 9 $\sin^2\psi$ diagram for coarse grained mild steel SS41 by the Gaussian curve method using the side-inclination method.

Fig. 10 $\sin^2 \psi$ diagram for annealed chromium powder

$\sin^2 \psi$ diagram; the η oscillation method gives the best linearity. Thus, the oscillation method, using the Gaussian curve method, similar to the one using the half-width method, proves to be effective for the measurement of coarse grained materials.

Figure 10 is the $\sin^2 \psi$ diagram for standard chromium powder; its peak position was determined by the half-width method and the Gaussian curve method using the iso- and side-inclination methods. Residual stress values of -3.1 to 2.8 MPa were obtained with standard deviations of 1.8 to 4.2 MPa. This result from stress free powder specimens indicates the high accuracy of the measurement system.

7. Conclusion

1) A programmable X-ray measurement system having many uses, such as stress measurement by the Gaussian curve method and the evaluation of diffraction profile width using the Gaussian curve parameter (GCP), has been developed.

2) The scaler-timer circuit in this system can be used not only for the fixed time method but also the fixed count method, which can equalize the precision

of all data points of X-ray intensity.

3) Three stepper motors in this system are used to change the diffraction angle and the X-ray incident angles for the iso- and side-inclination methods. Simultaneous control and drive of the three stepper motors allows not only rapid measurement but also the use of various methods such as η oscillation and fixed ψ methods using the side-inclination method which was not viable with the previous system developed by the authors.

4) Residual stress values of a hardened structural steel S45C were measured by the iso- and side-inclination methods using the fixed time and fixed count methods and the standard deviations of the stress were calculated. As a result, the stress values measured by the four methods fell within the scatter band of the measured values.

5) Residual stress of a coarse grained annealed mild steel SS41 was measured by the Gaussian curve methods with the fixed η , the η oscillation, and the SIM-fixed ψ methods. As a result, the η oscillation method gave the best linearity of the $\sin^2 \psi$ diagram. Thus, the stress of coarse grained materials can be measured by the oscillation method using the Gaussian curve method.

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