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COMPARISON OF MEASURING TIMES FOR X-RAY DETERMINATION OF RESIDU--ETC(U)  
MAR 77 M R JAMES, J B COHEN

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COMPARISON OF MEASURING TIMES FOR X-RAY DETERMINATION OF RESIDUAL STRESSES:  
A NORMAL DETECTOR AND A POSITION SENSITIVE DETECTOR

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COMPARISON OF MEASURING TIMES FOR X-RAY DETERMINATION OF RESIDUAL STRESSES:

A NORMAL DETECTOR AND A POSITION SENSITIVE DETECTOR

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ABSTRACT

The use of a position sensitive detector (PSD) in the measurement of residual stress by the X-ray technique has been described previously (see T.R. No. 11). To quantify the actual time savings for the stress determination, a complete comparison was carried out between a normal diffractometer measurement and a PSD measurement. The speed of the PSD is dependent on the profile characteristics and the desired accuracy of the measurement. Therefore, measurements were carried out on six steel samples covering the full range of peak breadths and for both a high and a low statistical counting accuracy. In all cases, the PSD was considerably faster than the normal detector, from 100 times quicker for the most favorable case (sharp profile, high statistical error) to twice as fast (broad profile, low statistical error) for the least favorable case.

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## CHAPTER 5 \*

## 5.1 INTRODUCTION

A position sensitive detector (PSD) offers many significant advantages over normal radiation detecting equipment. The ability to monitor an entire peak as it is being accumulated allows early judgement of the quality of an experiment<sup>(54,55)</sup> which is an inherent drawback of film or counter techniques. Also, the possibility of background reduction can be achieved by energy discrimination and/or background subtraction.<sup>(45)</sup>

For quantitative measurements experimental advantages include no moving parts thereby eliminating the expensive diffractometer, fluctuations in the incident power level do not distort the scattering pattern and experimental procedures may be simplified. As an example, Schelton and Hendricks<sup>(91)</sup> report that for small angle X-ray scattering (SAXS) it becomes reasonable to use point collimation as the time loss due to low intensity (due to collimation in both directions) is recovered when using the PSD by simultaneous recording of the data at many scattering angles. The large mathematical corrections for slit-smearing effects are eliminated when using point collimation. The PSD, then, allows direct observation of the true scattering function.

For most experiments, the real advantage of the PSD system is the shortening of data accumulation time because information is recorded simultaneously over a large scattering angle. Dupont, et al,<sup>(55)</sup> report a decrease in time of two orders of magnitude over conventional film methods when the PSD was applied to SAXS studies of biological membranes.

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\* Chs. 1-3 constitute TR No. 14, Ch. 4 will be TR No. 16 and is covered in summary form in TR No. 12. This Ch. 4 concerns programming for automation of stress measurements and is not necessary reading for this report.

This particular advantage makes the PSD a practical tool to use in residual stress analysis by X-ray diffraction.

To quantify the actual time savings for stress determinations and the accuracy of the PSD system, a complete comparison was carried out between a normal diffractometer measurement and a PSD measurement of residual stress. The speed of the PSD is dependent on the profile characteristics and the desired accuracy of the measurement, therefore, measurements were carried out on six steel samples covering the full range of peak breadths and for both a high and a low statistical counting accuracy. In all cases, the PSD was considerably faster than the normal diffractometer method; from 100 times quicker for the most favorable case (sharp profile, large statistical error) to only twice as fast (broad profile, small statistical error) for the least favorable case.

## 5.2 EXPERIMENTAL PROCEDURE

Residual stress measurements using the two tilt method were carried out with the automated residual stress package described in ONR T.R. No. 16. The Picker diffractometer was utilized. For the PSD system, the detector was mounted on the 2 $\theta$  arm which was fixed at 156°2 $\theta$ . Calibration of the PSD was accomplished using a stress free 1090-1 specimen using the procedure described in ONR T.R. No. 11. The residual stress on each sample was measured to predetermined statistical counting errors of  $\pm 3.5$  MPa ( $\pm 500$  psi) and  $\pm 27.6$  MPa ( $\pm 4000$  psi) with

each measurement being repeated three times.

The total time for the measurement includes all the time during data accumulation but not the time for input/output on the teletype since the latter depends solely on the type of information desired by the operator. The results presented are an average over the three measurements for the residual stress and total time for each specimen.

### 5.3 RESULTS AND DISCUSSION

Table 5.1 records the results using the normal diffractometer method of residual stress analysis. The second, third and fourth columns give data on the profile for each sample. The size of the X-ray beam on the samples was 5 mm high and 2.5 mm and 5 mm wide for the 1° and 2° divergent slits (column 3) respectively.

The automated X-ray step-scan diffractometer measurement requires using five successive step scans to determine the peak position, as described in ONR T.R. No. 16.

The first four steps-scans, called the preliminary scans, serve to refine the peak position and determine the time spent in the final step-scan to obtain a predetermined statistical counting error. This procedure for peak location serves to determine the peak to a preset error in the minimum time. For each sample the stress and time of analysis is given in columns 5 and 6 of Table 5.1 for a low counting error and columns 7 and 8 for a high counting error.

For the 1090-1 sample there is only a small difference in total time between the measurement for the low and the high statistical precision because most time (~ 400 sec) is spent in the preliminary scans. As the breadth of profile (column 2) increases, more time is

TABLE 5.1  
 TOTAL TIME FOR MEASUREMENT OF RESIDUAL STRESS USING A STANDARD DIFFRACTOMETER  
 (TWO TILT METHOD - THREE POINT PARABOLIC FIT)  
 (211 CrK<sub>α</sub> Peak)

Sample	Breadth (°2θ)	Divergent Slit (°2θ)	Peak Intensity (CPS)	Low Error		High Error	
				Stress (MPa)	Time (SEC)	Stress (MPa)	Time (SEC)
1090-1	.45	1	1180	15.4 ±3.5*	430	18.2 ±21.9	410
1045-1	1.5	1	850	-179.5 ±3.4	800	-206.0 ±24.2	480
1045-3	3.45	2	2230	-701.2 ±3.5	2600	-680.0 ±26.7	480
1045-2	5.1	2	1450	-402.0 ±3.6	9500	-383.4 ±25.9	570
TBA G-5	5.8	2	1180	16.0 ±5.4	11100	10.7 ±27.1	750
1090-2	6.0	2	1760	-314.1 ±5.0	12900	-304.8 ±29.4	860

\* Statistical counting error in MPa.

spent in the final step-scan to achieve the desired precision. Therefore, the difference between times for the low and high errors increase, although the time spent in the preliminary scans remains essentially constant.

The PSD provides position information with a high count rate capability enabling the entire diffraction profile to be obtained simultaneously, eliminating the preliminary step scans. This is somewhat offset by the lower counting efficiency of the PSD as compared to a scintillation detector.\* Using the same size primary beam as given in Table 5.1, the total time of analysis for residual stress measurements using the PSD are given in Table 5.2. A comparison of the two tables shows that in all cases the PSD exhibits a remarkable decrease in time over the conventional diffractometer measurement with good agreement in the measured residual stress.

For the 1090-1 sample and a statistical error of  $\pm 27.6$  MPa ( $\pm 4000$  psi) the time is decreased by 100 when using the PSD. For the lower error of  $\pm 3.5$  MPa ( $\pm 500$  psi) the PSD is 14 times faster. Although the lower efficiency of the PSD works against the other features when long counting times are necessary, the accumulation of many data points offsets this liability. This is most dramatically shown in the high precision measurement of the 1090-2 sample where, with a normal diffractometer, 97% of the time is spent in the final step scan. The PSD is still twice as fast indicating that the collection of many data points more than offsets the five-fold decrease

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\* In ONR TR No. 11 the efficiency of the PSD was shown to be only 20 pct that of a scintillation detector due to a combination of the decreased probability for photon absorption and the small slit height required for satisfactory spatial resolution along the detector axis.



TABLE 5.2

**TOTAL TIME FOR MEASUREMENT OF RESIDUAL STRESS  
USING A POSITION SENSITIVE DETECTOR  
(TWO TILT METHOD)**

Sample	Number of Data Points	Low Error		High Error	
		Stress (MPa)	Time (SEC)	Stress (MPa)	Time (SEC)
1090-1	9	25.4 $\pm$ 5.4	30	17.0 $\pm$ 21.9	4
1045-1	27	-175.3 $\pm$ 4.6	300	-188.9 $\pm$ 25.5	10
1045-3	45	-680.7 $\pm$ 3.8	2000	-687.8 $\pm$ 22.9	60
1045-2	95	-382.0 $\pm$ 5.2	2000	-363.7 $\pm$ 30.0	60
TBA G-5	111	14.2 $\pm$ 4.8	4000	12.4 $\pm$ 37.4	70
1090-2	131	-336.2 $\pm$ 4.4	5000	-321.7 $\pm$ 32.3	100

in counting efficiency of the PSD compared to the scintillation detector on a normal diffractometer.

This improvement is enhanced when one considers the true, observable precision and not just the statistical counting error. In Sec. 3.6.3 it was shown that the counting error is not a good estimate of the precision found from replicate testing when the three point fit and two-tilt method were used with samples having a broad diffraction profile. In this case it was shown that only for the  $\sin^2\psi$  method using a three point fit with background subtraction was the counting error similar to the observed error over replicate measurements. Therefore, measurements were made on the normal diffractometer using the 1090-2 sample having the broadest diffraction profile. Four tilts were used in the  $\sin^2\psi$  method, a three point parabola with background subtraction being utilized to define the peak position. For a precision of  $\pm 29.4$  MPa ( $\pm 4260$  psi) the total time was 1300 seconds and for  $\pm 5.5$  MPa ( $\pm 795$  psi) the time was 24,500 seconds. Comparing these times with those obtained for the 1090-2 sample in Table 5.2 shows how dramatic the real time saving is. For the PSD, many data points are obtained as seen in column 2, Table 5.2, so the errors tabulated are true indications of the reproducibility (see ONR TR No. 14 for a comparison of counting error and observed error for the PSD). Compared to the normal diffractometer measurement of residual stress, the PSD is faster both for sharp profile samples where elimination of the preliminary scans constitutes most of the time savings and for broad profiled samples where many accumulated data points further emphasizes the advantages of the PSD.

When using the PSD one can estimate the counting time for a specific accuracy very easily. The statistical error in peak location can be written as:

$$\sigma(2\theta_p) = At^{-\frac{1}{2}} \quad (5.1)$$

where  $t$  is the time and  $A$  is a proportionality factor which depends on the breadth and intensity of the profile. For the two tilt method, the error in stress is given by:

$$\sigma(\sigma_\varphi) = K(2\sigma^2(2\theta_p))^{\frac{1}{2}} \quad (5.2)$$

where it is assumed that both peaks are obtained to the same accuracy. Substituting Eq. 5.1 into Eq. 5.2 and combining the constants gives:

$$\sigma(\sigma_\varphi) = A^* t^{-\frac{1}{2}} \quad (5.3)$$

where  $A^*$  is a proportionality factor that may be determined from one measurement by relating the time to the statistical error. This allows the time for any other accuracy to be determined. For example, the 1045-1 sample gives a value for  $A^*$  of 80.6 for the high error measurement. Calculating the time for the low error measurement yields 307 sec, very close to the actual time of 300 sec. From Eq. 5.3 it is seen that the error goes down as  $t^{\frac{1}{2}}$ . From Table 5.2 an estimate of the time of analysis for large errors of  $\pm 70$  MPa (10,152 psi) can be made; e.g., the 1045-3 sample gives a time of 6.4 sec. Such times enable

dynamic residual stress measurements to be obtained in manufacturing processes to assure quality control. In the past, this has been done with dedicated units such as Fastress<sup>(2)</sup> taking at least one to two minutes.<sup>(92)</sup> Obviously, the PSD offers an excellent potential for making accurate residual stress measurements in short enough time for industrial use. This will be demonstrated in later reports.

## REFERENCES

45. C. J. Borkowski and M. K. Kopp, *IEEE Trans. Nucl. Sci.*, 17, 340 (1970).
46. J. Hough and A. Drever, *Nucl. Inst. and Meth.*, 103, 365 (1973).
47. H. Foeth, R. Hammerstrom, and C. Rubbia, *Nucl. Inst. and Meth.*, 109, 521 (1973).
48. D. G. Smith and K. A. Pounds, *IEEE Trans. Nucl. Sci.*, 15, 541 (1968).
49. R. Gott, W. Parks and K. A. Pounds, *Nucl. Inst. and Meth.*, 81, 152 (1970).
50. J. Hough, *Nucl. Inst. and Meth.*, 105, 323 (1972).
51. G. I. Miller, A. Senator, and R. Stensgraad, *Nucl. Inst. and Meth.*, 91, 389 (1971).
52. G. P. Westphal, *Nucl. Inst. and Meth.*, 106, 279 (1973).
53. H. W. Fulbright, R. G. Markham, and W. A. Langford, *Nucl. Inst. and Meth.*, 108, 125 (1973).
54. J. L. C. Ford, J. Gomes, Del Campo, R. L. Robinson, B. H. Stelson, and S. T. Thornton, *Nuclear Physics*, A226, 189 (1974).
55. A. Gabriel and S. Bram, *Febs. Letters*, 39, 307 (1974).
56. Y. Dupont, A. Gabriel, MChabre, T. Gulik-Krzywicki, and E. Schechter, *Nature*, 238, 331 (1972).
57. E. Mathieson, *Nucl. Inst. and Meth.*, 97, 171 (1971).
58. H. P. Klug and L. E. Alexander, X-Ray Diffraction Procedures, John Wiley and Sons, Inc., New York (1974) p. 329.
59. Ibid., p. 635.
60. F. Y. Yap, dissertation (The John Hopkins University, Baltimore, Maryland, 1967).
61. A. J. C. Wilson, *Acta. Cryst.* 23, 888 (1967).
62. J. Thomsen and F. Y. Yap, *J. of Research of Nat. Bureau of Standards*, 72A, 187 (1968).
63. A. J. C. Wilson, *Brit. J. Apply. Phys.*, 16, 665 (1965).
64. M. J. Cooper and A. V. Glasspool, *J. Apply. Cryst.* 9, 63 (1976).

65. International Tables for X-Ray Crystallography, Vol. III, Kynoch Press, Birmingham, England (1968).
66. J. B. Cohen, "Report on Tungsten Lattice Parameter Round Robin," X-Ray Subcommittee of SAE Iron and Steel Technical Committee, Division 4, (November 1964).
67. J. Fukura and H. Fujiwara, J. of the Society of Materials Science, Japan, 15, 825 (1966).
68. S. Aoyama, K. Satta and M. Tada, J. of the Society of Materials Science, Japan, 17, 1071 (1968).
69. D. N. French, J. Amer. Cer. Soc., 52, 271 (1969).
70. W. E. Baucum and A. M. Ammons, Adv. in X-Ray Analysis, 17, 371 (1973).
71. "Standard Method for X-Ray Stress Measurement", The Committee on Mechanical Behavior of Materials, The Society of Materials Science, Japan (1973).
72. Residual Stress Measurements by X-Ray Diffraction-SAE J784a, 2nd Edition, Society of Automotive Engineers, Inc., (1971), p. 51.
73. J. Sekita, K. Oguro, U. Kaminago and Y. Oguro, X-Ray Study on Strength and Deformation of Metals, The Society of Materials Science, Japan (1971) p. 81.
74. A. J. C. Wilson, Mathematical Theory of X-Ray Powder Diffractometry, Philips Technical Library (1963).
75. W. Parrish and T. C. Huang, Profile Tilting: A Powerful Method of Computer X-Ray Instrumentation and Analysis, IBM Research (1976).
76. O. Davies and P. Goldsmith, Statistical Methods in Research and Production, Hafner Publ. Co., New York (1952) p. 72.
77. X-Ray Diffraction, Ed. L. Azaroff, McGraw Hill, New York (1974) p. 439.
78. C. J. Kelly and M. A. Short, Adv. in X-Ray Analysis, 14, 377 (1970).
79. Koves and Ho, Norelco Reporter, 11, 99 (1964).
80. C. J. Kelly and E. Eichen, Adv. in X-Ray Analysis, 16, 344 (1972).
81. T. Hayama and S. Hashimoto, J. of the Society of Materials Science, 23, 75 (1975).
82. K. Paavola, M.S. thesis, Northwestern University, Evanston, IL., (1970); Jrnl. of Appl. Cryst. 4 (1971) 524 with M. Richesson, L. Morrison and J. B. Cohen.

83. D. Schneider and B. Smith, PS/8 FOCAL 1971, Student Research Center, Oregon Museum of Science and Industry (1971).
84. J. B. Nelson and D. P. Riley, Proc. Phys. Soc. (London) 57, 160 (1948).
85. M. Ezekiel and K. Fox, Methods of Correlation and Regression Analysis, 3rd Edition, (1965).
86. W. Volk, Applied Statistics for Engineers, McGraw-Hill Book Co., Inc., New York (1958).
87. E. Macherlauch, Exp. Mech., 6, 1940 (1966).
88. H. Neerfield, MiH. KWI Eisenforsch., 24, 61 (1942).
89. R. Hill, Proc. Phys. Soc. (London), 65, 349 (1952).
90. P.S. Prevey, Adv. in X-Ray Analysis, 20 (1976), in press.
91. J. Schelten and R. W. Hendricks, J. Appl. Cryst., 8, 421 (1975).

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