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NBS TECHNICAL NOTE 709

An X-Ray Diffraction Method for Determining the Amount of Austenite in an Austenite-Ferrite Mixture

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An X-Ray Diffraction Method for Determining the Amount of Austenite in an Austenite-Ferrite Mixture

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by

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A method for determining the relative phase volume of austenite in an austenite-ferrite mixture is described in detail. Results obtained by applying this method to an austenitic-ferritic stainless steel composite material are presented. The method can be extended to other multi-phase materials.

Keywords: Austenite; computer; ferrite; phase measurement; quantitative analysis; retained austenite and x-ray diffraction.

INTRODUCTION

In the characterization of alloys it is often important to determine the volume fraction of certain phases. In the case of ferrous alloys it is necessary to measure the amount of austenite retained after thermo-mechanical processing since it will affect important mechanical properties. Several different techniques have appeared in the literature (see references in (1) and (2)) for determining the relative amount of austenite, employing x-ray diffraction methods. This note will discuss in detail the method in use in our laboratory, which differs in some respect from these other techniques. Principally, integrated intensity measurements from as many resolved, individual, diffraction lines as possible are combined to determine a best value for the volume fraction of austenite. Computer processing permits rapid collection of data and calculation of results. An example of the application of this method to a recent NBS standard reference material will be given.

THEORY

The theory concerned with the calculation of the volume fraction of a phase in a mixture using a diffractometer has been summarized and discussed by Ogilvie(2). That method assumes, as is done here, a random orientation of the diffracting crystallites. Modifications of the method to account for preferred orientation in, for example, rolled material, have been proposed(3) and will not be considered here.

The integrated intensity of a reflection (hkl) is given by:

$$P_{hkl} = K \cdot F^2 \cdot T \cdot L \cdot p \cdot j \cdot A \cdot V / v_c^2 \tag{1}$$

where

K = scale factor (constant for a given experiment)

f = scattering factor

F = f·S, the structure factor (and $F^2 \equiv FF^*$)

T = $e^{-2B/4d^2}$, the temperature factor, where B is a constant and d the interplanar spacing.

S = symmetry factor with S = 2 (body-centered-cubic)
S = 4 (face centered cubic)

L·p = $(1 + \cos^2 2\Theta)/(\sin^2\Theta \cdot \cos\Theta)$, Lorentz polarization for powder specimen

j = multiplicity factor

A = absorption factor (nearly constant for thick specimens)

V = volume fraction of the phase of interest, and

v_c = unit cell volume.

The known terms are collected into a quantity R^{hkl} so that eq (1) can be written

$$P_{hkl} = K \cdot R^{hkl} \cdot V \tag{2}$$

where $R^{hkl} \equiv F^2 \cdot T \cdot L \cdot p \cdot j \cdot A / v_c^2$

Forming the ratio for two phases from eq (2), we have

$$\frac{P_1^{hkl}}{P_2^{h'k'l'}} = \frac{KR_1^{hkl}V_1}{KR_2^{h'k'l'}V_2} \tag{3}$$

and if $V_1 + V_2 = 1$ (two phase material) then

$$V_2 = \frac{R_1^{hkl}\ P_2^{h'k'l'}}{R_2^{h'k'l'}\ P_1^{hkl} + R_1^{hkl}\ P_2^{h'k'l'}} \tag{4}$$

or

$$V_2 = \frac{(R_1/P_1)^{hkl}}{(R_1/P_1)^{hkl} + (R_2/P_2)^{h'k'l'}}. \tag{5}$$

Letting $I = R/P$ and averaging over all reflections in a phase, eqn. 5 becomes

$$\bar{V}_2 = \frac{\bar{I}_1}{\bar{I}_1 + \bar{I}_2} \tag{6}$$

and the standard deviation is given by

$$s_{V_2} = \bar{V}_2(1 - \bar{V}_2)[(s_{\bar{I}_1}/\bar{I}_1)^2 + (s_{\bar{I}_2}/\bar{I}_2)^2]^{1/2}. \tag{7}$$

The method for determining the average volume fraction $\bar{V}_2$ from calculated values R_i and measured values P_i was programmed for computer calculation and is described in the next section.

MEASUREMENT METHOD AND APPLICATION

The experimental conditions used with the application of the method to an austenitic-ferritic stainless steel composite will be described. Appropriate adjustments in, for example, radiation, stepping intervals and so forth, can be made for studies of other materials. In the present instance cobalt radiation (iron filtered) was obtained from a constant potential x-ray generator operated at 35kV. The step-scanning diffractometer used a 1° scattering slit and a .024 inch receiving slit. Counting periods of 50 to 100 s were chosen, with a 2Θ step interval of 0.05 deg (this choice is determined in part by the sharpness of the peaks). For the purpose of minimizing the effect of grain size, the specimen was rotated in its plane at 117.8 rpm during measurement.

The scaler output was recorded directly on punched paper tape to facilitate loading the data into a digital (time-shared) computer. Other input information, such as the interval for background corrections, the interval over which the peaks were integrated, appropriate hkl, multiplicity, and symmetry factor were supplied from a previously loaded control file. Integration of the diffraction lines was accomplished using Simpson's rule. The diffraction line center-of-gravity along the 2Θ axis was also determined and used to specify the angular position of the line. The computer program (described in the Appendix) then calculated the d-spacing, Lorentz polarization factor (with or without monochromator), unit cell volume, and temperature factor. The scattering factor was calculated from the relation (4):

$$f_{hkl} = A \exp(-B/4d^2) + C \exp(-D/4d^2) + E$$

where A, B, C, D, E are constants (see Appendix). A correction was made to these calculated values in order to account for anomalous scattering.

The computer program provides for a linear correction of background intensity. The theoretical relative intensity (R in eq 2) of the principal diffraction lines of each phase is calculated and printed out together with the observed integrated intensity and relative integrated intensity for comparison. As a further check the multiplicity, temperature factor, scattering factor correction, Lorentz polarization factor, 2θ angle, d-spacing and average lattice constant are also printed out. To minimize systematic biases in the final calculated phase volume fraction, the calculations are done for as large a number of planes as possible and averaged together. In the austenite-ferrite example, presented next, this involved 5 planes for the fcc phase and 4 planes for the bcc phase.

The output obtained from measurements on an austenitic-ferritic stainless steel composite specimen is shown in table I. The average volume percent austenite present is shown followed by individual values obtained from pairs of diffraction lines. The scatter shown indicates the need for caution in basing phase volume determinations on only a few observed diffraction lines, particularly at low relative values.

This program can be used with slight modification for other phases with simple structure and can be extended to more complicated structures with the introduction of the appropriate equations for the symmetry factor. A modification of this program has also been used to study preferred orientation resulting from the directional growth of crystals on electrodeposited plates in various plating solutions.

REFERENCES

[1] Beu, K. E., Austenite Retained in Hardened Steels: Quantitative Determination by X-Ray Diffraction Techniques", in the Encyclopedia of X-Rays and Gamma Rays, George L. Clark, ed., Reinhold Publishing Corp., New York, pp. 71-77 (1963).

[2] Ogilvie, R. E., Retained Austenite by X-Rays, Norelco Reporter, p. 60-63 (1959).

[3] Morris, P. R., Measurement of Retained Austenite in Precipitation-Hardening Stainless Steels, Trans. AIME, 239, pp. 1586-1589 (1967).

[4] Forsyth, J. B. and Wells, M., On an Analytic Approximation to the Atomic Scattering Factor, Acta, Cryst. 12, pp. 412-515 (1959).

APPENDIX

The computer program listed below is written in the BASIC language and should execute on most time-shared computer terminals accepting a BASIC language, except perhaps for minor changes in input and output statements. This program has executed on four different systems available in our laboratory.

Because of size limitations this program initially consisted of three programs but they were recently combined. It should be possible to subdivide again if necessary for a particular computer system.

The program permits greater details to be printed at execution time or listed after execution. This was incorporated to "shake down" the original program, facilitate transfer between time-sharing services, and permit inspection of data used for background correction and data over which the peaks were to be integrated.

A punched paper tape of this program can be provided on request to the author.

Table I. Computer output showing calculated and observed integrated diffraction line intensities, and relative phase volume in a study of an austenitic-ferritic stainless steel composite.

```
 XBASIC B
RUN
 DEFINE FILE
 1=CONTRL
 DEFINE FILE
 2=HJL4


SPECIMEN DESIGNATION--! HICHO JL4

DETAILED PRINTOUT DESIRED?  ENTER Y IF YES AND N IF NO! N

FOR LP, ENTER 1 IF MONOCHROMATOR., 2 IF NOT.  ! 2

ANOMALOUS SCAT. FACT. CORRECTION, ENTER 0 IF NONE DESIRED.  ! 2.5

TEMPERATURE CONSTANT R=  .4
```

AUSTENITE

PLANE	M	SCAT.F	TEMP.F	LP	F	D-SPACING
111	8	16.36	.954	8.27	63.93	2.072
200	6	15.20	.940	5.82	58.93	1.797
220	12	11.82	.883	2.83	44.42	1.268
311	24	10.10	.844	2.94	37.12	1.084
222	8	9.61	.830	3.30	35.03	1.037

PLANE	TWO-THETA	RELATIVE CALC.I	RELATIVE OBSERVED I	OBSERVED INTEGRATED I
111	51.190	100.00	100.00	156.60
200	59.771	44.84	32.68	51.18
220	89.854	24.78	25.75	40.33
311	111.325	35.97	33.56	52.55
222	119.436	11.98	10.82	16.94

LATTICE CONSTANT= 3.591

FERRITE

PLANE	M	SCAT.F	TEMP.F	LP	F	D-SPACING
110	12	16.18	.952	7.81	31.57	2.024
200	6	13.10	.907	3.44	24.96	1.434
211	24	10.96	.864	2.73	20.38	1.172
220	12	9.39	.824	3.56	17.03	1.015

PLANE	TWO-THETA	RELATIVE CALC.I	RELATIVE OBSERVED I	OBSERVED INTEGRATED I
110	52.493	100.00	100.00	4558.26
200	77.254	13.78	14.73	671.23
211	99.628	29.12	31.52	1436.58
220	123.684	13.28	13.80	629.12

LATTICE CONSTANT= 2.868

AVERAGE % AUSTENITE= 3.862 STD. DEV. = .501

95% CONFIDENCE INTERVAL IS 3.444 TO 4.279

RETAINED AUSTENITE BY PAIRS

PLANES AUSTENITE	PLANES FERRITE	% AUSTENITE
111	110	4.37
200	110	3.22
220	110	4.53
311	110	4.09
222	110	3.96
111	200	4.10
200	200	3.02
220	200	4.25
311	200	3.83
222	200	3.72
111	211	4.05
200	211	2.99
220	211	4.20
311	211	3.79
222	211	3.67
111	220	4.21
200	220	3.11
220	220	4.37
311	220	3.94
222	220	3.82

```
100 FILES *,*'PERMITS DESIGNATION OF CONTROL AND DATA FILES AT RUN TIME
110 FMT I4,I6,F9.2,F11.3,F12.2,F12.2,F12.3
120 DIM X(600),Y(500),Z(500),H(2,5),C(2,5),P(2,5),O(2,5),U(2,5),M(2,5)
130 DIM B(2,5),N(2),A(2),F(9),G(9),A$(2),T(2,5),D(2),J(2)
140 FMT //
150 FMT I4,F15.3,F12.2,F12.2,F14.2,F12.3
160 FMT "LATTICE CONSTANT=" F6.3,///
170 FMT "AVERAGE % AUSTENITE=",F9.3,"   STD. DEV. =",F7.3,//
190 FMT "95% CONFIDENCE INTERVAL IS",F9.3,"     TO",F9.3,//
200 FMT "RETAINED AUSTENITE BY PAIRS"//"       PLANES"
210 FMT "AUSTENITE  FERRITE  % AUSTENITE"
220 FMT I6,I10,F12.2
230 FMT "TEMPERATURE CONSTANT R=",F4.1
240 FMT I8,I5,I5,I5,I5
250 READ #1,N(1),N(2),M1,M2,M3,W1,P1,T9,A(1),A(2),A$(1),A$(2)
260 PRINT,140
270 PRINT"SPECIMEN DESIGNATION--";
280 INPUT T$  'COMPUTER CALLS FOR SPECIMEN DESIGNATAON'
290 PRINT
300 PRINT "DETAILED PRINTOUT DESIRED?  ENTER Y IF YES AND N IF NO";
310 INPUT T$
320 PRINT
330 PRINT"FOR LP, ENTER 1 IF MONCH., 2 IF NOT.  ";
340 INPUT N1
350 PRINT
360 PRINT"ANOMALOUS SCAT. FACT. CORRECTION, ENTER O IF NONE DESIRED.
370 INPUT S9
380 PRINT
390 READ #2,Q9
400 *  AT THIS POINT PROGRAM READS IN SCALER FILE AND SORTS DATA FOR
410 *BACKGROUND CORRECTION, AUSTENITE PEAKS, AND FERRITE PEAKS.
420 *CONTROL IS EXERCISED FROM THE CONTROL FILE WHICH IS AMENDED FOR
430 *EACH RUN IF NECESSARY.  ALL ANGULAR DATA IS IN TERMS OF
440 *TWO-THETA TIMES 1000.
450 READ #1, A,S,T,A3
460 *A=STARTING ANGLE AND A3=ENDING ANGLE OF DIFFRACTOMETER RUN
470 *S=STEPPING ANGLE AND T=COUNTING TIME INTERVAL.
480 A=A-S
490 READ #1,N,A1,A2
500 *N=SORTING INSTRUCTIONS, 1--BACKGROUND, 2--AUSTENITE PEAK
510 *3--FERRITE PEAK; A1 AND A2 STARTING ANGLE AND ENDING ANGLE OF
520 *THE SCAN OF INTEREST.
530 A=A+S
540 READ #2, B
550 B=B+Q9
560 IF A<>A1 THEN 530
570 ON N GOTO 620,740,870
580 IF T$="N" THEN 600
590 PRINT,240,A;M1;M2;M3
600 IF A<>A3 THEN 490
610 GOTO 1030
620 M1=M1+1
630 X(M1)=A1
640 M1=M1+1
650 X(M1)=B
660 IF A<>A2 THEN 700
```

```
670 M1=M1+1
680 X(M1)=-1
690 GOTO 580
700 A=A+S
710 READ #2,B
720 B=B+Q9
730 GOTO 640
740 M2=M2+1
750 Y(M2)=A1
760 N(1)=N(1)+1
770 M2=M2+1
780 Y(M2)=B
790 IF A<>A2 THEN 830
800 M2=M2+1
810 Y(M2)=-1
820 GOTO 580
830 A=A+S
840 READ #2,B
850 B=B+Q9
860 GOTO 770
870 M3=M3+1
880 Z(M3)=A1
890 N(2)=N(2)+1
900 M3=M3+1
910 Z(M3)=B
920 IF A<>A2 THEN 960
930 M3=M3+1
940 Z(M3)=-1
950 GOTO 580
960 A=A+S
970 READ #2,B
980 B=B+Q9
990 GOTO 900
1000 *END OF INPUT FROM SCALER FILE
1010 *CALCULATES CONSTANTS TO BE USED IN LINEAR CORRECTION OF BACK-
1020 *GROUND. ALSO READS IN HKL'S AND MULTIPLICITY FACTORS.
1030 S1=S/1000
1040 READ #1,E9,M1,M2,M3
1050 FOR E1=1 TO E9
1060 Y=0
1070 X1=0
1080 X2=0
1090 Z=0
1100 N=0
1110 FOR E=1 TO 2
1120 M1=M1+1
1130 A=X(M1)/1000
1140 FOR E3=1 TO 500
1150 M1=M1+1
1160 B=X(M1)
1170 IF B<0 THEN 1250
1180 Y=Y+B
1190 X1=X1+A
1200 X2=X2+A*A
1210 Z=Z+A*B
1220 N=N+1
```

```
1230 A=A+S1
1240 NEXT E3
1250 NEXT E
1260 X4=(X2*N-X1*X1)
1270 F(E1)=(Z*N-Y*X1)/X4
1280 G(E1)=(X2*Y-X1*Z)/X4
1290 NEXT E1
1300 FOR E=1 TO 2
1310 FOR E1=1 TO N(E)
1320 READ #1,N,H(E,E1),C(E,E1),P(E,E1)
1330 B(E,E1)=G(N)
1340 M(E,E1)=F(N)
1350 NEXT E1
1360 NEXT E
1370 *PRINTS SAMPLE BACKGROUND CORRECTIONS IF DESIRED.
1380 READ #1,M1,M2,M3,M7,M8
1390 IF T$="N" THEN 1540
1400 FOR N=1 TO 2
1410 FOR E1=1 TO N(N)
1420 READ A3
1430 PRINT A3
1440 PRINT,150,H(N,E1),B(N,E1),M(N,E1)
1450 FOR A3=A3 TO A3+2
1460 B1=A3*M(N,E1)+B(N,E1)
1470 PRINT,150, A3;B1
1480 NEXT A3
1490 PRINT
1500 NEXT E1
1510 NEXT N
1520 DATA 50,59,88,110,118,52,76,99,121
1530 *DNTEGRATES PEAKS AND FINDS TWO-THETA ANGLES.
1540 PRINT,230,T9
1550 FOR N3=1 TO 2
1560 A9=0
1570 PRINT,140
1580 PRINT TAB(22+N3);A$(N3)
1590 PRINT,140
1600 PRINT"PLANE    M    SCAT.F       TEMP.F         LP              F";
1610 PRINT"       D-SPACING"
1620 FOR E1=1 TO N(N3)
1630 IF N3<>1 THEN 1670
1640 M7=M7+1
1650 A1=Y(M7)/1000
1660 GOTO 1690
1670 M8=M8+1
1680 A1=Z(M8)/1000
1690 A3=A1
1700 FOR E=1TO500
1710 IF N3<>1 THEN 1750
1720 M7=M7+1
1730 M1=Y(M7)
1740 GOTO 1770
1750 M8=M8+1
1760 M1=Z(M8)
1770 IF M1<0 THEN 1820
1780 B1=M(N3,E1)*A3+BTN3,E1)
```

```
1790 A3=A3+S1
1800 X(E)=(M1-B1)*S1/T
1810 NEXT E
1820 A2=A1
1830 M1=X(1)
1840 M2=A2*X(1)
1850 FOR F1=3 TO E-2 STEP 2
1860 A2=A2+2*S1
1870 M1=M1+4*X(F1-1)+2*X(F1)
1880 M2=M2+4*(A2-S1)*X(F1-1)+2*A2*X(F1)
1890 NEXT F1
1900 F1=(A2+S1-A1)/S1
1910 T(N3,E1)=M1+X(F1)
1920 O(N3,E1)=(M2+(A2+S1)*X(F1))/T(N3,E1)
1930 NEXT E1
1940 *CALCULATES THEORETICAL RELATIVE PATTERN AND COMPARES WITH
1950 *OBSERVED INTEGRATED PATTERN.
1960 FOR E=1 TO N(N3)
1970 D=W1/(2*SIN(P1*O(N3,E)))
1980 A=D*SQR(C(N3,E))
1990 X=1/(4*D*D)
2000 S=15.01*EXP(-4.958*X)+3.573*EXP(-46.50*X)+7.375  'FE SC. FACT.'
2010 S=S-S9
2020 L3=(SIN(P1*O(N3,E)))↑2*COS(P1*O(N3,E))
2030 ON N1 GOTO 2040,2060
2040 L1=(1+(COS(2*P1*52.78)*COS(2*P1*O(N3,E)))↑2)/L3
2050 GOTO 2070
2060 L1=(1+(COS(2*P1*O(N3,E)))↑2)/L3
2070 T1=EXP(-T9*X)
2080 F1=T1*A(N3)*S
2090 U(N3,E)=P(N3,E)*L1*F1*F1
2100 PRINT,110,H(N3,E),P(N3,E),S,T1*T1,L1,F1,D
2110 A9=A9+A
2120 NEXT E
2130 A9=A9/N(N3)
2140 PRINT,140
2150 PRINT TAB(25);"RELATIVE";TAB(37);"RELATIVE";TAB(51);"OBSERVED"
2160 PRINT"PLANE          TWO-THETA      CALC.I      OBSERVED I    INTEGRATED I
2170 FOR E=1 TO N(N3)
2180 U(N3,E)=U(N3,E)/A9↑6
2190 M2=U(N3,E)/U(N3,1)*100
2200 A1=T(N3,E)/T(N3,1)*100
2210 PRINT,150,H(N3,E),O(N3,E),M2,A1,T(N3,E)
2220 NEXT E
2230 PRINT
2240 PRINT,160,A9
2250 NEXT N3
2260 FOR N3=1,2
2270 F(N3)=0
2280 G(N3)=0
2290 FOR E=1 TO N(N3)
2300 *CALC. AVERAGE,VARIANCES,CONFIDENCE INTERVAL
2310 M1=T(N3,E)/U(N3,E)
2320 F(N3)=F(N3)+M1
2330 G(N3)=G(N3)+M1*M1
2340 NEXT E
```

```
2350 F(N3)=F(N3)/N(N3)
2360 D(N3)=(G(N3)-F(N3)*F(N3)*N(N3))/(N(N3)-1)
2370 NEXT N3
2380 V=1/(1+F(2)/F(1))
2390 V3=100*V*(1-V)*SQR(D(1)/F(1)↑2+D(2)/F(2)↑2)
2400 V=V*100
2410 PRINT,170,V,V3
2425 V3=2.5*V3/SQR(N(1)+N(2)) 'VALID FOR APPROX. 5 TO 7 DEG. OF FREE.'
2430 PRINT,190,V-V3,V+V3
2440 PRINT, 200
2450 PRINT, 210
2460 FOR E=1 TO N(2)
2470 FOR F=1 TO N(1)
2480 R1=T(1,F)*U(2,E)/(T(1,F)*U(2,E)+T(2,E)*U(1,F))*100
2490 PRINT,220,H(1,F),H(2,E),R1
2500 NEXT F
2510 NEXT E
2515 PRINT,140
2520 END
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16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

A method for determining the relative phase volume of austenite in an austenite-ferrite mixture is described in detail. Results obtained by applying this method to an austenite-ferrite stainless steel composite material are presented. The method can be extended to other multi-phase materials.

17. KEY WORDS (Alphabetical order, separated by semicolons) Austenite; computer; ferrite; phase measurement; quantitative analysis; retained austenite; x-ray diffraction.

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