Correction Factors For On-Line Microprobe Analysis of Multielement Alloy Systems

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PRE FACE

An on-line correction technique has been developed for the conversion of electron probe X-ray intensities into concentrations of emitting elements. This technique consisted of off-line calculation and representation of binary interaction data which were read into an on-line minicomputer to calculate variable correction coefficients. These coefficients were used to correct the X-ray data without significantly increasing computer core requirements. The polynomial coefficients representing most of the common binary interactions at different accelerating potentials were generated and are included. Results are presented for the analyses of several alloy standards to demonstrate the applicability of this correction procedure.
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INTRODUCTION

Electron probe microanalysis normally consists of (1) collecting characteristic X-ray intensities from a specimen as well as from standards for each of the elements in the specimen and (2) performing corrections to convert the experimental intensities into concentrations of emitting elements. These corrections are usually performed on a large off-line computer by using programs such as MAGIC IV (ref. 1) or COR 2 (ref. 2). However, with the advent of automated microprobes equipped with minicomputers, on-line correction of the experimental intensities is desirable. If a sequence of measurements at various points of a specimen surface is being performed, on-line data correction is particularly helpful to the operator in selecting the next step. Typically, the minicomputers used for microprobe automation are large enough to provide hardware control and data collection from all spectrometers and still have sufficient memory to execute small- to moderate-size programs. However, programs such as MAGIC IV and COR 2, which have been widely used for off-line data correction, require large core storage and, therefore, are not suitable for on-line data correction with minicomputers.

Two basically different approaches have been pursued in the development of on-line correction procedures. One approach has been to reduce the memory requirement of one of the large correction programs by making the simplifications and approximations necessary to make the program fit within the available memory of the minicomputer. This approach was taken by Yakowitz, Myklebust, and Heinrich (ref. 3) in the development of an on-line correction program entitled "FRAME."

An alternate approach has been twofold: first, to perform the lengthy and time-consuming corrections for binary pairs of elements off-line by using a large correction program such as MAGIC IV and, second, to use the results of these calculations in an on-line correction program requiring only a minimal amount of core storage. This "hybrid" (off-line—on-line) approach has been taken in the present study because it preserves the accuracy of large correction programs. This approach originated with early work by Ziebold and Ogilvie (ref. 4) and was subsequently refined by Bence and Albee (ref. 5), Bolon and Lifshin (ref. 6), and Leitner (ref. 7). Leitner represented the data for each binary interaction by polynomial coefficients obtained by least-squares fitting a third-order polynomial for C/K as a function of K, where C and K are the concentration and relative intensity of the element being considered. This method accurately describes most binary calibration curves. However, for binaries with strong absorption or fluorescence effects, the polynomial for C/K as a function of K does not accurately describe the calibration curve.

The current investigation was undertaken to (1) develop an improved method of representing the calibration curves for binaries in which absorption and fluorescence produce significant nonlinearity in the binary calibration curve, (2) incorporate these results into a modified Ziebold and Ogilvie correction scheme, and (3) demonstrate the applicability of this hybrid approach.

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SYMBOLS

C composition in weight fraction
C/K ratio of C to K derived from MAGIC IV program
K intensity measured for an element in unknown sample divided by intensity measured from pure element standard
k intensity of characteristic X-ray line
n total number of elements in alloy ABC...N
p polynomial coefficient
α binary interaction coefficient
β multicomponent correction factor describing combined influence of all elements taken together

Subscripts:

A element A in binary alloy A-B
B element B in binary alloy A-B
AB effect of element B on X-ray intensity emitted by element A
ABC...N multicomponent alloy consisting of n elements
i ith element in multicomponent alloy ABC...N
j jth element in multicomponent alloy ABC...N
ij effect of jth element on X-ray intensity emitted by ith element

M1,M2,M3 methods 1, 2, and 3 investigated to represent binary calibration curves by polynomial coefficients
0,1,2,3 polynomial coefficient designations (see eq. (6))

Superscripts:

s standard sample
u unknown sample
ANALYSIS DEVELOPMENT

Background

Ziebold and Ogilvie (ref. 4) have shown that calibration curves for many binary metal alloy systems can, within the variance of the data, be described by the relation

\[ \frac{1 - K_A}{K_A} = \alpha_{AB} \frac{1 - C_A}{C_A} \]  

(1)

where \( C_A \) is the weight fraction of element A in binary alloy A-B, \( K_A \) is intensity (corrected for dead time and background) of a characteristic radiation line of element A in alloy A-B relative to that of pure element A, and the constant \( \alpha_{AB} \) is the binary interaction coefficient for the effect of element B on the X-ray intensity emitted from element A. Values of \( \alpha \) less than unity generally indicate that fluorescence dominates in the interaction parameter, whereas values greater than unity indicate that absorption dominates. Rearrangement of equation (1) shows a linear relationship for the correction factor with composition

\[ \left( \frac{C_A}{K_A} \right)_{AB} = C_A + \alpha_{AB} C_B \]  

(2)

This linear variation of the correction factor with composition was extended to multicomponent systems by using the compositionally weighted average of the binary interaction coefficients (ref. 4)

\[ \beta_A = \left( \frac{C_A}{K_A} \right)_{ABC...N} = \frac{C_A + \alpha_{AB} C_B + \alpha_{AC} C_C + \alpha_{AD} C_D + \ldots + \alpha_{AN} C_N}{C_A + C_B + C_C + \ldots + C_N} \]  

(3)

This parameter \( \beta_A \) has been referred to as the multicomponent interaction coefficient because it gives the combined effect of all elements in the sample on the intensity emitted from a characteristic X-ray line of element A in the sample. The validity of using binary interaction coefficients to compute multicomponent interaction coefficients has been verified for a number of ternary copper-base alloys by Ingersoll, Taylor, and Derouin (ref. 8).

Although the linear relationship for the calibration curve (eq. (2)) is a good approximation for several binaries, not all binary calibration curves are linear, especially the ones involving significant absorption or fluorescence. A more general polynomial relationship applicable for linear or nonlinear binaries is described in a later section.

On-Line Correction Procedure

If the variation of \( \left( \frac{C_A}{K_A} \right)_{AB} \) with \( C_A \) is known from theory or experiment, the coefficient \( \alpha_{AB} \) can be computed for a given value of \( C_A \) as follows:

\[ \alpha_{AB} = \frac{\left( \frac{C_A}{K_A} \right)_{AB} - C_A}{1 - C_A} \]  

(4)
The assumption is made that a concentration dependent $\alpha_{AB}$ (satisfying eq. (2)) is the appropriate binary interaction parameter to use in equation (3) to determine the multicomponent correction coefficient $\beta_A$. Thus, if $(C_A/K_A)_{AB}$ is not a linear function of $C_A$, the value of $C_A$ must be known before equation (4) and, hence, equation (3) can be solved for $\alpha_{AB}$ and $\beta_A$. However, the purpose for computing $\alpha_{AB}$ and $\beta_A$ is to estimate $C_A$ by using

$$C_A = \beta_A K_A$$  \hspace{1cm} (5)

Therefore, an iterative method of solution is used to determine the concentrations of each of the elements in the sample.

A general correction scheme using these relationships was developed to perform an on-line ZAF (atomic-number-absorption-fluorescence) correction of microprobe data. The essential elements of this correction scheme are as follows:

1. The X-ray intensities are measured from all the standards and are corrected for dead time and background to give $k_i^S$.

2. The X-ray intensities are measured from all elements in the sample and are corrected for dead time and background to give $k_i^U$.

3. The relative intensity $K_i$ of each element in the specimen is calculated by taking the ratio of $k_i^U$ to $k_i^S$.

4. As a first approximation, the unknown concentrations $C_i$ are estimated as

$$K_i = \frac{\sum_{j=1}^{n} K_j}{\sum_{j=1}^{n} K_j}.$$  \hspace{1cm} (6)

5. The binary calibration polynomials, calculated off-line with a ZAF correction program, are solved for each binary permutation at $C_i$ to give $(C_i/K_i)_{ij}$.

6. The binary interaction coefficients $\alpha_{ij}$ are calculated by using (eq. (4))

$$\alpha_{ij} = \frac{[(C_i/K_i)_{ij} - C_i]}{(1 - C_i)}.$$  \hspace{1cm} (7)

7. The multicomponent correction factors $\beta_i$ are computed by using equation (3).

8. The unknown concentrations $C_i$ are calculated by multiplying $K_i$ by $\beta_i$ (eq. (5)).

9. Steps 7 and 8 are repeated until no significant changes occur in $C_i$.

10. The concentrations $C_i$ are used in steps 5 to 9 to compute refined $(C_i/K_i)_{ij}$, $\alpha_{ij}$, $\beta_i$, and $C_i$. This process is repeated until no significant changes occur in the elemental concentrations $C_i$. 


Off-Line Representation of Binary Calibration Curves

In order to perform the on-line correction scheme outlined, calibration data are required for all binary permutations in the specimen under consideration. A quantitative microprobe analysis program entitled "MAGIC IV" developed by Colby (ref. 1) was used for this purpose. MAGIC IV was written to perform a ZAF correction to convert the measured intensities into chemical compositions. MAGIC IV was also programed to run in a reverse mode where intensity ratios could be calculated from known concentrations. This program was modified so that any number of binary permutations of elements could be treated in the reverse mode. Intensity ratios were calculated for 22 compositions (C = 0.01, 0.05, 0.10, . . . , 0.95, 0.99, 1.00) for each binary pair.

An example of the type of results calculated is shown in figure 1. The C/K for boron Kα radiation is plotted as a function of boron concentration for the B-Si system at four different accelerating potentials. Because the boron Kα intensity is strongly absorbed by silicon, the values of C/K in the low concentration range are large. For 20 kV, the value of C/K varies from 290 on the low concentration end to unity at C = 1. Although the span in C/K is greatly reduced at lower excitation potentials, the variation in C/K is still much larger than generally found for binary systems where the elements have nearly equal atomic weights.

In order to minimize the memory required to execute the on-line correction scheme, each binary calibration curve must be represented by as few terms as possible. This is conveniently done by least-squares curve fitting a third-order polynomial through the calibration data. A subroutine was added to MAGIC IV program to accomplish this. Table 1 shows polynomial coefficients for the effect of element B on the intensity from element A determined for several binary interactions at 20 kV accelerating potential. These coefficients are shown for two different methods of representing C/K; that is,

\[ C/K = p_0 + p_1 C + p_2 C^2 + p_3 C^3 \]  

(6)

\[ C/K = p_0 + p_1 K + p_2 K^2 + p_3 K^3 \]  

(7)

As C approaches unity, K will approach unity; therefore, C/K should converge to unity as C or K approaches unity. Thus the sum of the polynomial coefficients in both cases should be unity, if they are usable for specimens where C approaches unity. As can be seen from table 1, the sums for C/K as a function of C are, in general, closer to unity than the corresponding sums for C/K as a function of K. Therefore, C/K as a function of C should better represent the calibration data.

The effect of elements such as titanium or silicon on the X-ray intensity of elements such as boron or carbon is so severe at 20 kV accelerating potential that even the polynomial coefficients for C/K as a function of C cannot accurately describe the interaction as C approaches unity. This is illustrated in table 2 where C is the concentration of boron in B-Si binary, K is the corresponding X-ray intensity of boron Kα from the sample relative to pure boron, and C/K is the resultant ratio. The values of \((C/K)_{M1}\) were obtained from equa-
tion (6) by using the polynomial coefficients of the third-order least-squares fit of the 22 data points of C/K as a function of C. Ideally, the values of (C/K)M and C/K should be the same at all data points. Although the deviations for C < 0.8 are within 1.0 percent, the deviation is about 94 percent as C approaches unity. Because the percentage deviation in the value of C computed by the correction procedure is directly proportional to the percentage deviation of (C/K)M from C/K, the high percentage deviations as C approaches unity must be minimized. Several computational methods were investigated to achieve this requirement.

In order to decrease the percentage deviation in the range C > 1, a constrained least-squares third-order polynomial was fitted by using Lagrange multipliers with C/K = 1 at C = 1 as the constraint. This approach forced the value of C/K to be unity at C = 1 but gave a maximum deviation of about 20 percent in the range C > 0.8. Another approach investigated was to fit a third-order least-squares polynomial for $\sqrt{C/K}$ as a function of C. This approach reduced the range in the function being fit by an order of magnitude and, hence, resulted in an improved fit. The results are shown as (C/K)M in table 2 where the maximum deviation is about 6.8 percent, occurring at C = 1.00. In order to further reduce this deviation, a correction term was added to (C/K)M which forced the value of (C/K)M to be unity at C = 1 and placed a decreasing correction for decreasing values of C. Results calculated by this approach are shown as (C/K)M in table 2. The values of (C/K)M were computed as

$$ (C/K)M = \left[ p_0 + p_1C + p_2C^2 + p_3C^3 + (1 - p_0 - p_1 - p_2 - p_3)C^{80} \right]^2 \quad (8) $$

where $p_0$, $p_1$, $p_2$, and $p_3$ are the polynomial coefficients obtained by least-squares fitting $\sqrt{C/K}$ as a function of C. The power of 80 in the correction term was chosen so that it placed an optimum localized correction for several systems investigated. As can be inferred from table 2, this approach using (C/K)M reduced the maximum deviation to within 2 percent.

Table 3 shows C/K and (C/K)M for the effect of titanium on calcium Kα at 20 kV accelerating potential. In this system, C/K increases with increasing C, as opposed to the reverse situation observed for silicon on boron Kα. The values of (C/K)M are in excellent agreement with the values of C/K determined from MAGIC IV. Several other binary combinations were checked, and in all cases the (C/K)M approach adequately described the binary calibration curves. Therefore, the polynomial coefficients for $\sqrt{C/K}$ as a function of C were generated for the most common binary interactions by using the (C/K)M approach and are presented in tables A1, A2, and A3. These tables are described and their use is discussed in the appendix.

**EXPERIMENTAL PROCEDURE**

In order to verify the correction procedure, several alloy standards were analyzed on an Applied Research Laboratories (ARL) electron microprobe. A minicomputer (16 K memory) with ARL supplied software was used for hardware control, data collection, and data correction. Several modifications were made to the software to incorporate the present correction scheme, to facilitate input opera-
tions, and to extend the applicability to more alloy systems. The required polynomial coefficients were determined off-line with the modified MAGIC IV program and punched on paper tape for input into the on-line computer. A single tape was used for any alloy containing any combination of elements for which data were included on the tape. Therefore, every alloy system did not require a different data tape. Also, the coefficients were not read in any particular order. The on-line program arranged them as required. The standard samples analyzed are listed in the following table:

<table>
<thead>
<tr>
<th>Material</th>
<th>Number of samples</th>
<th>Number of elements</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel and cobalt base superalloys</td>
<td>4</td>
<td>9</td>
<td>Cabot Corporation Stellite Division Kokomo, Indiana</td>
</tr>
<tr>
<td>Monel</td>
<td>3</td>
<td>8</td>
<td>International Nickel Co., Inc. Huntington Alloy Products Division Huntington, West Virginia</td>
</tr>
<tr>
<td>Low carbon steel</td>
<td>1</td>
<td>8</td>
<td>National Bureau of Standards</td>
</tr>
<tr>
<td>Free machining brass</td>
<td>1</td>
<td>6</td>
<td>Washington, D.C.</td>
</tr>
<tr>
<td>Waspaloy</td>
<td>1</td>
<td>10</td>
<td>(certified analyses)</td>
</tr>
<tr>
<td>René 41</td>
<td>1</td>
<td>10</td>
<td></td>
</tr>
</tbody>
</table>

The nickel and cobalt base superalloys and monel samples were supplied by Applied Research Laboratories and are the same samples analyzed and reported on by Leitner (ref. 7).

The electron microprobe was operated at 20 kV accelerating potential and 0.1 μA sample current. Pure element calibration standards were used. In order to compensate for sample inhomogeneities, the beam was slightly defocused and X-ray data were collected from several different locations on the samples. The experimental X-ray intensities were corrected both on-line with the procedure outlined in the previous sections and off-line with the MAGIC IV program.

RESULTS AND DISCUSSION

The elemental concentrations determined by the present on-line scheme and the off-line MAGIC IV program are given in tables 4 to 9 for all samples analyzed. The reported concentrations supplied with the samples are also given in the tables. The concentrations determined by the on-line procedure are in good agreement with those calculated with MAGIC IV and with the reported values. The absolute differences between the reported weight percent concentrations and present analyses are less than 1.1 weight percent. Also, the differences in the weight percent concentrations between MAGIC IV and present analyses are less than 0.5 weight percent. These differences are well within the accuracy generally ascribed to microprobe analyses.
Tables 4 to 9 include analyses on 15 different elements. A total of 92 elemental analyses were performed, 37 of which represented elements whose reported concentration values were less than 1 weight percent and 29 of which represented elements with 10 weight percent concentration or greater. Table 10 summarizes the relative errors in the present analyses with respect to MAGIC IV analyses and reported values. For the purpose of comparison, the weight percent concentration is divided into three intervals (<1, 1 to 10, and >10), and the percent of the present analyses of each concentration range that is within a given deviation (±2 percent, ±5 percent, ±10 percent) are listed. A ±10 percent deviation for 0.5 weight percent concentration corresponds to 0.5 ± 0.05. Therefore, ±10 percent may be a reasonable deviation for analyses with concentrations less than 1 weight percent. Similarly, ±5 percent for 1 to 10 weight percent concentration and ±2 percent for greater than 10 weight percent concentration may be reasonable deviations. These combinations are denoted by asterisks in table 10. As can be seen, the present analyses gave results which are in good agreement with MAGIC IV results. Also, the agreement for <1 weight percent concentration range was as good as the agreement for >10 weight percent. The present technique gave concentration values in good agreement with the reported values for elements which were present in excess of 10 weight percent, but the agreement was not as good for lower concentrations. However, comparison of the present results with the MAGIC IV results is the significant test of the on-line correction procedure. Comparisons with reported concentrations reflect the overall accuracy which comprises the experimental procedures as well as the correction procedures.

CONCLUDING REMARKS

An on-line correction technique has been developed for the conversion of electron probe X-ray intensities into concentrations of emitting elements. This technique consisted of off-line calculation and representation of binary interaction data and on-line incorporation of variable correction coefficients without significantly increasing computer core requirements. The binary interaction data were obtained by running Colby's MAGIC IV program in the reverse mode. The data for each binary interaction were represented by polynomial coefficients obtained by least-squares fitting a third-order polynomial for $\sqrt{C/K}$ as a function of C. These polynomial coefficients were generated for most of the common binary interactions at different accelerating potentials and were tabulated. The on-line correction was performed by a minicomputer which also handled electron-probe hardware control and data collection.

Experimental data were collected on different alloy standards to demonstrate the applicability of the present correction procedure. In all cases, the present scheme gave compositions which were within 1.1 of the reported weight percent concentrations. Also, the experimental intensity ratios were input into MAGIC IV program off-line, and the resulting concentrations were in good agreement with the present analyses. These comparisons demonstrated that the present technique
for the on-line correction of microprobe data with a minicomputer of modest memory gives accuracy comparable with large off-line correction programs.

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REFERENCES


### TABLE 1.- POLYNOMIAL COEFFICIENTS FOR EFFECT OF ELEMENT B ON $K_\alpha$ X-RAY INTENSITY FROM ELEMENT A AT 20 kV ACCELERATING POTENTIAL

<table>
<thead>
<tr>
<th>Element A</th>
<th>Element B</th>
<th>$p_0$</th>
<th>$p_1$</th>
<th>$p_2$</th>
<th>$p_3$</th>
<th>$\sum_{i=0}^{3} d_i$</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>B</td>
<td>46.347</td>
<td>-82.844</td>
<td>41.679</td>
<td>-4.186</td>
<td>0.996</td>
</tr>
<tr>
<td>Si</td>
<td>B</td>
<td>0.965</td>
<td>0.024</td>
<td>0.015</td>
<td>-0.006</td>
<td>1.000</td>
</tr>
<tr>
<td>Ti</td>
<td>B</td>
<td>1.080</td>
<td>-0.060</td>
<td>-0.022</td>
<td>0.001</td>
<td>1.000</td>
</tr>
<tr>
<td>B</td>
<td>C</td>
<td>2.938</td>
<td>-2.294</td>
<td>0.292</td>
<td>0.063</td>
<td>1.000</td>
</tr>
<tr>
<td>Si</td>
<td>C</td>
<td>1.107</td>
<td>-0.126</td>
<td>0.030</td>
<td>-0.011</td>
<td>1.000</td>
</tr>
<tr>
<td>Ti</td>
<td>C</td>
<td>1.158</td>
<td>-0.129</td>
<td>-0.030</td>
<td>0.001</td>
<td>1.000</td>
</tr>
<tr>
<td>B</td>
<td>Si</td>
<td>290.069</td>
<td>-365.112</td>
<td>-51.609</td>
<td>126.713</td>
<td>0.600</td>
</tr>
<tr>
<td>C</td>
<td>Si</td>
<td>34.656</td>
<td>-39.619</td>
<td>-4.820</td>
<td>10.724</td>
<td>0.941</td>
</tr>
<tr>
<td>Ti</td>
<td>Si</td>
<td>1.211</td>
<td>-0.221</td>
<td>0.013</td>
<td>-0.003</td>
<td>1.000</td>
</tr>
<tr>
<td>B</td>
<td>Ti</td>
<td>38.814</td>
<td>-33.914</td>
<td>-19.621</td>
<td>15.578</td>
<td>0.958</td>
</tr>
<tr>
<td>C</td>
<td>Ti</td>
<td>6.840</td>
<td>-4.608</td>
<td>-2.344</td>
<td>1.105</td>
<td>0.993</td>
</tr>
<tr>
<td>Si</td>
<td>Ti</td>
<td>1.321</td>
<td>-0.260</td>
<td>-0.068</td>
<td>0.006</td>
<td>1.000</td>
</tr>
</tbody>
</table>

C/K = $p_0 + p_1 c + p_2 c^2 + p_3 c^3$

<table>
<thead>
<tr>
<th>Element A</th>
<th>Element B</th>
<th>$p_0$</th>
<th>$p_1$</th>
<th>$p_2$</th>
<th>$p_3$</th>
<th>$\sum_{i=0}^{3} d_i$</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>B</td>
<td>30.660</td>
<td>-250.935</td>
<td>541.031</td>
<td>-324.184</td>
<td>-3.428</td>
</tr>
<tr>
<td>Si</td>
<td>B</td>
<td>0.965</td>
<td>0.023</td>
<td>0.016</td>
<td>-0.005</td>
<td>1.000</td>
</tr>
<tr>
<td>Ti</td>
<td>B</td>
<td>1.080</td>
<td>-0.065</td>
<td>-0.021</td>
<td>0.005</td>
<td>1.000</td>
</tr>
<tr>
<td>B</td>
<td>C</td>
<td>2.895</td>
<td>-5.243</td>
<td>5.951</td>
<td>-2.622</td>
<td>0.980</td>
</tr>
<tr>
<td>Si</td>
<td>C</td>
<td>1.107</td>
<td>-0.138</td>
<td>0.046</td>
<td>-0.015</td>
<td>1.000</td>
</tr>
<tr>
<td>Ti</td>
<td>C</td>
<td>1.158</td>
<td>-0.151</td>
<td>-0.017</td>
<td>0.009</td>
<td>1.000</td>
</tr>
<tr>
<td>B</td>
<td>Si</td>
<td>161.451</td>
<td>-2049.053</td>
<td>4604.001</td>
<td>-2718.706</td>
<td>-2.307</td>
</tr>
<tr>
<td>C</td>
<td>Si</td>
<td>25.971</td>
<td>-198.415</td>
<td>416.562</td>
<td>-246.316</td>
<td>-2.198</td>
</tr>
<tr>
<td>Ti</td>
<td>Si</td>
<td>1.210</td>
<td>-0.266</td>
<td>0.070</td>
<td>-0.014</td>
<td>1.000</td>
</tr>
<tr>
<td>B</td>
<td>Ti</td>
<td>29.602</td>
<td>-231.621</td>
<td>479.422</td>
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C/K = $p_0 + p_1 k + p_2 k^2 + p_3 k^3$
TABLE 2.- THREE REPRESENTATIONS OF CALIBRATION CURVE FOR EFFECT OF SILICON ON BORON K\(_{\alpha}\) AT 20 kV ACCELERATING POTENTIAL

<table>
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<th>C</th>
<th>K</th>
<th>C/K</th>
<th>((C/K)_{M1})</th>
<th>((C/K)_{M2})</th>
<th>((C/K)_{M3})</th>
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<td>0.2864E+03</td>
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<td>0.2720E+03</td>
<td>0.2720E+03</td>
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<td>0.2532E+03</td>
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<td>0.2525E+03</td>
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<td>0.2160E+03</td>
<td>0.2148E+03</td>
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Note: The symbol E is used to denote times 10 to the power.
### Table 3. Representation of Calibration Curve for Effect of Titanium on Calcium $K\alpha$ at 20 kV Accelerating Potential

<table>
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<tr>
<th>C</th>
<th>K</th>
<th>C/K</th>
<th>(C/K)$_{M3}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
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<td>0.7688</td>
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<tr>
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<td>.8090</td>
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### Table 4. Analysis of Low Carbon Steel at 20 kV Accelerating Potential

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<tbody>
<tr>
<td></td>
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<td>MAGIC IV</td>
</tr>
<tr>
<td>Fe</td>
<td>0.6393</td>
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<td>Cr</td>
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TABLE 5.- ANALYSIS OF WASPALOY AT 20 kV ACCELERATING POTENTIAL

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<td>Cu</td>
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TABLE 6.- ANALYSIS OF FREE MACHINING BRASS AT 20 kV ACCELERATING POTENTIAL

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<td>Ni</td>
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<td>Element</td>
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<td>Weight percent</td>
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### TABLE 8. - ANALYSES OF MONEL AT 20 kV ACCELERATING POTENTIAL

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TABLE 9.- ANALYSES OF NICKEL AND COBALT BASE SUPERALLOYS
AT 20 kV ACCELERATING POTENTIAL

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| Ni      | 0.5459            | 54.21          | 54.23          | 54.10          |
| Mo      | 0.1240            | 16.08          | 16.08          | 16.10          |
| Cr      | 0.1750            | 16.43          | 16.88          | 16.32          |
| Fe      | 0.0546            | 4.69           | 5.02           | 5.05           |
| W       | 0.0323            | 4.21           | 4.19           | 4.42           |
| Co      | 0.0171            | 1.71           | 1.74           | 1.74           |
| Si      | 0.0028            | 0.48           | 0.48           | 0.59           |
| Mn      | 0.0067            | 0.59           | 0.63           | 0.55           |
| V       | 0.0023            | 0.22           | 0.22           | 0.21           |

| Ni      | 0.1052            | 10.01          | 10.03          | 9.80           |
| Mo      | 0.0052            | 0.68           | 0.70           | 0.72           |
| Cr      | 0.2231            | 19.89          | 20.41          | 19.95          |
| Fe      | 0.0231            | 2.16           | 2.23           | 2.33           |
| W       | 0.1115            | 14.12          | 14.13          | 14.26          |
| Co      | 0.4892            | 48.84          | 48.99          | 49.88          |
| Si      | 0.0031            | 0.51           | 0.51           | 0.94           |
| Mn      | 0.0216            | 1.77           | 1.91           | 1.79           |

| Ni      | 0.0089            | 0.39           | 0.38           | 0.31           |
| Mo      | 0.0077            | 0.98           | 0.99           | 0.91           |
| Cr      | 0.2900            | 26.04          | 26.26          | 26.44          |
| Fe      | 0.0341            | 3.05           | 3.11           | 3.27           |
| W       | 0.0395            | 5.09           | 5.09           | 5.36           |
| Co      | 0.5858            | 60.51          | 60.55          | 61.02          |
| Si      | 0.0074            | 1.25           | 1.24           | 1.39           |
| Mn      | 0.0057            | 0.46           | 0.50           | 0.45           |
### TABLE 10. — RELATIVE ERRORS IN PRESENT ON-LINE ANALYSES WITHIN GIVEN PERCENT DEVIATION FROM MAGIC IV OR REPORTED VALUES

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<th>Range of weight percent concentration</th>
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<td>64</td>
<td>*80</td>
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<tr>
<td>&gt;10</td>
<td>*97</td>
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*Reasonable deviation.*
Figure 1.- Effect of silicon on boron $K_{\alpha}$ intensity at different accelerating potentials, computed by using MAGIC IV program with normal beam incidence and 52.5° X-ray emergence angle.
This appendix presents tabulated polynomial coefficients for the common binary interactions for different acceleration potentials. The elements and their appropriate X-ray lines were selected so that these tables are concise and yet include most of the common binary interactions.

For each binary interaction, X-ray intensity ratios were computed for 22 concentration values by running Colby's MAGIC IV program (ref. 1) in the reverse mode. These concentration values were 0.01, 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45, 0.50, 0.55, 0.60, 0.65, 0.70, 0.75, 0.80, 0.85, 0.90, 0.95, 0.99, and 1.00. Several methods were investigated to adequately represent the calibration data for each binary interaction by as few terms and constants as possible. Least-squares fitting of the calibration data with the equation

\[
\sqrt{C/K} = p_0 + p_1 C + p_2 C^2 + p_3 C^3 \quad (A1)
\]

gave the best results. Although the polynomial coefficients thus obtained represented the calibration data rather accurately for values of \( C \) not approaching unity, they did not adequately represent the range \( C \rightarrow 1 \). However, use of the equation

\[
\frac{C}{K} = \left( \left( p_0 + p_1 C + p_2 C^2 + p_3 C^3 \right) + \left[ (1 - p_0 - p_1 - p_2 - p_3)C^{80} \right] \right)^2 \quad (A2)
\]

with \( p_0, p_1, p_2, \) and \( p_3 \) from equation (A1) gave values of \( C/K \) which were in excellent agreement (±2 percent deviation) with the calibration values. The correction term in the brackets forces the value of \( C/K \) to be unity at \( C = 1 \) and places a decreasing correction for decreasing \( C \) values where the fit is already adequate.

The polynomial coefficients \( p_0, p_1, p_2, \) and \( p_3 \) are listed in tables A1, A2, and A3 for several binary interactions at 10, 15, and 20 kV accelerating potentials for normal beam incidence and an X-ray emergence angle of 52.5°. These coefficients correspond to the effect of element B (listed under column B) on the X-ray intensity from element A (listed under column A). Under column A, the lines \( K_\alpha, L_\alpha, \) and \( M_\alpha \) are denoted by \( KA, LA, \) and \( MA; \) for example, \( BKA \) denotes the boron \( K_\alpha \) line. The symbols \( Z_A \) and \( Z_B \) denote the atomic numbers of elements \( A \) and \( B, \) and should be helpful in searching for the appropriate interaction. The wavelengths in angstroms of the affected X-ray lines are listed under \( \lambda_A. \) These values should be helpful in selecting the proper spectrometer crystal for the analysis of element A. Comparison of the \( p_0 \) values for a given interaction at different acceleration potentials can aid the probe operator in choosing an optimum acceleration potential for analysis of the sample. The amount by which \( p_0 \) deviates from unity is a direct indication of the extent of the corrections involved in converting X-ray intensity data to chemical compositions.
APPENDIX

The polynomial coefficients are read into the minicomputer in the form of a punched paper tape representing as many as 170 binary interactions at a given accelerating potential. Once a data tape is made up representing common interactions, it can be used repeatedly, thus avoiding the need to make up a new data tape for every alloy system.
**APPENDIX**

**TABLE A1. - POLYNOMIAL COEFFICIENTS FOR 20 kV ACCELERATING POTENTIAL**

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## APPENDIX

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## APPENDIX

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## APPENDIX

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### APPENDIX

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## APPENDIX

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### APPENDIX

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### APPENDIX

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**Title and Subtitle**

CORRECTION FACTORS FOR ON-LINE MICROPROBE ANALYSIS OF MULTIELEMENT ALLOY SYSTEMS

**Author(s)**

Jalaiah Unnam, Darrel R. Tenney, and William D. Brewer

**Performing Organization**

NASA Langley Research Center
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**Supplementary Notes**

Jalaiah Unnam: Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

**Abstract**

An on-line correction technique has been developed for the conversion of electron probe X-ray intensities into concentrations of emitting elements. This technique consisted of off-line calculation and representation of binary interaction data which were read into an on-line minicomputer to calculate variable correction coefficients. These coefficients were used to correct the X-ray data without significantly increasing computer core requirements. The binary interaction data were obtained by running Colby's MAGIC IV program in the reverse mode. The data for each binary interaction were represented by polynomial coefficients obtained by least-squares fitting a third-order polynomial for \( \sqrt{C/K} \) as a function of \( K \), where \( C \) and \( K \) are the concentration and relative intensity of the element being considered. These polynomial coefficients were generated for most of the common binary interactions at different accelerating potentials and are included. Results are presented for the analyses of several alloy standards to demonstrate the applicability of this correction procedure.
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