Utilization of a Personal Computer for the Microstructural Analysis of a Duplex Stainless Steel

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The combination of a personal computer and EPMA has been utilized in order to develop a simplified microanalysis equipment system which has nearly the same analyzing power in specifications as CMA or MA in image analysis.

Image analyzed data for a duplex stainless steel 329J1 from the present system was compared with that from CMA and found to be in fairly good agreement with color mapping of the concentrations of Cr and Ni.

The phase identification for α and γ phases by Cr/Ni partition ratio was also accomplished.

KEY WORDS: image analysis; personal computer; EPMA; duplex stainless steel; phase identification; color mapping.

1. Introduction

Computer Aided Microanalyzer (CMA) and MacroAnalyzer (MA) were recently developed for the qualitative and quantitative evaluation of metallic microstructures.^{1,2)} These analyzers consist of a skillful combination of EPMA (electron probe micro-analyzer) with image processing, and are effective tools for microstructure analysis because of very rapid response times and excellent sensitivity in a relatively wide area of microstructures.

In the present work, a trial was made utilizing the combination of a personal computer with EPMA for microanalysis which has nearly the same analyzing power as CMA in specifications in order to develop a more conventional and lower cost analyzing equipment system. Some applications and limitations of usage are also discussed.

2. Experimental Procedure

The EPMA (JCXA-733) and the personal computer (PC, NEC-PC9801VM2) were connected by RS232C cables as shown in Fig. 1. The characteristic X-ray signal for a specific element existing on the surface of a specimen is input into the PC directly from the EPMA which was driven in surface scanning mode under the following operating conditions: accelerating voltage 15 kV, emission current 6 nA, beam diameter 2 μ m, counting time per a point 2 s. A duplex stainless steel 329J1 composed of α and γ phases in Table 1 was used for the image analysis of microstructure. The specific elements to be measured were Cr and Ni, and the X-ray signals of these elements were directly input into the PC and then converted to concentrations after ZAF calibration³⁾ in the PC.

Multi-faced outputs are available using these data. Concentration distribution of 329J1 was color-mapped in 16 levels in different colors. The area fraction for

Fig. 1. Schematic illustration of a newly developed image analyzer composed of a personal computer and EPMA.

Table 1. Chemical composition of specimens used.

	Mn	Cu	Ni	Cr	V	Mo	C	P
329J1	—	—	4.25	24.72	—	1.83	0.016	0.032
16 Cr	2.88	0.11	9.9	16.1	0.032	0.12	—	—
18.5 Cr	3.38	0.14	9.4	18.5	0.064	0.12	—	—
20.5 Cr	4.62	0.24	10.1	20.5	0.12	0.23	—	—

each concentration level was also calculated. The identification of α and γ phases through the partition ratio of Cr/Ni and the estimation of area fraction in both phases was also conducted. Some austenitic stainless steels, listed in Table 1, were also used in order to compare the analytical value of Cr obtained by this system with that of chemical analysis.

3. Results and Discussion

The microstructure of 329J1 solution-treated for 3.6 ks at 1 573 K, followed by γ transformation at 1 273 K for 21.6 ks and etched by Villera reagent is shown in Fig. 2. Spheroidal or rodlike precipitates are γ phases and the matrix is α phase.

Fig. 3 shows the relation between concentrations of

Fig. 2. Microstructure of 329J1 stainless steel solution-treated at 1573 K followed by γ formation at 1273 K for 21.6 ks.

Table 2. Example of analysis data input to PC9801 from EPMA.

<i>X</i>	<i>Y</i>	<i>XR</i> (2)	<i>XR</i> (3)
16 124	31 782	3 330	1 147
16 126	31 782	3 407	1 131
16 128	31 782	3 418	1 212
16 130	31 782	3 166	645
16 132	31 782	3 313	563
16 134	31 782	3 284	563
16 136	31 782	3 222	574
16 138	31 782	3 281	545
16 140	31 782	3 239	557
16 142	31 782	3 241	582
16 144	31 782	3 181	563
16 146	31 782	3 271	580

Fig. 3. Relation between Ni and Cr concentration and counting time of X-ray signal in 329J1 stainless steel.

Ni and Cr and X-ray counting time. The longer the counting time, the smaller the scatter of concentrations. From the viewpoint of prompt image processing, 2 s counting was adopted, since in the range from 2 to 20 s counting, the mean values were within the range of allowances.

X-ray intensity data input from the EPMA to the PC is shown in Table 2. Columns, *X* and *Y*, show positions analyzed on the *X* and *Y* axes. *XR*(2) and *XR*(3) are the *X*-ray intensity of Cr and Ni, respectively. The values in the *X* and *Y* columns show that the electron beam whose diameter is fixed at 2 μ m was driven laterally along the *X* axis and the characteristic *X*-ray signals generated for each 2 μ m square and integrated for 2 s are listed. These data were converted to concentrations by ZAF (Atomic number correction, Absorption correction and Fluorescence correction) calibration.³⁾

The flow chart of image analysis in the present system is shown in Fig. 4. Figs. 5 and 6 show the color mapping of the concentrations of Cr and Ni in the microstructure of 329J1, respectively, for 2 s counting. In figures composed of 51 $\times$ 51 fine picture

Fig. 4. Flow chart in image analyzing of the present system.

Fig. 5. Color mapping of Cr concentration in 329J1 stainless steel by the present system. (A.F.: Area fraction)

Fig. 6. Color mapping of Ni concentration in 329J1 stainless steel by the present system. (A.F.: Area fraction)

elements (pixels) $2\mu\text{m} \times 2\mu\text{m}$ in size, the same as the output of the CMA, visual color presentation of concentration level with 16 colors can be accomplished. Each concentration level, whose range can be set arbitrarily, is colored and measured in area fractions so that estimation of the segregation of atoms is possible. It was found, in comparison with the microstructure in Fig. 2, that Cr segregates in α phase, Ni in γ phase and that each element is not uniformly distributed in each phase. Table 3 shows the concentrations of Cr and Ni in 10 randomly selected point areas and the respective partition ratio of Cr/Ni. The value of Cr/Ni is a measure for identifying both phases since it closely corresponds to the phases which are determined by conventional etching techniques.

In this computer identification of phases, the binarizing threshold is thought to be between 4.052 and 6.476, since the values are between 6.476 and 6.642 in α phase and between 3.920 and 4.052 in γ phase,

Fig. 8. Phase identification by partition ratio (Cr/Ni) 5.5 in 329J1 stainless steel.

clearly identified by the etching. Fig. 7 shows the relation between the threshold value and γ phase area fraction. The optimum threshold value is considered to be 5.5 where the tangential of the curve is minimum and the γ phase area fraction yields 34 %. Fig. 8 is composed of 51×51 picture elements, each element being $2 \mu\text{m}$ square in size, and shows the γ phase presentation obtained at a threshold value of 5.5. Taking into consideration of the magnification in Figs. 2 and 8, this computer-identified picture yields fairly good agreement with the microstructure in Fig. 2. In identifying microstructures by this way, various advanced image analyses, size distributions, mean free paths, *etc.*, are available. The EPMA operating conditions and binarizing threshold, in data treated thus, are of very importance, since the amount of characteristic X-ray signal which is highly dependant upon it is the sole information for estimation of the concentration on the spot. In this sense, enhancing the X-ray flux is effective. However, from the standpoint of rapid image processing, which is a primary concept in image analysis, this demand is incompatible. Accordingly the optimum conditions for image analysis are placed on a balance between these two demands. Fig. 9 shows the reliability in the estimation of chemical composition by the present system when compared with traditional chemical analysis. Determination of the concentration of Ni

Table 3. Cr and Ni concentration and partition ratio in α and γ phases in 329J1 stainless steel.

Position		Cr (wt%)	Ni (wt%)	Cr/Ni	Phase
X	Y				
16 164	31 836	24.131	3.726	6.476	α
16 188	31 836	22.328	5.655	3.948	γ
16 138	31 838	24.182	3.654	6.618	α
16 128	31 844	24.215	3.688	6.566	α
16 156	31 848	22.557	5.567	4.052	γ
16 214	31 848	22.304	5.649	3.948	γ
16 172	31 850	24.548	3.696	6.642	α
16 200	31 858	24.292	3.682	6.598	α
16 166	31 864	22.452	5.728	3.920	γ
16 212	31 872	22.412	5.642	3.972	γ

Fig. 7. Effect of the binarizing condition on area fraction of γ phase in 329J1 stainless steel.

and Cr in this work were made for 10 points selected randomly in austenitic stainless steels (16.1, 18.5 and 20.5 Cr steels). The chemical composition analysis of Ni and Cr by the present system is highly reliable. It is, however, noted that the change in either the operating conditions of the EPMA or the binary threshold value may give rise to some little deviation in the composition analysis results.

Figs. 10 and 11 show the comparison between the present system and the CMA in color mapping of the concentrations of Ni and Cr in 329J1. Area fraction distributions by the present system and the CMA showed the same peak concentration ranges in Cr and Ni. As a whole, both of the image analyzed data were found to be in fairly good agreement, though the values by the present system, especially in Cr, were not in accord with those by the CMA. The difference between these two systems is possibly caused by characteristics of the equipment and operating conditions, further studies will be needed for

Fig. 9. Reliability of the present system for Cr concentration compared with chemical analysis of Cr concentration.

Fig. 10. Comparison of Cr concentrations by the present system and by CMA for 329J1 stainless steel.

Fig. 11. Comparison of Ni concentrations by the present system and by CMA for 329J1 stainless steel.

detailed evaluations for the present system.

In conclusion, the present system (EPMA-PC9801) can be easily introduced by utilizing personal computers and EPMA, and the application of this system for image analysis for small areas and the estimation of precipitation behavior possess high enough merit to compensate for the longer counting time for characteristic X-ray signals.

4. Summary

The utilization of the combination of a personal computer with EPMA was made in order to develop a conventional and low cost microanalysis equipment

system which has nearly the same analyzing power as the CMA, and its application for metallic microstructure was investigated. The main results obtained are as follows;

(1) The analysis data was acceptable for 2 s counting of X-ray signals from the EPMA being operated under the following conditions: accelerating voltage 15 kV, emission current 6 nA, beam diameter 2 μ m.

(2) Color mapping of the concentrations of Ni and Cr and the estimation of area fractions of these elements for a duplex stainless steel were evaluated, and it was confirmed that the results were in fairly good agreement with those of the CMA.

(3) Identification of α and γ phases by Cr/Ni partition ratio and color presentation were also possible using the present system.

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