

Methods for Detecting Phosphorus Segregates in Steel*

Yoshiko Funahashi**
Hidenari Kitaoka***

Yasuharu Matsumura**
Sen-ichi Harimaya****

1 Introduction

In response to recent demand for more diverse and sophisticated steel products, remarkable progress has been made in techniques to reduce centerline segregation. The conventional sulfur print test is largely ineffective in detecting solidification segregates in advanced products, while on the other hand, stricter customer requirements mean attention must be given to the segregation of carbon and phosphorus in addition to that of sulfur. To meet these requirements and improve the quality control system, a series of new methods collectively called the P-print test method was developed for detecting solidification segregates of phosphorus. This paper presents an outline of the P-print method and discusses its characteristics and applications.

2 Outline of P-Print Test Method

2.1 Principles and Procedures

The P-print test procedure and typical print images are shown in **Fig. 1** and **Photo 1** respectively. A wet test sheet bearing an etching solution is first applied to the test surface, and phosphorus (or iron) eluted from the surface and absorbed onto the sheet is then developed for color. Phosphorus segregates appear as silver images (Silver-method), red images (Red-method), or blue images (Blue-method), depending on the etching and/or color-developing reagents used.

The Blue-method can also be applied to stainless steels if electrolysis is used to etch the test surface. In stainless steels, as chemical etching cannot proceed because of the formation of passive films on the test surface, the test surface is etched by electrolysis with a

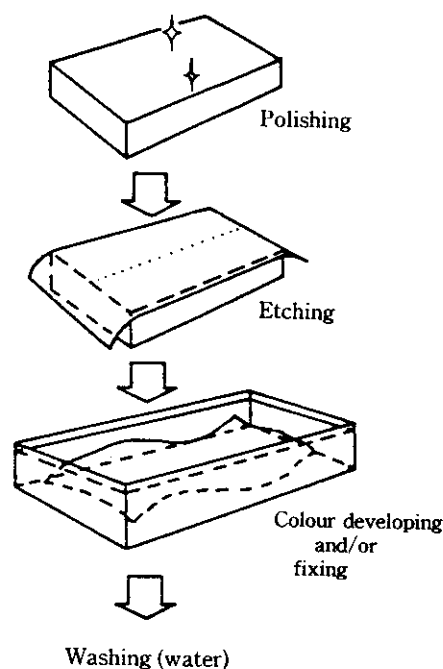


Fig. 1 Procedure of P print

special etching apparatus (**Photo 2**). Eluted phosphorus is then developed as a blue image.

2.2 Characteristics

Table 1 shows the characteristics of the three P-print methods. With roughly polished surfaces, broad surface areas can easily be evaluated. Processing with these methods takes from 10 to 35 minutes. The Blue-method is superior in detectability and makes possible the detection of solidification structures, white bands, and internal cracks in addition to center-line segregation.

As electrolysis etching is used with stainless steels, the print test process can be repeated as many times as desired without repolishing the test surface. The method can also be applied to low phosphorus steels, because the amount of etching of the test surface can be increased by controlling the etching time and current density.

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** Senior Researcher, Instrumentation & Analytical Science Research Center

*** Senior Researcher, Steelmaking Lab., Iron & Steel Res. Labs.

**** General Manager, Chemical Analysis and Physical Testing Center, Kawasaki Steel Techno-research Corp.

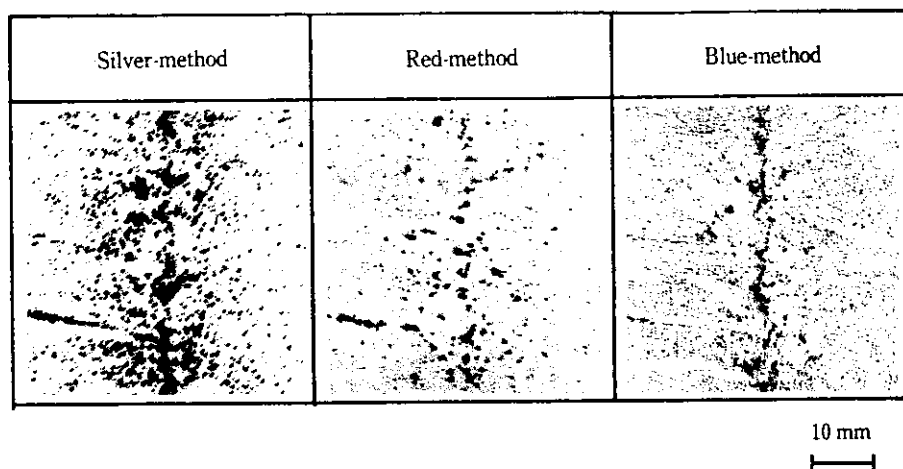
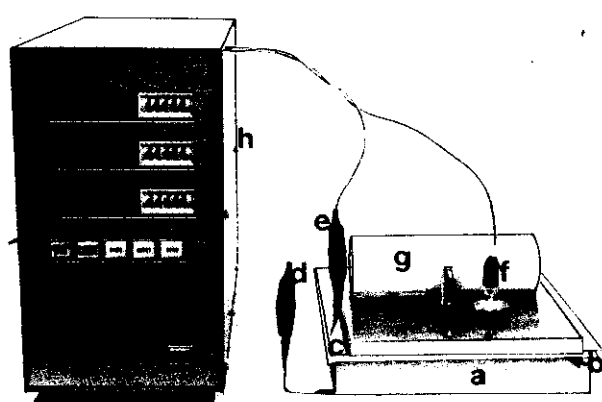


Photo 1 Examples of P print results of specimen cast by segregation simulator (midsection of CC slab, P = 0.02%)



a: Steel sample e: Cathode terminal
b: Test paper f: Reference electrode
c: Cathode plate g: Pressure weight
d: Anode terminal h: Etching apparatus

Photo 2 Etching Apparatus for P print

Table 1 Characteristics of three P print methods

	Silver-method	Red-method	Blue-method
Applicable steel	Carbon	Carbon	Carbon Stainless
Detectability ^{a)}			
Macro segregation	○	○	○
Semi-macro segregation	△	×	○
Solidification structure	×	×	○
White band	×	×	○
Internal crack	△	×	○
Resolution ^{b)}	++	+	+++
Operation time (min)	20	10	35

^{a)} ○ detectable, △ faintly detectable, × undetectable

^{b)} + low, ++ intermediate, +++ high

3 Applications

The P-print method was applied to the evaluation of measures to reduce center-line segregation. Test results of a steel specimen cast by a segregation simulator are described below.

3.1 Dispersion of Phosphorus Segregates in Electromagnetic Stirring

Photo 3 shows P-print images of austenitic stainless steel specimens cast with and without electromagnetic stirring. Without electromagnetic stirring, growth of dendritic structures proceeded to the internal area of the specimen, and concentrated phosphorus segregates were observed in the center area. In contrast, phospho-

rus segregates were finely dispersed along a V-shaped segregation line when electromagnetic stirring was used.

3.2 Improvement of Phosphorus Segregation and Internal Cracks with Heat Treatment

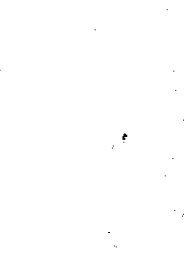
Three specimens were cut from the center of a CC slab. Two were heated (1050°C, 1250°C) for 8 hours and air cooled, while the other was not given heat treatment. P-print tests were carried out for each specimen (Photo 4). Phosphorus segregates in the center area were virtually eliminated with heating, demonstrating the effectiveness of heat treatment in the dispersion of phosphorus.

P-print images of a cross section of a martensite stainless steel slab are shown in Photo 5. The internal cracks which were observed in this specimen disappeared after heat treatment at 750°C for 10 h.

(a) without EMS



(b) with EMS

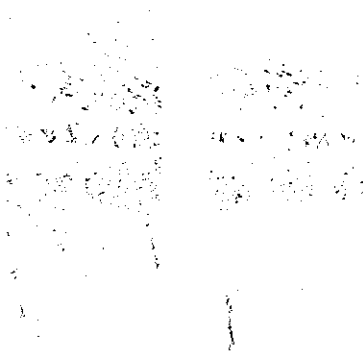


10 mm

Photo 3 Prints of centerline segregation in a stainless steel slab cast with or without electromagnetic stirring (specimen cast by segregation simulator, midsection of CC slab, $P = 0.025\%$)

(a) As cast

(b) $1050^{\circ}\text{C} \times 8 \text{ h, AC}$ (c) $1250^{\circ}\text{C} \times 8 \text{ h, AC}$



10 mm

Photo 4 Prints of centerline segregation in a carbon steel before and after heat treatment (specimen cast by segregation simulator, midsection of CC slab, $P = 0.02\%$)

(a) As Cast

(b) $750^{\circ}\text{C} \times 10 \text{ h, AC}$



10 mm

Photo 5 Prints of internal cracks in a stainless steel before and after heat treatment (specimen cast by segregation simulator, midsection of CC slab, $P = 0.02\%$)

4 Concluding Remarks

Phosphorus segregation in steel products is closely related to their chemical composition and thermal history. The P-print test method discussed here can be applied to advanced steels which cannot be evaluated by the conventional sulfur print method. Because the Blue-method can detect the segregation of phosphorus itself,

effective information can be obtained for the investigation of the relationship between the internal structure and the distribution and magnitude of phosphorus segregates.¹⁾

Reference

- 1) Y. Funahashi: *Nihon Kinzoku Gakkai-ho (Bulletin of the Japan Institute of Metals)*, **26**(1987)6, 517.