# NEW GRAIN—DETECTION METHODS FOR GRAIN—ORIENTED Si-Fe COATING

by

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## Summary

New grain-detection methods for Si-Fe are described namely (1) a ferromagnetic colloid technique, and (2) by surface flux measurements. These methods are fast, safe, pollution-free and easy to automate, and give 100% quality-control accuracy, because the coating does not have to be removed.

## Introduction

Grain detection is one of the most important quality control parameters in the production of low-loss and low-noise Si-Fe sheets with improved grain orientation, optimal grain size, and a tensile coating. Conventional grain detection is exclusively carried out by removing the coating using powerful chemicals for long periods. This destructive test results in an accuracy of less than 100% in quality control. In this paper, new grain detection methods which do not require the coating to be removed are described, namely (1) a ferromagnetic colloid technique, and (2) by surface flux measurements. These methods are fast, safe, pollution-free, and easy to automate, and give 100% quality control accuracy because the coating does not have to be removed. These non-destructive techniques also make it possible to measure the relation between local magnetic properties and the grain structures of Si-Fe sheets with the tensile coating still in position.

## Ferromagnetic Colloid Technique

Since various defects are concentrated at grain boundaries, the grain contour can be observed on the coating by using a ferromagnetic colloid technique. In our experiments, specimens were magnetized with D.C. field of approximately 100 oe and then a magnetite colloid was directly applied to the coating in order to obtain clear grain patterns.

Fig. 1 illustrates magnetizing methods using (a) coils, (b) U-shape yoke, and (c) single sheet

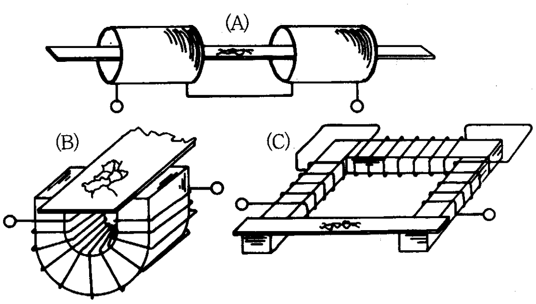


**Fig. 1 Magnetizating methods for ferromagnetic colloid technique**

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tester yoke. The U-shape yoke was used in the experiments because it enabled the sheets to be magnetized in various directions.

Fig. 2 shows an experimental ferromagnetic colloid pattern for grains on the coating of a 3% Si-Fe (HI-B) Epstein sample. A D.C. field of approximately 100 oe is applied in the direction perpendicular to the rolling direction, Fig. 2 (a). Magnetite particles are collected by the effect of a large magnetic field gradient at the grain boundaries, and hence a clear contour of grains is obtained.

The clarity of the colloid patterns of the grain depends on the direction and magnitude of the applied field ; because if the sheet is magnetized with a field that is too large ferromagnetic particles are collected not only at the grain boundaries but also at small surface defects which then grow into fine striations perpendicular to the magnetization vector and hence obscure the grain patterns. This behaviour is shown in Fig. 2 (b), where a D.C. field of 100 oe was applied in the rolling direction. Grain patterns are clearer on the sheet when magnetized in the direction perpendicular to the rolling direction than when magnetized parallel to the rolling direction assuming that a D.C. field of correct value is used : that is less that 100 oe for the perpendicular direction and less than seventy oe for the parallel direction. It is considered that this is due to the fact that the magnetization in each grain does not align perpendicularly to the rolling direction, Fig. 2 (a).

Fig. 3 shows experimental colloid patterns for a 3% Si-Fe sheet with small grains. It is easier to observe the grain shapes by using colloid technique than the conventional coating removal method, because the colloid patterns are chamically stable and produce line patterns. It is considered the colloid technique is suitable for direct observation of grain patterns, but is not easy to automatically measure the statistical values of grain sizes in a lamination.

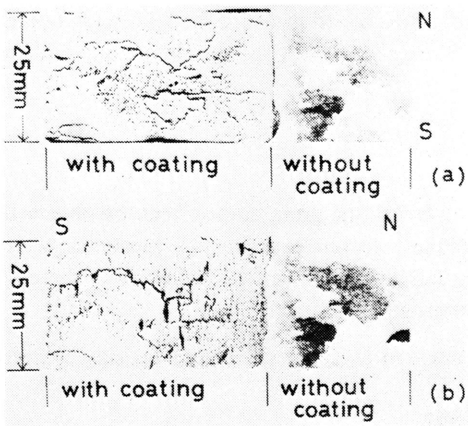



**FIg. 2 Experimental colloid patterns of grains on the coating of a 3% Si-Fe (HI-B) sample magnetized in the direction**
**(a) perpendicular to the rolling direction**
**(b) parallel to the rolling direction**

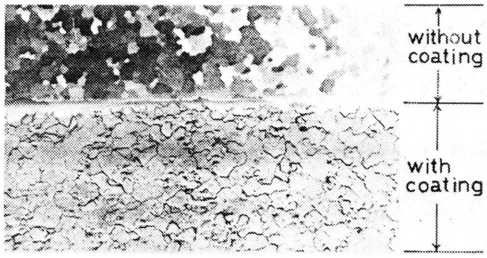



**Fig. 3 Experimental colloid patterns of a conventional 3% Si-Fe**

## Surface Flux Measurements

The grain boundaries can also be detected on the coating by measuring the normal surface flux on a sheet magnetized with a D.C. field since various defects are concentrated at the grain boundaries and hence a leakage flux is generated there.[1].

Filg. 4 illustrates a schematic representation of a system for measuring normal surface flux. A small Hall element is swept across the coating using a micromotor drive. The Hall probe is connected via a gaussmeter to an oscilloscope, and threrfore the variation of normal surface flux $\phi^*$, with surface position $x$, can be displayed since the Hall element is moved across the coating at a constant speed.

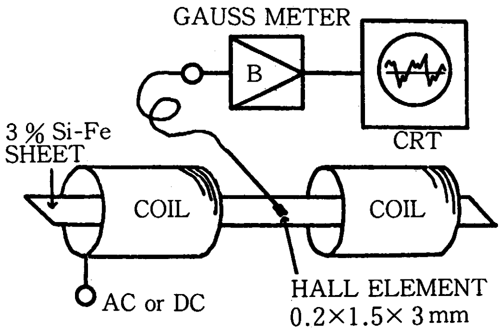



**FIg. 4 Circuit diagram for surface flux measurements**

Fig. 5 shows a model for the relation between the $\phi^*$ against $x$ characteristics and the grain boundary positions at which magnetic poles appear when a D.C. field is applied. Therefore, grain boundaries can be detected at the position of the sharp peaks, illustrated by black circles, on the $\phi^*$-$x$ characteristic.

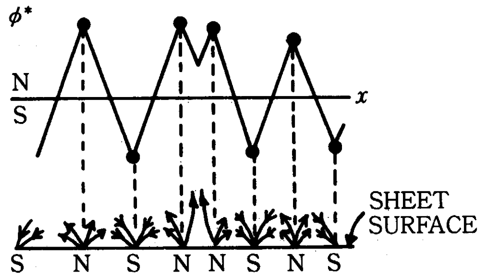
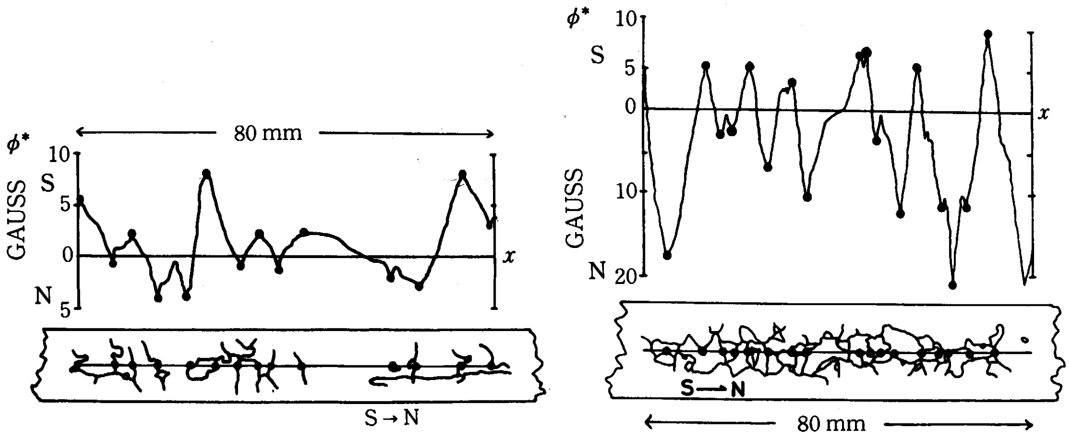



**Fig. 5 A model for the relation of nomal surface flux versus grain boundary positions**

Fig. 6 shows experimental $\phi^*$-$x$ characteristics for (a) 3% Si-Fe (HI-B) Epstein sample, and (b) a conventional 3% Si-Fe Epstein sample. Both of the sheets were magnetized with a D.C. field of 90 oe in the rolling direction. In (a), the predicted grain-boundary positions exactly coincide with the positions observed after removing the coating, whilst the grain detection accuracy was about 80% in (b). The average grain size can also be predicted by counting the number of zero-crossing points in the $\phi^*$-$x$ curves instead of counting the sharp peak points, although the zero-crossing point does not coincide with the grain boundary. For automatically measuring the average grain size, the zero-crossing point method, which produced 10-15% higher value of grain size than the peak point method is preferred since the construction of the electronic measuring system is easier than for the sharp peak point method. In surface flux measurements, it is difficult to obtain useful $\phi^*$-$x$ curves near the edge of the sheet due to the existence of a large demagnetizing field, Hd. A technique to minimize the effect of Hd, is shown in FIg. 7, by which the useful $\phi^*$-$x$ curve can be measured up to about I cm from the edge of the sheet, to which another Si-Fe sheet is butted.




(a) (b)

**Fig. 6 Experimental results of $\phi^*$-x curves for (a) HI-B, (b) conventional 3% Si-Fe**

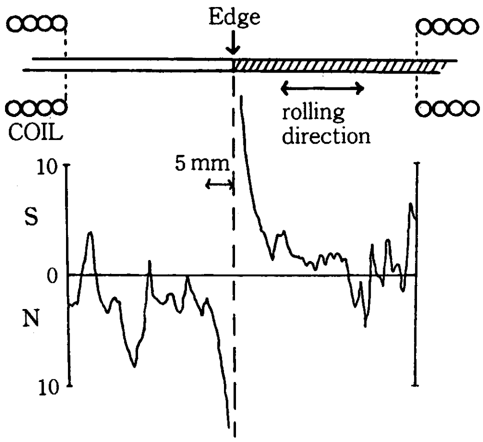



**Fig. 7 A technique for measurements near the end of a sheet**

## Additional Discussions

Fig. 8 illustrates the relation between $\phi^*$ and applied stressed in a 3% Si-Fe (HI-B ; 0.3 mm thick, 10 mm wide, and 500 mm long) sample magnetized with a D.C. field of 90oe : $\phi^*$ increases with increasing tension (a), whilst $\phi^*$ decreases with increasing compression (b). From these tesults, it is considered that small holes at the grain boundaries are expanded by tension, and are contracted by compression. When the sheet is bent, (c) $\phi^*$ decreased therefore the compressive effect is considered to be dominant at the grain boundaries during bending.

When the sheet was magnetically annealed, $\phi^*$ decreased as shown in Fig. 9. The result is considered to be due to the internal strain which was removed by magnetic annealing.

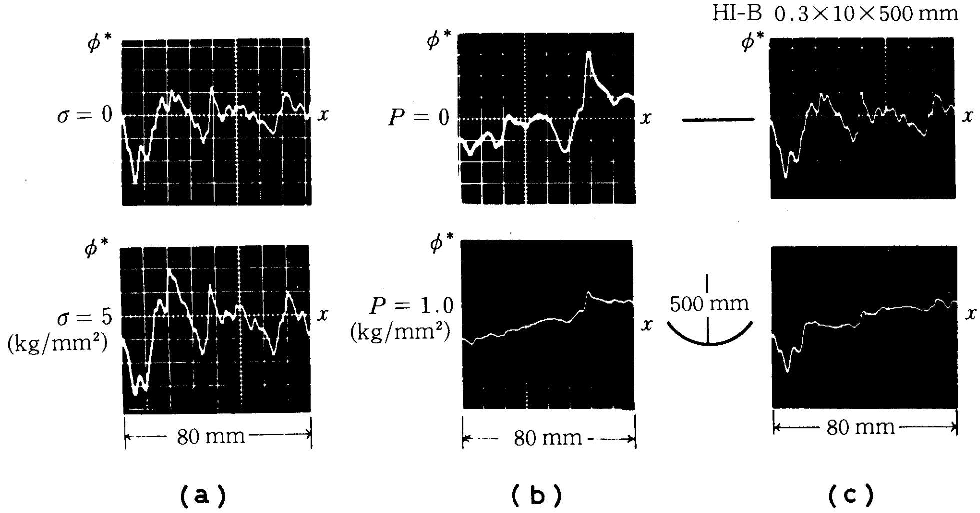



**Fig. 8 Variation of $\phi^*$ with stresses : for (a) tension, (b) compression and (c) bending**

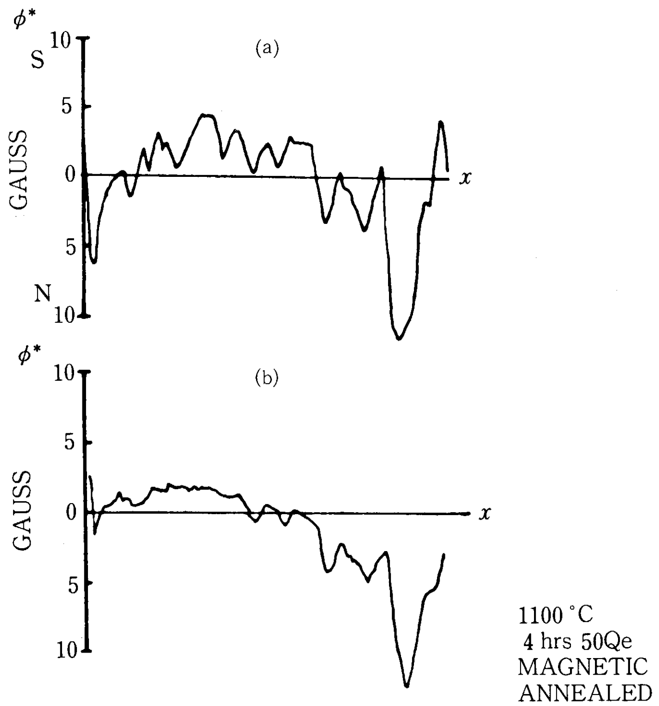



**Fig. 9 Effect of mangetic annealing on $\phi^*$**

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## References

1. K.Mohri and T. Fujimoto, 8th Ann. Conf. on Magnetics, 20a B-4 1976, (Japan).