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ON-LINE PHASE AND ALLOYING DEGREE MEASUREMENT SYSTEM FOR GALVANNEALED COATINGS

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Abstract − An on-line measurement system for phase fraction and alloying degree of galvannealed steel has been developed using an X-ray diffraction method. Intensities of diffraction peaks of Γ, δ, ζ and η iron-zinc phases were utilized for the determination of each phase fraction and alloying degree. Calibration curves were constructed using laboratory prepared standard reference samples and multiple regression technique. The system has been employed in POSCO's galvannealing line at Kwang Yang Works for process monitoring.

Keywords: galvannealed steel, alloying degree, iron-zinc inter-metallic phase

1. INTRODUCTION

Galvannealed coated steel is commercially produced by post annealing of hot dip galvanized (zinc-coated) steel. During the process, several iron-zinc inter-metallic phases are formed by inter-diffusion of zinc and iron from steel. The best properties can be obtained when the galvannealed coatings have only $\zeta(FeZn_{13})$ and $\delta(FeZn_7)$ phases. But, if the alloying is insufficient, η phase present in the coatings results in diminished paintability. On the other hand, excessive alloying makes $\Gamma(Fe_5Zn_{21})$ phase present between the substrate and coating interface and the coatings easily can be separated during press forming or bending. Therefore, the alloying degree (iron content in coating) and each phase fraction must be monitored and controlled in order to optimise the process parameters.

It is difficult to analyse galvannealed coatings by conventional x-ray fluorescence methods since the iron is contained in both the coating and steel. Some researchers propose a method of X-ray fluorescence using two take-off angles [1]. On-line measurement systems have been developed using X-ray diffraction [2] or combined with X-ray fluorescence technique [3], but none of above methods can determine each phase fraction and alloying degree simultaneously.

In this paper, we report the results of the development of on-line measurement system for the determination of alloying degree as well as the amount of each phase fraction for galvannealed steel whose operational principle is based on the X-ray diffraction method.

2. INSTRUMENTATION

The schematic diagram of the on-line phase and alloying degree measurement system is shown in Fig. 1. The system is composed of a measuring head, traversing stand, electronic controller and computer. X-rays are generated and detected at the measuring head, which moves across the galvannealed steel. The electronics controller consists of the X-ray and detector power supplies, pulse height analyzer, counter, I/O and CPU boards. The computer receives the measured data by TCP/IP from the main controller and displays the measured data for monitoring.

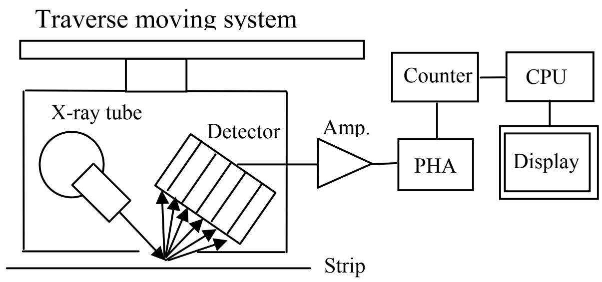

Fig. 1. Schematic diagram of the on-line alloying degree and phase measurement system.

The measuring head consists of one X-ray tube and six detectors. Intensities of diffraction peaks for ζ, δ, η and Γ phases are measured simultaneously by scintillation detectors. Two of which measure background intensities. The measured intensities were converted to alloying degree. A Cr target broad beam-type X-ray tube was employed for minimum X-ray fluorescence from steel. A V filter was employed with focusing slit to eliminate diffracted peaks from Cr Kβ radiation. Since the temperature of the galvanneled strip at the measuring position is sometimes above 150^{o}C, water cooling for the measuring head is necessary. Strip vibration has been minimized by using two guide rolls at the front and back sides of the measuring head.

Photo 1 shows the developed system installed in the galvannealed line at KwangYang Works in Korea.

Photo 1. The on-line phase and alloying degree measurement system installed in galvannealing line.

3. RESULTS AND DISCUSSIONS

3.1. Determination of alloying degree

Standard reference samples were prepared by annealing the galvanized steel in a salt bath. The annealing temperature was at 480°C for preparation of low alloying samples, but the temperature was raised to 520°C for high alloying samples. Six samples were selected for the construction of a calibration curve for the developed system. Alloying degrees were determined from the intensities of four diffracted peaks and the coefficients were regression analyzed using six standard reference samples.

Fig. 4 shows the alloying degree measured by ICP (inductively coupled plasma spectrometry) and the developed system. A good correlation between the two methods was found for a broad range of alloying degrees from 0.5 to 23wt.%Fe. The analytical accuracy σ(= $\sqrt{\sum d^2/(n-1)}$, d= the difference between the two methods, n= the number of samples) of alloying degree was 0.75%.

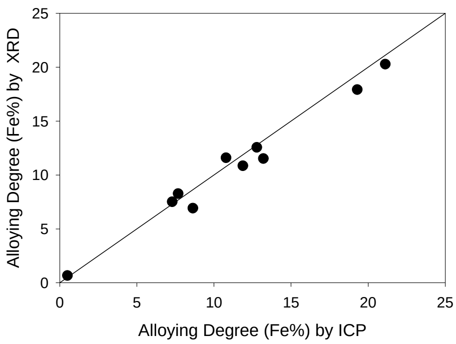

Fig. 2. Comparison of alloying degree measurements by ICP and those obtained by the developed system

3.2. Determination of phase fraction

Diffraction intensities of each phase for selected standard reference samples were measured. By assuming that the intensity increases linearly with increasing the amount of the corresponding phase, the phase fraction of each standard reference sample was determined. Using the coefficient determined from the regression method, each phase fraction of galvannealed steel was determined. The sum of the four phases was made to equal 100%.

Fig. 3 shows the on-line measurement results for the galvannealed production line. Different colours represent different phases. It shows that the alloying degree has a close relationship with the amount of Γ and δ phase fraction.

Fig. 3. On line measurement results of phase fraction (upper part) (red color: Γ, Blue: δ, green: ζ, yellow: η) and alloying degree (lower part).

4. CONCLUSIONS

The on-line measurement system of phase and alloying degree of galvannealed steel has been developed using an X-ray diffraction method. Intensities of diffraction peaks of Γ, δ, ζ, and η phases were utilized for the determination of each phase fraction and alloying degree, simultaneously. Calibration curves were constructed using laboratory prepared standard reference samples and multiple regression techniques. The developed system achieved an analytical accuracy for alloying degree of 0.75%. The system has been installed in galvannealing production line and operated for about one and half years without any trouble.

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