# AN X-RAY STUDY OF THERMODIFFUSION PROCESSES ON TYRE WIRE COATING①

Wang Yandong, Zhang Fang, Liu Yandong, Xu Jiazhen

*Department of Materials Science and Engineering, Northeastern University, Shenyang 110006*

**ABSTRACT** The thermodiffusion behaviors of brass coating on tyre wire obtained by electro-deposition of copper and zinc with controlled thickness, followed by continuous annealing in industry and isothermal annealing at laboratory, respectively, were studied by means of X-ray diffraction analysis. With the information of X-ray diffraction, the fraction of $\beta$ phase during different thermodiffusion processes was given and the phase homogeneity of $\alpha$ phase in the films was discussed.

**Key words** tyre wire electro-deposition diffusion X-ray diffraction

## 1 INTRODUCTION

In the recent years, there has been of great interest in the study of manufacturing processes of tyre wire for the application of tyre industry. As the reinforcement of pneumatic tyre, tyre wire is a kind of high strength and toughness steel cord with brass coating, and brass coating is one of the significant step during process of the steelcord. The newest way of coating on tyre wire is electro-deposition diffusion processes, which not only overcome environment pollution caused by the old ways of using cyanide, but also raised efficiency and quality. However, the unstability of coating processes has been one of the main problems which lead to the poor physical and chemical properties of tyre wire. It is shown by Starinshak[1] that the adhesion between the tyre wire and the rubber depends on the chemical composition of the brass layer. In fact, the relative fractions of the $\alpha$ and $\beta$ phase have also important influence on the wet drawing processes of tyre wire. Compared to the $\alpha$-brass with *fcc* structure, the $\beta$-brass with *bcc* structure has less easy slip system. Thus, the presence of $\beta$-brass leads to higher shearing stress making the deformation during drawing more difficult, increasing the loss of brass.

X-ray diffraction analysis can not only decide the phase contents of materials[2], but also detect phase composition homogeneity quickly and exactly in thin films. The investigations into the thermo-diffusion processes of tyre wire by means of X-ray diffraction have been reported in Ref[3]. To improve the quality of tyre wire for automotive industry in China, the thermodiffusion behaviors of the brass coating on steel cord obtained by electro-deposition of copper and zinc with controlled thickness, followed by electro-thermal annealing in industry and isothermal annealing at laboratory, respectively, were studied in present paper. The effect of heat-treatment on the fraction of $\beta$ phase and the composition homogeneity of $\alpha$ phase during the thermodiffusion after electro-deposition are discussed in detail.

## 2 EXPERIMENTAL PROCEDURES

The specimens of 1.68 mm (Specimen A) and 1.38 mm (Specimen B) in diameter, respectively, with successive layers of copper and zinc were provided by Shenyang Steel

① Financed by the State Educational Commission, Liaoning Province Science & Technology Commission; Received May 30, 1995, accepted Sep. 25, 1995

Cord Factory. For X-ray diffraction analysis, four 30 mm-long sections, schematically shown in Fig. 1, were cut from each specimen which had been annealed through continuous annealing in industry and isothermal annealing (as shown in Table 1) at laboratory, respectively.

**Table 1 Heat-treatment condition of specimen A and B in the Laboratory**

| Thermodiffusion temperature/℃ | Diffusion time/min. | Cooling condition* |
|---|---|---|
| 400 | 3, 8, 15, 22 | A.C. |
| 450 | 1, 4, 7, 10 | W.C. A.C. F.C. |
| 500 | 1, 2, 3, 5 | A.C. |

* A.C.—air cooled; W.C.—water cooled; F.C.—furnace cooled

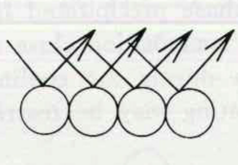

**Fig. 1 The schematic diagram to show the X-ray diffraction geometry for cord steel**

The experiments were performed in a Rigaku Dmax-3A diffractometer with Cu-Ka radiation and Ni filter. The tube voltage and current were 37.5 kV and 25 mA, respectively. The scanning step width and present time are 0.04° and 4 s, respectively. The volume fractions of $\alpha$ and $\beta$ phase were determined through comparing the $\{111\}_\alpha$ with the $\{110\}_\beta$ diffraction peak. The overlapping diffraction peak was separated according to Gauss Function[4] and the height of each peak was used as intensity for quantitative analysis.

## 3 RESULTS AND DISCUSSION

### 3.1 *Volume Fraction of β Phase for Industry Annealing Specimens*

Fig. 2(a), (b) show that the $\{110\}_\beta$ and $\{111\}_\alpha$ diffraction peaks of specimen *A* and *B* with electrothermal annealing (in industry). The $\{110\}_\beta$ diffraction peak can be observed obviously in both specimens. The volume fractions of $\beta$ phase in specimens *A* and *B* are 7.28% and 13.5%, respectively. Comparing with specimens with isothermal annealing at laboratory (Section 3.2) and previous results[3], the fraction of $\beta$ phase is greater, which indicates that the industry annealing parameters should be adjusted.

Fig. 2 (c) gives the $\{111\}_\alpha$ and $\{110\}_\beta$ diffraction peaks of specimen *B* separated a thin layer of about 1 mm thick on the surface.

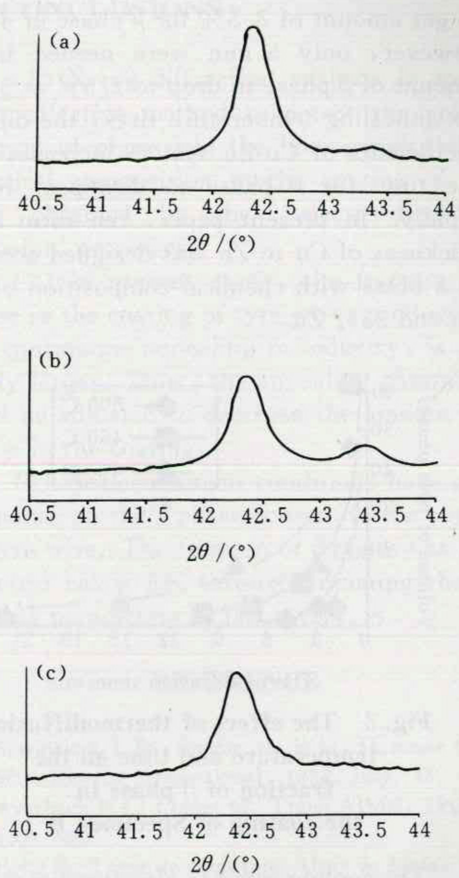


**Fig. 2 The X-ray diffraction profiles of tyre wire with brass coating**

(a)—Specimen A thermodiffused in industry;
(b)—Specimen B thermodiffused in industry;
(c)—Specimen B thermodiffused in industry and separated a thin layer

Comparing with previous sample, the intensity of $\{110\}_\beta$ peak decreases. X-ray quantitative analysis shows that the fraction of $\beta$ phase dropped from 13.5%~8.57%, which indicates that the relative much $\beta$ brass in the coating may be lost in the wet drawing processes.

## 3.2 *Effect of Heat-treatment on the Fraction of β Phase in the Coating*

Fig. 3 shows the volume fraction of $\beta$ phase as a function of the time of thermodiffusion at 400 ℃, 450 ℃ and 500 ℃, respectively. As shown in Fig. 3, it takes about 22 min to get amount of 3.5% for $\beta$ phase at 400 ℃, however, only 5 min were needed for the amount of $\beta$ phase to drop to 2.5% at 500 ℃. As annealing temperature rises, the diffusion coefficients of Cu-Zn system increase, thus, the time for $\beta$ phase to disappear reduces rapidly. In present paper, the ratio of the thickness of Cu to Zn was designed according to a brass with chemical composition of 67% Cu and 33% Zn.

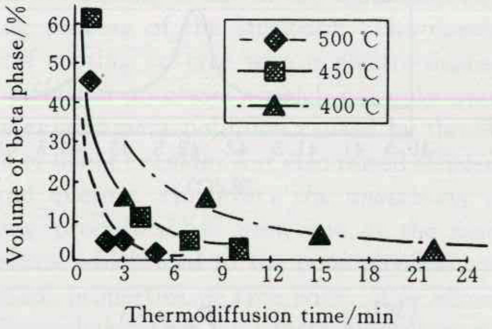


**Fig. 3 The effect of thermodiffusion temperature and time on the fraction of β phase in the coating of Specimen B**

The cooling rate has also significant influence on the fraction of $\beta$ phase. Fig. 4 gives the volume fraction of $\beta$ phase of both specimens heated in the annealing furnace at laboratory at 450 ℃ for 10 min and cooled in various conditions. The smaller amount of $\beta$ phase are detected in the samples with furnace cooling and water cooling, however, it is seen that the larger amount of $\beta$ phase are evident in those with air cooling. The above results indicate that cooling rates is another significant factor to influence the fraction of $\beta$ phase in the coating. As shown in the Cu-Zn phase diagram[5], phase content is controlled by two important processes, both of which are controlled by the thermodiffusion processes. One is phase transformation of $\beta \rightarrow \alpha$ at high temperature, the other is the precipitation of $\beta$ phase from $\alpha$ phase during cooling process. The furnace cooled specimens hold for a long time at high temperature will transform completely from $\beta$ to $\alpha$ and $\alpha$ phase has enough time to become homogeneous, thus, the smaller amount of $\beta$ phase precipitation occurs during the following cooling. However, as the air or water cooled samples are held for short time at high temperature, the inhomogeneous $\alpha$ phase will be produced in the coating, thus, larger amount of $\beta$ phase precipitated from $\alpha$ phase with chemical composition close to the solution limitation during air cooling, while $\beta$ phase precipitating may be restrained in the water cooling.

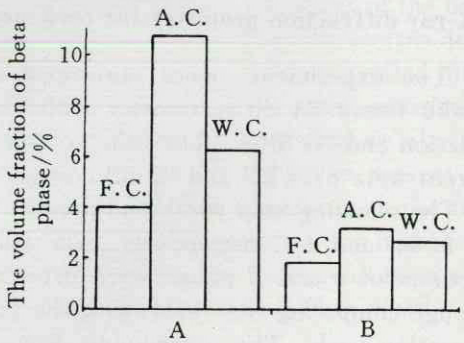


**Fig. 4 The effect of various cooling rates on the volume fraction of β phase**

## 3.3 *Study of Homogeneity of α Phase in Coating*

The chemical composition of $\alpha$ phase obtained by the processes of electro-deposition diffusion may be inhomogenous. The homogeneity of $\alpha$ phase may be analyzed through the shape and position of diffraction peak of $\{200\}_\alpha$ of $\alpha$ phase. Fig. 5 gives the diffraction peak of $\{200\}_\alpha$ of specimen B thermodiffused at 450 ℃ for various time. As shown in Fig. 5, the broader diffraction peak is observed for the sample thermodiffused for 1 min at 450 ℃,

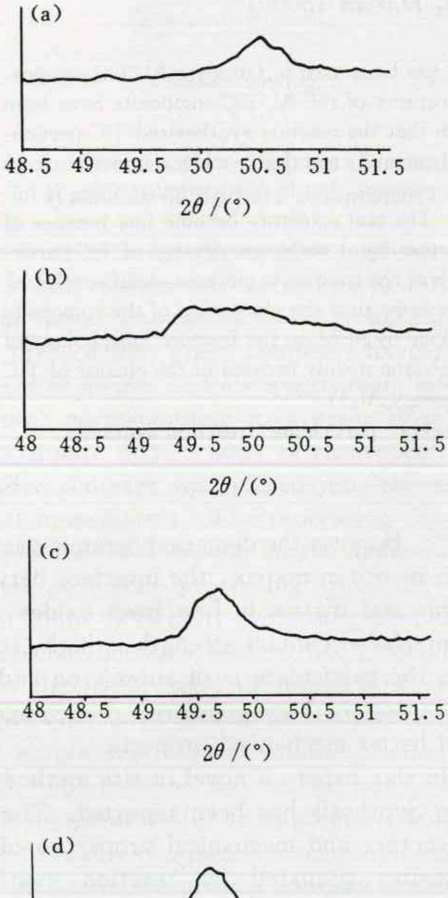


**Fig. 5 Diffraction peak of $\{200\}_\alpha$ of Specimen B obtained by**

(a)—electro-deposition of Cu and Zn with controlled thickness and following by annealing for 1 min (b), 4 min (c) and 10 min (d) respectirely, at 450 ℃

which indicates that the chemical composition of $\alpha$ phase is inhomogeneous and its zinc concentration in copper layer varies from 0%~38% ( Zn solution limitation in $\alpha$ phase). With increase of diffusion time, zinc concentration of $\alpha$ phase increases and the lattice constant of $\alpha$ phase increases, which leads to the movement of X-ray diffraction peak of $2\theta$ from higher to lower angle, while the width of diffraction peak becomes narrow which means that the homogeneity of $\alpha$ phase increases. Above discussion shows that the homogeneity of $\alpha$ phase can be determined precisely by X-ray diffraction analysis.

## 4 CONCLUSIONS

(1) X-ray diffraction analysis is one of most effective method to detect the volume fraction of phase and the homogeneity of its chemical composition in the coating of tyre wire obtained by the 'electro-deposition diffusion' processes.

(2) In present study, the fraction of $\beta$ phase in the coating of tyre wire, produced by the continuous annealing in industry, is relatively larger. Thus, the annealing parameters need be adjusted to decrease the amount of $\beta$ phase in the coating.

(3) Heat-treatment conditions have great influence on the $\beta$ phase content in the coating of tyre wire. The fraction of $\beta$ phase has been dropped below 5% through adjusting the annealing parameters at laboratory.

## REFERENCE

1 Starinshak T W, Shemenski R M, Hammer G E. Wire Journal International, 1988, July: 45.
2 Averbach B L, Cohen M. Trans AIME, 1943, 154: 306.
3 Bolle B. These de doctorate. Univ de Metz, 1994.
4 Wang Yandong *et al*. Transaction of Nonferrous Metals Society of China, 1992, 2: 47.
5 Weat E G. Copper and Its alloys. Ellis Horwood Limited, 1982, 57.

**(Edited by Zhu Zhongguo)**