China Steel Technical Report, No. 32, pp.30－38, (2019)

Effects of Furnace Atmosphere on the Post-Annealing Adhesion Capability of Insulation Coating to Electrical Steel Substrates

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Electrical steels (ES) are the core of electrical motors, power generators, etc. To restore the electromagnetic properties of ES after stamping, stress-relieving annealing is often adopted. However, annealing can be very destructive to the insulation coating on ES. Inappropriate annealing usually results in coating adhesion failure, which greatly affects the performance of ES facilities. In this paper, effects of furnace atmosphere on the post-annealing adhesion capability of ES coating were investigated to reveal the individual influence of O_2, CO_2, H_2, and H_2O, respectively. The presence of CO_2 has little effect while both O_2 and H_2O can deteriorate the post-annealing adhesion capability. Contrarily, H_2 has protecting effects so that good post-annealing adhesion capability of coating can be fulfilled. Therefore, to ensure good post-annealing adhesion capability to the coating, low contents of oxygen & humidity (dew point) in the furnace atmosphere is required and the presence of some hydrogen in the furnace atmosphere can further help.

Keywords: Electrical steel, Annealing, Insulation coating, Adhesion, Atmosphere

1. INTRODUCTION

Electrical steels (ES), also known as silicon steels, are an important family of steels that consist of Fe-Si alloys. Such alloys have special electromagnetic properties and are the core of ES facilities (e.g., motors, power generators, compressors, and transformers). Therefore the properties of electrical steel are very critical to the efficiency of energy transformation.

The loss during the energy transformation (i.e., the "iron loss") is composed of the hysteresis loss and the eddy current loss.[1] The former is an intrinsic property and is mainly controlled by the alloying composition and process history of electrical steels. On the other hand, the latter depends more on extrinsic parameters, e.g., the design of the energy-transforming systems and the geometry of the ES core. Eddy current loss is proportional to the square of the thickness of the conductor; therefore electrical steels are arranged as a stack of thin sheets. Besides, Eddy current loss is also inversely proportional to the interlayer resistivity between ES sheets. Therefore most modern ES coils are coated with an insulating layer.[2,3]

Since ES are used in stacks of predesigned stamped shapes, ES coils must be slit in to hoops of the required width first. Then the hoop runs through a high-speed punch process to form shaped stampings. However, the punch process unavoidably leaves residual stress on the thus formed stampings. Therefore, electromagnetic properties (e.g., the iron loss) of ES always deteriorate after the punching process. To eliminate the residual stress and to restore the electromagnetic properties of the ES stampings, thermal treatment (typically 700~850°C) is usually carried out after the punch process. And such kinds of thermal treatment are termed as "stress-relieving annealing (SRA)".[4,5]

Practically, lubricant oil must be applied in the punch process of ES. Therefore, the punched ES stampings are unavoidably covered by oil. Such residual oil can be problematic and needs to be removed before SRA. For that, a "burn-off" process by heating ES stampings *in air* is usually carried out before SRA. As compared with the annealing process, the required burn-off temperature is significantly lower (typically 250°C ~500°C).

Though thermal treatments as described above can recover and even improve the electromagnetic properties of ES stampings after the punch process, it can also be very destructive to the insulating coating on ES. Inappropriate thermal treatments often lead to failures in coating adhesion and interlayer resistance (electrical insulation between stampings). Ultimately, such failures

would result in serious problems such as increased iron loss, lowered efficiency, and abnormal temperature rise for ES facilities. In other aspects, parameters (e.g., the furnace atmosphere, the heating profile, etc.) and equipment for thermal treatment are so diversified that the result of annealing can vary considerably, especially in the coating adhesion capability and the appearance of annealed stampings. Hence, the SRA process is undoubtedly the most difficult to control among all the working processes of ES. And in many cases, failures were resulted from variations or deviations of the above thermal treatment parameters.[6~8]

Therefore, effects of furnace atmosphere (O_2, H_2O, H_2, and CO_2) on the post-annealing adhesion capability of ES coating were investigated in this study. Results from this study can be used as guidelines to optimize the SRA process of ES.

2. EXPERIMENTAL METHOD

2.1 The materials

Two grades of electrical steel were used as the substrate. The major alloying elements (Si and Al) for these substrates are shown in Table 1. For the coated samples, substrates were coated with a waterborne chromium-free (phosphate-based) paint and then baked by heating, after which the coating can be cured. And thus the obtained ES samples were coded as SA and SB, respectively.

2.2 Anti-corrosion capability

The anti-corrosion capability of the investigated coated ES (before thermal treatment) was evaluated on edge-sealed samples, following the ASTM-B117 method (the neutral salt spray test, SST). The salt spray was produced from 5% NaCl aqueous solution at a temperature of 35°C with a rate of 1~2 mL/hr. The anti-corrosion capability of the samples was then judged by the rusting area percentage after 16hrs SST.

2.3 Thermal treatment (Burn-off & Stress-Relieving Annealing)

Thermal treatments were performed in a single-chamber furnace. Since this furnace is *not airtight*, nitrogen at a high flow rate (>130L/min) was required to apparently repel the air out of the chamber (but traces of oxygen and humidity could still remain in the chamber). If the nitrogen flow rate was not high enough, air could significantly leak into the furnace resulting in surface oxidation being very recognizable on the annealed samples.

A 7-stage heating profile was adopted for the thermal treatments (Figure 1). Stages 1 and 2 consist of the "burn-off period" and stages 3~7 function as the "stress-relieving annealing (SRA) period". The maximal temperature for burn-off and annealing are 275°C and 780°C, respectively. Table 2 shows the furnace atmosphere for the above thermal treatment process. Six different combinations of atmospheres (I ~ VI) were tested. Burn-off was carried out in air for Atmospheres II ~ VI. As a comparison, the burn-off stage for Atmosphere I was done in a nitrogen atmosphere. And for SRA stages, five kinds of atmosphere were tested: N_2 (neutral), O_2-containing N_2 (oxidative), H_2O-containing N_2 (wet), H_2-containing N_2 (reductive), and CO_2-containing N_2. In the furnace, two configurations for placing the samples were adopted. Samples were "individually isolated" or "stacked by a pressurized fixture" to simulate the most outer and the inner stampings of ES cores. For each test, isolated and stacked samples were annealed simultaneously in the same batch of thermal treatment.

2.4 Adhesion capability of coating of the annealed samples

After thermal treatment, adhesion capability of the coating to substrate was evaluated by Scotch tape (type 600). Upon fast removal of the tape from the annealed sample, clean tape without stain and intact surface of the sample without substrate exposure stand for excellent adhesion of the coating to the substrate. On the contrary, if coating adhesion deteriorates significantly after thermal treatment, exposure of bare substrate (pealing-off of the coating) and stains on the tape would take place. And the adhesion capability of the coating after thermal treatment could be differentiated by the extent of substrate exposure and tape stains.

2.5 Cross-sectional analysis of the coatings

Specimens were first vapor deposited by a thin layer of platinum (Pt). Then the surface was milled by dual-beam focused gallium ion beams (DB-FIB) (FEI Nova-200 NanoLab Compatible) to cut out a rectangular hole, from which cross sectional images could then be taken by scanning electron microscopy (SEM). And the

Table 1 Composition of the major alloying elements in the ES substrates of the two samples (SA, and SB).

Sample code	Composition of the major alloying elements	
	Si	Al
SA	~ 3.0%	~ 0.6%
SB	~ 1.9%	~ 0.3%

Table 2 Furnace atmospheres for the thermal treatment indicated in Fig.1.

Atmosphere code #	Stage of the thermal treatment and the used atmosphere						
	Burn-off		Stress-relieving annealing (SRA)				
	1	2	3	4	5	6	7
I	Neutral N_2		Neutral N_2 (145L/min)				
II	Air		Neutral N_2 (145L/min)				
III	Air		Oxidative N_2 (105L/min, to allow air leakage)				
IV	Air		Wet N_2 (135L/min, flow passing over 20°C water surface)				
V	Air		Reductive N_2 (130L/min N_2 + 20L/min H_2)				
VI	Air		CO_2-containing N_2 (125L/min N_2 + 25L/min CO_2)				

distribution of elements over cross sections could be revealed by mapping (the energy dispersion spectroscopy, EDS): Fe map for the substrate, P map for the coating, and O map for evaluating the extent of oxidation.

3. RESULTS AND DISCUSSION

In the thermal treatment process, stampings in ES cores are either exposed directly to the furnace atmosphere (the most outer surface) or are covered by adjacent sheets (probably under pressure, of the inner parts). Physical and chemical conditions of the thermal treatment in these two cases are thus different for the ES coatings. Practically, the appearance of stampings in an annealed ES core can be very different for the outer and the inner parts. To simulate the most outer and inner stampings of ES cores in thermal treatments, two configurations for placing coated ES samples ("individually isolated" and "stacked by a pressurized fixture") were adopted in this study to investigate effects of furnace atmosphere.

3.1 Samples annealed in the "individually isolated" configuration

After the thermal treatment (Figure 1 and Table 2), Scotch 600 tape was attached with finger pressure onto each sample and then torn off rapidly to evaluate the adhesion capability of the coating. Table 3 shows the scanned images of the ES samples annealed in the "individually isolated" configuration after the tape test.

The most obvious result in Table 3 is that the appearance of annealed samples can differ so much with the furnace atmosphere. In addition, for a specific SRA atmosphere, the post-annealing appearance of samples also depends on the substrate, suggesting that the alloying composition of ES should have effects. For a specific substrate, samples annealed in Atmospheres I and II (burn-off: air or N_2; SRA: both N_2) bear close resemblance. Obviously, burn-off at 275°C in neutral N_2 and in air should have similar effect. Interestingly, dark and

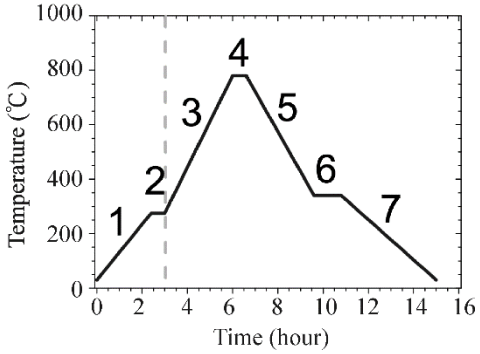
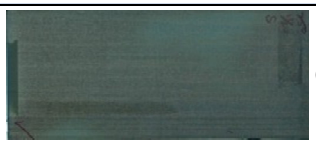


Fig.1. The heating profile for the investigated thermal treatments.

uniform appearance is provided by the thermal treatment in Atmosphere V (H_2-containing nitrogen, reductive) for both samples. As compared with other Atmospheres, the distinctness of Atmosphere V reveals that atmosphere-induced oxidation played a key role in the thermal treatment process of ES samples.

Both samples annealed in Atmosphere III (O_2-containing nitrogen, oxidative) revealed a very different appearance from Atmospheres I and II. Annealed sample SA is of pale color, whilst sample SB is reddish. Undoubtedly, such results should be related to the different chemical reactivity (i.e., resistance to oxidation) of the two grades of ES substrate. Since the chemical inertness of ES substrates does affect the anti-corrosion capability of coated ES, the salt spray test (SST, 16hrs) was also done on the two samples (as received, before thermal treatment). As shown in Table 4, sample SA shows only a few rust spots after 16hrs SST, whilst significant corrosion has occurred in Sample SB after the same test. Interestingly, Tables 3 and 4 reflect a clear correspondence between results of SST and oxidative SRA (Atmosphere III). This suggests SST can probably be an alternative to evaluate the capability of coated ES for SRA in oxidative atmospheres.

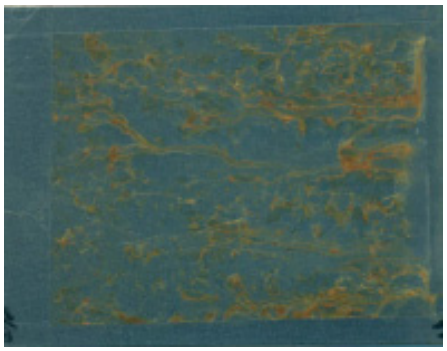
Table 3 Appearance of the ES samples annealed in the "individually isolated" configuration after the tape test (Scanned images: The black belt marks on the scanned images were caused by the scanner not a result of the test samples.)

	Atmosphere of thermal treatment		Annealed samples after the tape test	
#	Burn-off	SRA	SA	SB
I	N_2	N_2	 ◎	 ◎
II	Air	N_2	 ◎	 ◎
III	Air	$N_2 + O_2$	 ◎	 X
IV	Air	$N_2 + H_2O$	 ◎	 X X
V	Air	$N_2 + H_2$	 ◎	 ◎
VI	Air	$N_2 + CO_2$	 ◎	 ◎

Judgement:

◎ intact, ○ slightly peeled, △ locally peeled, X Significantly peeled, XX completely peeled

Table 4 Salt spray test (SST, 16hrs) results for the investigated coated ES samples (Scanned images).

SA	SB
	

SRA in the atmosphere IV (wet nitrogen, dew point: ca +20°C) turned both samples into grey appearance. After SRA in the Atmosphere VI (CO_2-containing nitrogen atmosphere), Sample SA also shows pale color, whilst SB is somewhat lavender.

Table 5 compares the tapes that were torn from the

Table 5 Tapes torn away from the ES samples annealed in the "individually isolated" configuration (Scanned images).

Atmosphere of thermal treatment			Tapes torn from the annealed samples			
#	Burn-off	SRA	SA		SB	
I	N_2	N_2		◎		◎
II	Air	N_2		◎		◎
III	Air	$N_2 + O_2$		◎		X
IV	Air	$N_2 + H_2O$		◎		X X
V	Air	$N_2 + H_2$		X X		X X
VI	Air	$N_2 + CO_2$		◎		◎

Judgement:
◎ no stain, ○ slightly stained, △ locally stained, X very stained, XX completely stained

annealed samples in the tape test (Tables 3 and 5 are not to the same scale). Most of the torn tapes are clean without any detachment or contamination. However, sample tapes from Atmosphere V (H_2-containing nitrogen, reductive) have carbon-like stains. Since there is no substrate exposure after the tape test, stains on the tape should be from the very surface of the annealed coating, while the main body of the coating still adhered firmly to the substrate. This result can be attributed to the coking process of the coating, in which oxidative pyrolysis loss of organic components was suppressed due to the presence of H_2. For Atmospheres I and II (neutral SRA atmosphere), all the annealed samples show neither substrate exposure (no peeling off for the coating after tape tearing) nor stains on the torn tapes, revealing good post-annealing adhesion capability in these two conditions.

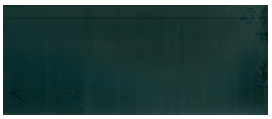
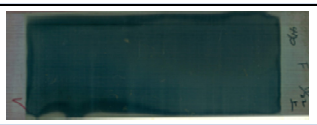
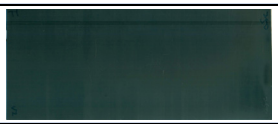
For Atmosphere III (O_2-containing nitrogen, oxidative), coatings of the annealed Sample SA still has very good post-annealing adhesion capability. However, Sample SB annealed in Atmosphere III shows moderate substrate exposure (coating peeled) in the tape test. An even worse result for Sample SB was obtained in Atmosphere IV (wet nitrogen, dew point: ca +20°C): the coating was torn away completely in the tape test and the substrate thus exposed (Table 3) is deep dark without gloss (probably due to the formation of Fe_3O_4 that resulted from the reaction between iron and humidity at high temperatures). On the contrary, Sample SA annealed in Atmosphere IV still shows good results. And this clearly reveals that substrate is also an affecting factor to the annealing capability for the coated ES products.

Samples SA and SB annealed in Atmosphere VI (CO_2-containing nitrogen) all retain good post-annealing adhesion capability for the coating. It follows that the influence of CO_2 on these samples should be insignificant or at least to a minor extent. As compared with Atmosphere II (neutral nitrogen) and III (O_2-containing nitrogen), Sample SB annealed in Atmosphere VI showed color in between. Therefore, Atmosphere VI should induce a degree of oxidation slightly higher than Atmosphere II but much less than Atmosphere III.

3.2 Samples annealed in the "stacked by a pressurized fixture" configuration

In addition to the "individually isolated" configuration, samples were also annealed as a pressured stack fixture. Table 6 shows the ES samples annealed in this configuration after the tape test. Tapes torn from these annealed ES samples are compared in Table 7 (Tables 6 and 7 are not to the same scale).

Table 6 Appearance of the ES samples annealed in the "stacked by a pressurized fixture" configuration after the tape test (Scanned images: The black belt marks on the scanned images were caused by the scanner, not a result of the test samples)

Atmosphere of thermal treatment			Annealed samples after the tape test			
#	Burn-off	SRA	SA		SB	
I	N_2	N_2		◎		◎
II	Air	N_2		◎		◎
III	Air	$N_2 + O_2$		◎		◎
IV	Air	$N_2 + H_2O$		◎		◎
V	Air	$N_2 + H_2$		◎		◎
VI	Air	$N_2 + CO_2$		◎		◎

Judgement:
◎ intact, ○ slightly peeled, △ locally peeled, X Significantly peeled, XX completely peeled

Table 7 Tapes torn away from the ES samples annealed in the "stacked by a pressurized fixture" configuration (Scanned images).

Atmosphere of thermal treatment			Tapes torn from the annealed samples			
#	Burn-off	SRA	SA		SB	
I	N_2	N_2		◎		○
II	Air	N_2		◎		△
III	Air	$N_2 + O_2$		◎		○
IV	Air	$N_2 + H_2O$		◎		△
V	Air	$N_2 + H_2$		X X		X X
VI	Air	$N_2 + CO_2$		◎		◎

Judgement:
◎ no stain, ○ slightly stained, △ locally stained, X very stained, XX completely stained

As compared with the "individually isolated" configuration (Tables 3 and 5), samples annealed as a pressured stack show relatively little difference in appearance and in the adhesion capability of the coating. All the annealed samples in this configuration are deep dark, regardless of furnace atmosphere and substrate grade. And the post-annealing adhesion capability of the coating is also good. Please recall that Sample SB annealed as "individually isolated" specimens in Atmospheres III (O_2-containing nitrogen, oxidative) and IV (wet nitrogen, dew point: ca +20°C) showed poor tape test results (Tables 3 and 5). Contrarily, Sample SB annealed as "a pressured stack" in these two atmospheres has good post-annealing adhesion capability for the coating (Tables 6 and 7). In addition, there are more or less carbon-like stains on the tapes torn from samples annealed in this configuration. Such results suggest that the oxidative pyrolysis loss of organic components in the coating had been somewhat suppressed, probably due to the unavailability of oxidants (i.e., O_2 and H_2O) within the pressured stack.

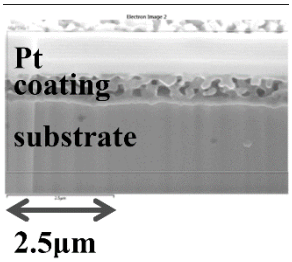
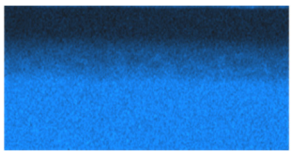
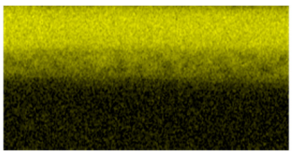
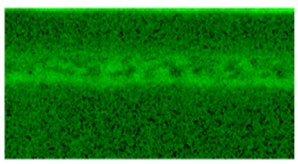
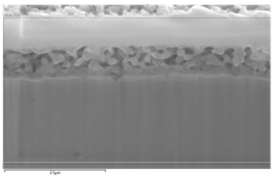
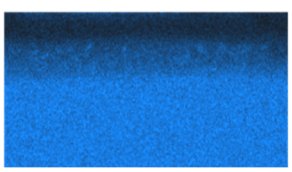
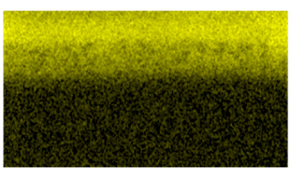
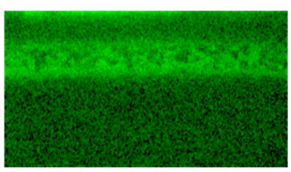
The above results are consistent with practical experiences. The inner part of annealed ES cores always has better batch-to-batch consistency than the outer stampings. Undoubtedly, stampings in a well-pressured stack are protected from the furnace atmosphere so that the atmosphere-induced reactions (e.g. oxidation) in the coating and on the ES substrate surface can be inhibited. Therefore, for ES stampings within a well-stacked core, thermal treatments should only lead to coking of the coating itself and the chemistry of ES substrate surface should not change much. However, for the outer part of ES core, chemical reactions with the atmosphere are more liable to occur. Therefore, for the outer part of ES core, careful control over thermal treatment parameters (atmosphere, temperature, time…etc.) is required for better batch-to-batch consistency in the appearance and the coating adhesion capability.

3.3 Effects of furnace atmosphere on the microstructure of the coating

In previous sections, the adhesion capability of the coating for samples annealed in different atmospheres has been revealed. Tables 3 and 5 clearly show that furnace atmosphere does have effects, but to what extent it can influence indeed depends on the ES substrate (the alloying composition). Among the two samples, SB is much more sensitive to the variation of furnace atmosphere. On the contrary, Sample SA always has good post-annealing adhesion capability, regardless of the furnace atmosphere adopted in the thermal treatments.

To further clarify, FIB/SEM/EDS analyses were carried out on samples annealed in the "individually isolated" configuration. And key results are shown in Tables 8 and 9 for Atmospheres II (neutral & dry nitrogen) and IV (wet nitrogen, dew point: ca +20°C), respectively. For all the images, it is the middle layer that presents the cross section of the coating. The top dense layer is the protective Pt film deposited prior to DB-FIB milling. And the bottom dense layer is the ES substrate.

Table 8 FIB, SEM, and EDS mapping of samples annealed in the "individually isolated" configuration with Atmosphere II (Cross section: platinum (the top dense layer); the coating (the middle layer); the ES substrate (the bottom dense layer)).

Sample	FIB / SEM 65000X	EDS mapping		
		Fe	P	O
SA				
SB				

Both samples annealed in Atmosphere II (Table 8) show well-defined cross sections, in which the interface between coating and substrate is clear-cut. This suggests that the chemistry of the substrate surface was changed little by the thermal treatment in Atmosphere II.

As shown in Table 9, sample SA annealed in Atmosphere IV also has clear cross sections. However, the microstructure of Sample SB annealed in Atmosphere IV is much more complicated and blurry. Comparing P maps of Sample SB annealed in Atmospheres II and IV, the coating layer is obviously thickened by the annealing in Atmosphere IV. Besides, Fe, P, and O maps of Sample SB annealed in Atmosphere IV clearly reveal the formation of an "oxidation zone on the substrate surface" (beneath the coating, i.e., the inner oxidation zone, IOZ) after this thermal treatment. These results could imply that the substrate of Sample SB kept reacting with the coating and the atmosphere for the duration of the thermal treatment in Atmosphere IV.

From Tables 3 and 5, Sample SB annealed in Atmosphere IV (in the "individually isolated" configuration) completely failed the tape test whereas that annealed in Atmosphere II still retained good adhesion capability. Table 8 shows that Sample SB annealed in Atmosphere II has no IOZ structure. Moreover, Sample SA annealed in Atmosphere IV, which still has good coating adhesion capability after the thermal treatment, doesn't have the IOZ structure either (Table 9). Undoubtedly, the adhesion failure of Sample SB annealed in Atmosphere IV should be attributed to the humidity-induced formation of IOZ in the surface of ES substrate (beneath the coating).

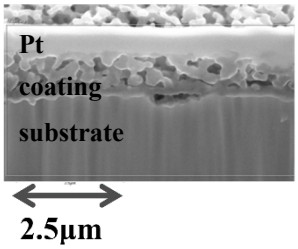
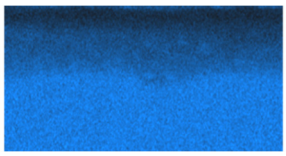
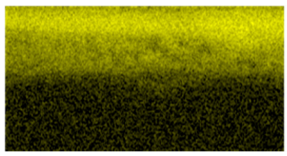
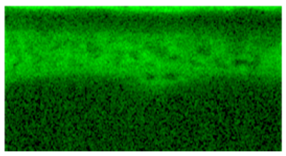
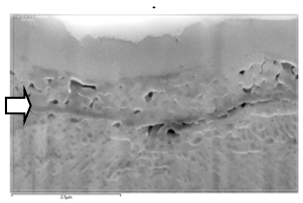
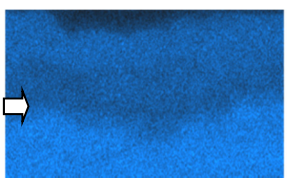
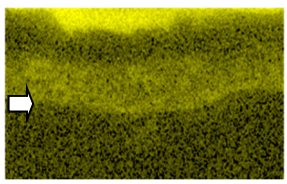
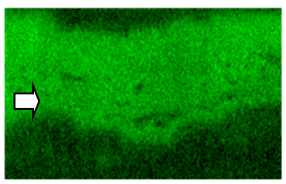
The formation of IOZ in ES has been reported for bare substrates by another group.[9] And we have also found that failure in the post-annealing adhesion capability of the coating can result from the formation of extended IOZ in coated ES that was annealed inappropriately.[6~8] In such cases, adhesion failure occurs on the "interface between the IOZ of substrate and the underlying un-oxidized ES" (interface located "within the substrate"), instead of the "interface between the coating and the substrate". It is not merely the coating but "the substrate IOZ attached to the coating" that is torn from the annealed specimen in the tape test.

The above findings reveal that both the furnace atmosphere and the ES alloying composition can be critical to the formation of IOZ in ES, which in turn will significantly affect the post-annealing adhesion capability of the coating. And the operation window of furnace atmosphere is dependent on the ES substrate of stampings that are going to be annealed. Therefore, optimization of furnace atmosphere by trial runs should be required for ES of different substrate grades to achieve optimal quality of thermal treatment results.

4. CONCLUSIONS

Our investigations have qualitatively revealed how the furnace atmosphere affects the post-annealing adhesion capability of the insulation coating to ES substrate. The outer stampings of ES stacks are much more liable to be affected by the atmosphere than the inner part. The presence of CO_2 has little effect and may be regarded as

Table 9 FIB, SEM, and EDS mapping of samples annealed in the "individually isolated" configuration with Atmosphere IV. White arrows indicate the presence of a inner oxidation zone (IOZ) formed in Sample SB after the thermal treatment.

Sample	FIB / SEM 65000X	EDS mapping		
		Fe	P	O
SA				
SB				

a neutral component in the atmosphere. Both O_2 and H_2O can deteriorate the post-annealing adhesion capability. But to what extent they can influence is whereas substrate-dependent. Upon inappropriate thermal treatments, the substrate surface (beneath the coating) can be oxidized significantly, by which the post-annealing adhesion capability of the coating will be fatally affected. Contrarily, H_2 has protecting effects so that good post-annealing adhesion capability of coating can be fulfilled regardless of the ES substrate grades. Therefore, low contents of oxygen & humidity (dew point) in the furnace atmosphere are required and the presence of some hydrogen in the furnace atmosphere can further help.

ACKNOWLEDGEMENTS

Mr. Chiu-Huo Li, Mr. Chi-Tsung Yeh, Mr. Yu-Ching Huang, and Dr. Hsin-Wei Lin are appreciated for their assistance in experiments.

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