Buildup Formation Mechanism of Carbon Sleeve in Continuous Annealing Furnace for Silicon Steel

Mingsheng He, Guohua Xie, Xuecheng Gong, Wangzhi Zhou, Jing Zhang and Jian Xu

Abstract Graphite, which has a unique stack structure that enables a small friction coefficient, is used as one kind of the best hearth rolls to support and convey silicon steel strip in continuous annealing furnace. However, the buildup formation in/on the surface of carbon sleeve has always been a worldwide difficulty in the silicon steel industry. Based on the analysis of the microstructure, topography and composition of the buildups, combined with simulation experiments, the mechanism of forming buildups is discussed. The results show that buildups experience the process from nucleating to growing-up, and buildups are formed by nano liquid phase sintering (NLPS). Meanwhile, some countermeasures to reduce or prevent the formation of the buildups are proposed.

Keywords Silicon steel · Buildup · Mechanism · Continuous annealing
Carbon sleeve

Introduction

Silicon steel strips are used in the core of a generator, a motor, a small size transformer and the like. Generally, silicon steel strip by hot or/and cold rolling process is decarburized, re-crystallized and annealed in continuous roller-hearth annealing furnace. Carbon sleeve is the most important kind of hearth rolls in the annealing furnace to support and convey silicon steel strips [1, 2]. Under the condition of the high temperature and H_2–N_2–H_2O weak oxidation or H_2–N_2 strong reducing atmosphere in the annealing furnace, buildups adhered (AD-buildup) to the surface of the carbon sleeve or buildups embedded (ED-buildup) in the surface of the carbon sleeve can form after using for a period of time, especially during the production of high-grade non-oriented silicon steel and some low- and medium-grade non-oriented silicon steel containing low melting point metal,

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ED-buildups are much easier to form. Once buildups come into being in/on the surface of carbon sleeve, they will easily indent, bruise and scratch the surface of steel strip, which seriously affects the quality of steel strip surface and even causes degraded or waste products [3]. The quality and life time of carbon sleeve have been troubling the production of continuous annealing line for silicon steel for worldwide iron and steel enterprises [1].

In recent years, it has greatly attracted research attentions to buildup formation cause and mechanism of carbon sleeve. Many researchers and specialists think that the following three points are the main causes of buildup formation for silicon steel strip during continuous annealing [4–7]: (1) Each kind of damage and defect (oxidization, grain boundary etching, score, pit or the like) is formed on the roll surface; (2) A formation of metal particles due to reduction of an oxide under a reducible atmosphere and an activation of metals; (3) Some oxides for example, Fe_2O_3, Fe_3O_4, FeO, SiO_2, Al_2O_3 and MnO are subjected to a solid phase reaction with each other. In general, iron and its oxides particles can not react with other oxides without external force at 800–950 °C, even below 800 °C to form very hard buildups. This study was to discuss the real reason and mechanism of buildup formation of carbon sleeve.

Materials and Methods

In order to determine the microstructure and compositions of buildups, the buildups are sectioned along radial or axial direction, inlaid, ground, and polished. And then the as-prepared samples were observed and analyzed by a scanning electron microscope (SEM, Quanta 400, FEI Co., Netherland) equipped with energy-dispersive X-ray spectroscopy (EDS).

Online sintering experiments: (1) Nano-Fe_2O_3 powder with an average particle size of 30 nm was put into the alumina crucible; (2) The crucible was placed in the zone easy to form buildups in continuous annealing furnace; (3) The sintering samples were taken out from the furnace after about three months, and the microstructures of sinter body of nano-Fe_2O_3 powder were examined.

Results and Discussions

Topography and Composition of Buildups

Figure 1 shows the ED-buildups forming in the surface of carbon sleeve during the decarburizing and annealing for non-oriented silicon steel strips. Figure 1a–c are the photos of ED-buildups in the surface of carbon sleeve in low- and medium-temperature zone of the annealing furnace. Figure 1d is the photo of

ED-buildup in the surface of carbon sleeve in high temperature zone of the annealing furnace. ED-buildups may be in different shapes, such as cylinder-like, cone-like, barrel-like, and truncated cone-like. Most of the ED-buildups are in cylinder shape, and the axial direction of buildups parallels to that of carbon sleeve. Generally speaking, ED-buildups in low- and medium-temperature zone are bigger than high-temperature zone. Meanwhile, the size and shape of ED-buildups have relations to the pore size and shape in the surface of carbon sleeve [1, 2].

Figure 2 shows the AD-buildups forming on the surface of carbon sleeve during the decarburizing and annealing for non-oriented silicon steel strips. Some AD-buildups are in flaky or willow leaf shapes (Fig. 2a), others are completely irregular in shape (Fig. 2b), and the formation of the buildups has no regularity.

Figure 3a is the cross section SEM micrograph of the ED-buildup along radial direction (Perpendicular to the axis of the carbon sleeve) in the low-temperature zone (LTZ) of annealing furnace; Fig. 3b is the longitudinal section SEM micrograph of the ED-buildup along axial direction (Parallel to the axis of the carbon sleeve) in the high-temperature zone (HTZ). As shown in Fig. 3a, it can be seen that the buildup is composed of light and deep color belts in alternately dark and bright annular concentric circles [1]. As shown in Fig. 3b, it can be seen that the HTZ buildup also has dark and bright annular structure same to the LTZ buildup.

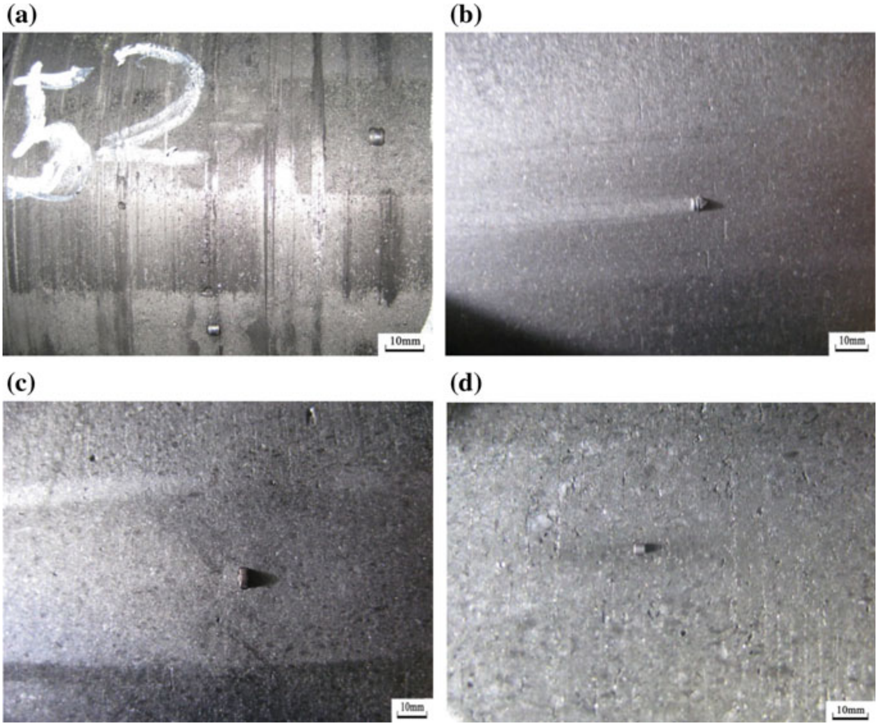

Fig. 1 Photos of ED-buildups in the surface of carbon sleeve

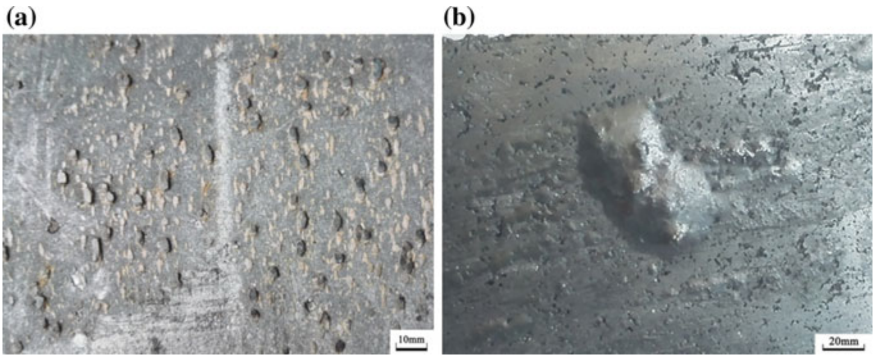

Fig. 2 Photos of AD-buildups on the surface of carbon sleeve: **a** flaky or willow leaf shapes; **b** irregular shapes

Therefore, the ED-buildup is formed gradually in the surface pore of carbon sleeve from layer to layer by accumulation. With the continuous growth of the buildup to a certain size, the buildup may have friction and wear with silicon steel strip. And then the buildup will further accelerate growth. Finally, the buildups can indent, bruise and scratch the surface of steel strip.

Figure 3c shows a ball-like ED-buildup forming in the surface pore of carbon sleeve, which is just bringing into contact with steel strip, but almost having no friction and wear with the strip. It was proved that the nucleation of buildup was formed in the surface pore of carbon sleeve and grown up gradually.

Figure 3d shows AD-buildups on the surface of carbon sleeve, which is irregular in shape. Although the AD-buildups were formed occasionally, it may result in the surface defects of steel strip. Once an AD-buildup was formed, it was very difficult to eliminate it by grinding rollers.

Figure 4a, b are the cross section partially enlarged SEM micrographs of the LTZ ED-buildup along radial direction of carbon sleeve. Figure 4c, d are the longitudinal section SEM micrographs of the HTZ ED-buildup along axial direction of carbon sleeve. Figure 4e, f are the partially enlarged SEM micrographs of the ED-buildup without abrasion in the surface of carbon sleeve and the AD-buildup on the surface of carbon sleeve, respectively. As shown in Fig. 4a, it can be seen that the grain sizes mostly fall in a range of 20–30 μm. However, the grain sizes (Fig. 4e, f) are only a few microns, even below 1 μm (Fig. 4b).

Figure 5a, b are respective EDS spectrums of the light belt and deep color belt of the LTZ ED-buildup. The EDS spectrum (Fig. 5a) of light color belt shows strong peaks of Fe and weak peaks of Al, Si, C and O. However, the EDS spectrum (Fig. 5b) of deep color belt shows strong peaks of Al, O, Fe, Si and weak peaks of Na, Mg and P. Figure 5c, d are respective EDS spectrums of the light color and deep color position of the HTZ ED-buildup. The EDS spectrum (Fig. 5c) of light color position shows strong peaks of Fe and weak peaks of Si. The EDS spectrum (Fig. 5d) of deep color position shows strong peaks of Si and weak peaks of Fe, Al,

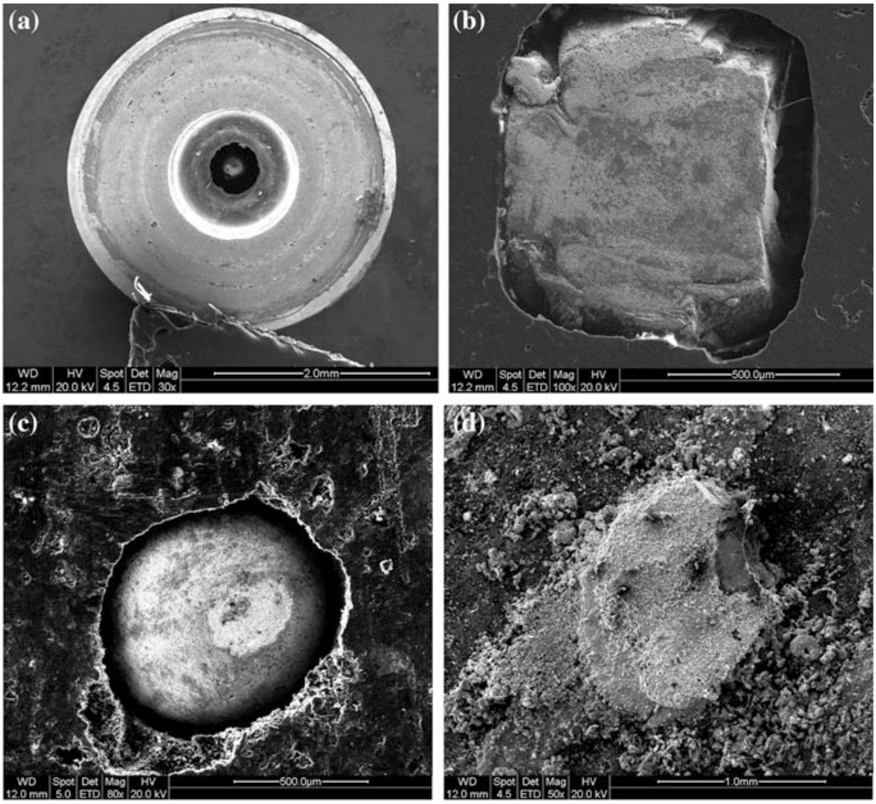

Fig. 3 SEM micrographs of the buildups: general view. **a** Cross section along radial direction; **b** longitudinal section along axial direction; **c** no friction and wear with steel strip; **d** AD-buildup

O, and Mn. Figure 5e, f are respective EDS spectrums of the light belt and deep color belt of the LTZ AD-buildup. The EDS spectrum (Fig. 5e) of light color position shows strong peaks of Fe, P and weak peaks of C and O in the AD-buildup. Nevertheless, the EDS spectrum (Fig. 5f) of deep color position shows strong peaks of Fe and weak peaks of Mn, P and O.

The results show that the main components of buildups are iron, a small amount of iron oxide, and oxides of Mn, Al, Si, or some compound oxides. Phosphorus content of some AD-buildups and ED-buildups of low- and medium-temperature carbon sleeve is relatively high [8]. Phosphate is widely used as an antioxidant in the production of low- and medium-temperature carbon sleeve. Meanwhile, phosphate is a high-temperature binder commonly used in various industries. Phosphate antioxidants can promote or accelerate the formation of buildups for low- and medium-temperature carbon sleeve during the continuous annealing of non-oriented silicon steel strips.

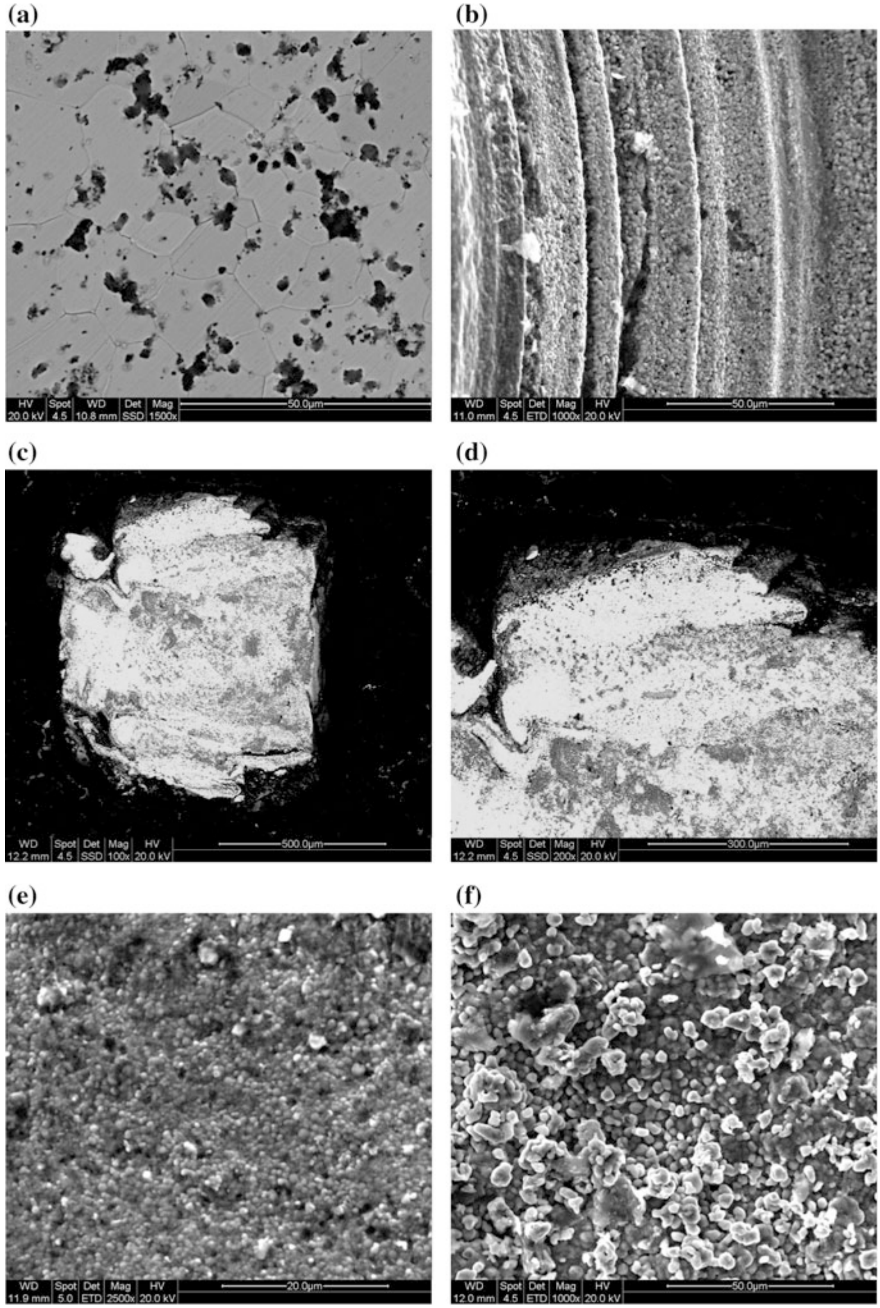

Fig. 4 SEM micrographs of the buildups: partially enlarged. **a** and **b** Along radial direction; **c** and **d** along axial direction; **e** no friction and wear with steel strip; **f** AD-buildup

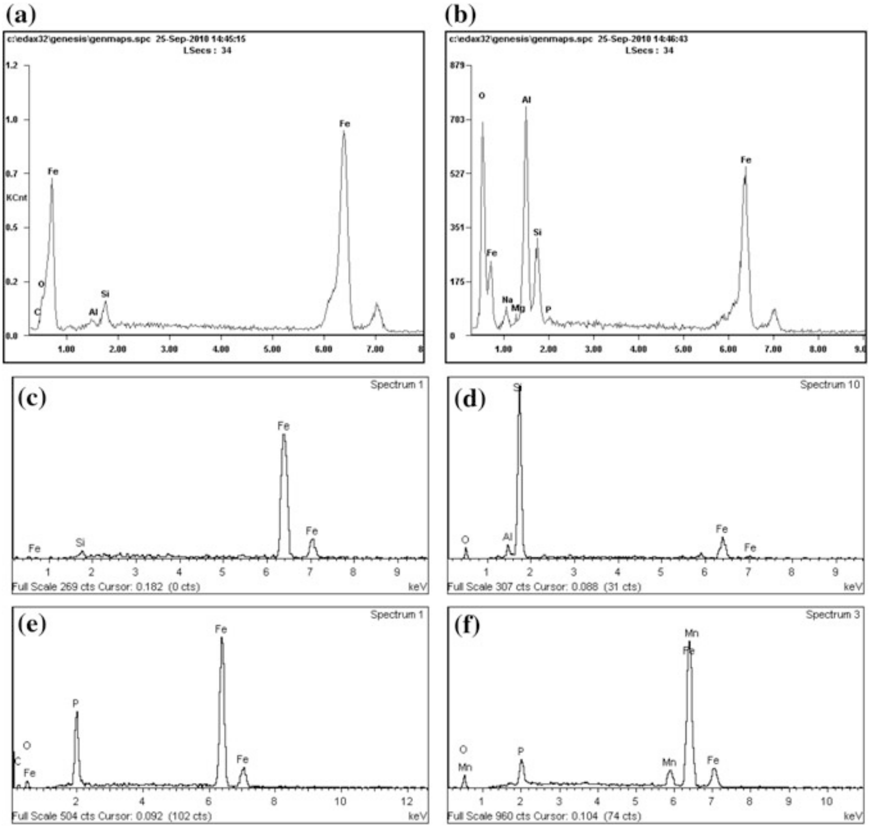

Fig. 5 EDS spectrums at different positions of the buildup. **a** Light color belt and **b** deep color belt in the LTZ; **c** light color and **d** deep color positions in the HTZ; **e** light grey and **f** dark grey position of AD-buildup

Causes for Buildup Formation of Carbon Sleeve

According to the results of analysis on the microstructure, topography and composition of the buildups, it can be seen that ED-buildups in the surface of the carbon sleeve experienced the "nucleation-growing up" process and the main components were iron, a small amount of binary oxides or compound oxides, and phosphide. Obviously, the two necessary conditions for ED-buildup formation were: (1) there exist big pores in the surface of carbon sleeve; (2) rich material sources such as iron scale, iron rust, greasy dirt, dust, etc [1, 2]. AD-buildups without regularity were formed on the surface of the carbon sleeve because of phosphate antioxidant, high temperature and special atmosphere in annealing furnace.

Graphite is chemically inert in non-oxygen media. At normal temperature and pressure, graphite does not make any chemical reactions except the long-term immersion in nitric acid, hydrofluoric acid or in fluorine, bromine and other strong oxidizing atmosphere leads to form intercalation compounds slowly. Graphite is not subject to any acid, alkali and salt corrosion and does not react with any organic compound. However, due to complex procedures and processes of producing carbon sleeves together with porous material itself, it is inevitable to form bigger holes ore pores in the surface during production and processing.

Generally speaking, graphitization degree of carbon sleeve is over 70%. Although the surface of carbon sleeve is smooth after soaking of various chemical substances and processing, graphite begin to react with water vapor over 700 °C. The carbon in silicon steel has great influence on the magnetic. Carbon not only strongly inhibits grain growth, but also expands γ phase zone. Hence, the excessive amount of carbon makes the shift quantity of two phases α and γ increase in normalizing treatment, refine crystal structure, and cause the increase in iron loss. One of the important purposes of the annealing to the finished product of silicon steel is decarburization. Therefore, holes or pores inevitably appear due to oxidation during the use of carbon sleeves in continuous annealing furnace for silicon steel. As a result, these holes or pores provide the necessary conditions for stubborn ED-buildups to form. When there are larger pores together with rich iron scale, iron rust, greasy dirt, dust, and other material sources, the stubborn ED-buildups could form in a relatively short time.

Many other factors influence buildup formation of carbon sleeves, such as atmosphere, dew point, uncleanness of alkali wash, outer-sync of the actual speed of carbon sleeves and the running line speed of steel strip, uncleanness of inside furnace, etc.

Buildup Formation Mechanism

The buildup has iron content from 70 to 95%. All the dense buildups are made up of micron sized particles, even smaller particles, and harder than silicon steel strip. However, how to form so dense and hard buildups without external force at 800–950 °C, even below 800 °C? It is well-known that the melting point is related to the size of the nanoparticle, and the smaller particles, the lower melting point. Based on investigation and analysis of the microstructure, topography and composition of the buildups, an idea was proposed that the buildup could be formed by liquid phase sintering of nano-Fe powder. Nevertheless, it was impossible that there were a great deal of nano-Fe powder on the surface of silicon steel trip or in the annealing furnace. Nano-Fe powder could come from nano-Fe_2O_3 or other nano-oxides of iron powder by the reduction of hydrogen or carbon.

Online simulation experiments were conducted in annealing furnace for silicon steel strip. Alumina crucibles containing nano-Fe_2O_3 powder were placed in different zone of annealing furnace. The results showed that all the powder had been completely sintered to form dense and hard sintering bulk after about three months. The microstructures and composition (Fig. 6) of sinter bulk were examined by

SEM and EDX. The grain sizes mostly fall in a range of 20–40 μm (Fig. 6b, c), and the chemical composition is only iron (Fig. 6d). The experimental results show that buildups experience the process from nucleating to growing-up, and buildups are formed by nano liquid phase sintering (NLPS).

Measures to Control and Reduce Buildups Formation

According to the present production technology, equipment and conditions, it is impossible to completely eliminate buildups, but it is possible to take some process control in technology or preventive measures to reduce buildups formation in continuous annealing furnace for silicon steel [1, 2]. The smaller pores in the surface of carbon sleeve are, and the less material sources for forming buildups such

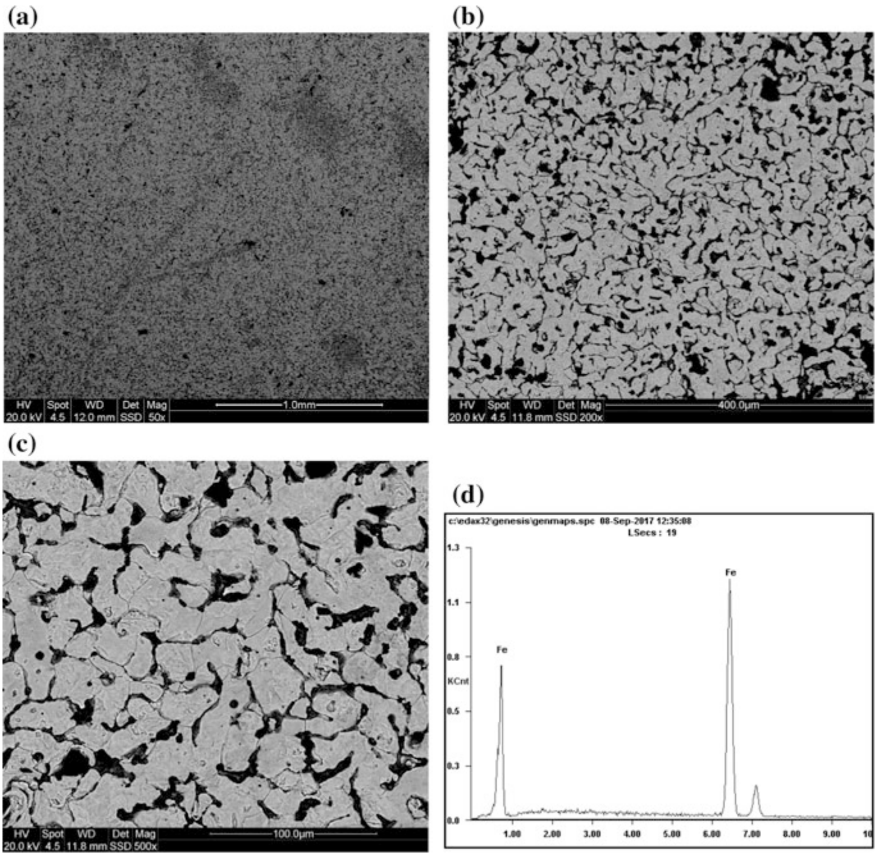

Fig. 6 SEM micrographs and EDS spectrum **d** of sinter bulk. **a** General view; (**b**) and **c** partially enlarged

as iron scale, iron rust, iron oxidation, greasy dirt on the surface of silicon steel strip are, the less the possibility of buildup formation is. In order to control and reduce buildup formation, extend the service life of carbon sleeve, the following methods and measures are proposed:

(1) Further improve the surface quality of carbon sleeve and enhance its oxidation resistance, wear resistance, and buildup formation resistance;
(2) Improve descaling equipment and process, strengthen the pickling, and reduce iron scale;
(3) Strictly monitor atmosphere and dew point to prevent steel strip oxidation;
(4) Strengthen the cleanness of steel strip and alkaline wash to reduce iron rust, greasy dirt;
(5) Clean dust regularly in continuous annealing furnace;
(6) Use the adjusting steel roll with good surface quality;
(7) Arrange the production properly;
(8) Monitor and correct the motor of carbon sleeves and adjust the inconsistent roller speed of carbon sleeves in time.

Conclusions

Buildups experience the process from nucleating to growing-up, and buildups are formed by nano liquid phase sintering. The pore sizes and shapes in the surface of carbon sleeve determine the sizes and shapes of ED-buildups. Phosphate antioxidants can promote or accelerate the formation of buildups for low- and medium-temperature carbon sleeve during the continuous annealing of non-oriented silicon steel. It is feasible and effective to take some process control in technology or preventive measures to reduce buildups formation and extend the service life of carbon sleeve and improve the production quality and production efficiency for silicon steel.

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