



Enhancement of siliconizing behaviors in pure iron induced by surface mechanical attrition treatment



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ABSTRACT

A gradient nanostructured surface layer of about 100 μm in thickness was synthesized on the pure iron induced by surface mechanical attrition treatment (SMAT). The average grain size in the top surface is about 20 nm. The formation behaviors of Fe-Si compound layer were investigated in nanostructured Fe during the electroless siliconizing process. In comparison with the coarse-grained (CG) sample, the thickness of compound layer on the SMAT sample is about twice larger than the CG sample and the diffusion activation energy decreased from 231 kJ/mol to 165 kJ/mol for the sample after SMAT 20 min. Evident inter-diffusion of elements such as Fe and Si could be found under the EDS analysis, suggested the achievement of metallurgical bonding. The porosity appeared at the interface of matrix and Fe₃Si layer in the CG sample due to Kirkendall effect, however, the bonding of the compound layer and matrix was tighter due to the promotion of the elements diffusion in the SMAT sample. The enhanced siliconizing kinetics is attributed to numerous grain boundaries (GBs) in the nanostructured surface and deformed layer.

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1. Introduction

Pure irons, as a kind of prominent soft magnetic materials, have been attracted enormous attention from both academia and industrial communities given their high saturation magnetic induction and steady performance [1,2]. However, the silicon content of pure iron, utilized in the field of altering magnetic, should be increased in order to lessen the eddy current loss. Therefore, silicon irons (Fe-6.5 wt.%Si) are utilized for transformer and motor cores, ascribed to their excellent magnetic permeability and minor iron loss [3–5]. It has been suggested that Fe-Si alloy (that is Fe₃Si compound) not only is of an excellent soft magnetic alloy but is characterized by good corrosion resistivity and high hardness value [6]. The precipitated phase Fe₃Si distributed homogeneously in the matrix pure irons and played an important role in soft magnetic properties. However, the plasticity of silicon iron is definitely reduced when the Si content exceeded 4 wt.% [5]. Siliconizing onto pure iron and reactive diffusion of Fe-Si alloy have been paid attention for numerous researches.

Several available approaches of Si incorporation into metals can be taken into account, including chemical vapor deposition (CVD) [5,7–11], pack cementation [12,13], electrodeposition [14–17] and electroless plating in the molten salt [18–22]. CVD and pack cementation approaches should be applied at the high temperature and in the airtight space. Otherwise, using the CVD method, Si content of the substrate

was not lower than 2.5 wt.% for prevention of phase transformation at high temperature. Rao et al. [15] electrodeposited silicon onto silver surfaces by electrolysis approach in fluoride melts under an inert atmosphere at 1018 K. By this means the temperature of siliconizing could be decreased, whilst it is required complex apparatus and rigorous conditions. Suzuki et al. [22] optimized the ingredient in the melts and siliconized on the refractory metals (Nb, Mo, Cr, Fe, Ni, Co) by electroless plating. Compared with electrodeposition, electroless plating was performed at lower temperature with the simple equipment under the condition of atmosphere. The thickness of Fe-Si layer depends mainly on the interdiffusion of Fe and Si [23].

New phases were synthesized through the interdiffusion of Fe and Si, such as Fe₃Si, FeSi, FeSi₂ [24] and Fe₂Si [23]. To investigate reactive diffusion kinetics, local equilibrium on the grain boundaries was hypothesized suggested by Wagner's researches [25], and the growth of the formed layer was quasi-steady. As to the mechanism of Fe-Si diffusion [26], Würschum et al. researched the Fe self-diffusion in both Fe-Si nanocrystallites and the CGs, and pointed out that the diffusion was enhanced in nanocrystallites, ascribed to low atomic ordering processes and the existence of thermal vacancy. It is well known that the high density of short circuit diffusion paths was provided through the crystal defects (that are grain boundaries, dislocations and vacancy etc.) in nanocrystalline materials, resulting in enhancement of the solute atoms diffusion [27–29]. Previous studies showed that the siliconizing behaviors would be enhanced on the nano/micro crystalline surface [30,31] but no experience had been found in the literature regarding the mechanism of the Fe-Si reactive diffusion.

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Surface mechanical attrition treatment (SMAT) has been proved to be a practical method to obtain nanostructured surface layers on metallic materials [32–37]. It has been demonstrated experimentally that reactive diffusion (or phase transformation) kinetics are facilitated in the SMAT samples at lower temperatures, such as chromizing [38], nitriding [39], galvannealing [40], aluminizing [41] and plasma electrolyte oxidation (PEO) [42,43]. A large number of GBs with various nonequilibrium defects would enhance nucleation and growth of new phases. In addition, the grain size in the new synthesized compounds formed on the SMAT samples was much smaller than those on the CG samples [38, 40], which would further enhance the reactive diffusion kinetics.

Siliconizing on the surface of pure iron or iron-base alloys has enhanced the soft magnetic property and protected alloys from corrosion [3,5,13,44]. The deformed layer of metallic surface induced by SMAT pre-process can enhance the surface energy and the density of defects to improve the reactive diffusion during the electroless siliconizing process. Otherwise, the effect of the plastic flow caused by ball collisions on mass transport and reaction was elucidated. In this study, the pure iron subjected to different SMAT time was siliconized using electroless plating in the molten salt. The formation mechanism of Fe-Si compound layers and reactive diffusion kinetics were discussed.

2. Experimental

2.1. SMAT process

As-extruded pure iron sheet with an elemental composition (wt.%) of 99.95% Fe was used as the experimental material. Pure iron plates were machined into coupons with dimensions of $\Phi 64 \times 3$ mm, annealed at 1203 K for 120 min in a vacuum furnace to eliminate processing stress and obtain homogeneous structure of α -Fe (approximately 100 μm). Prior to SMAT, samples were mechanically grounded by silicon carbide paper progressively down to 800 grit.

SMAT process was accomplished in a surface nanocrystallization machine (SNC-2) under vacuum conditions at room temperature. Fig. 1 illustrates the experimental apparatus of the SMAT process used in the present work. The vibrating head was operated with an ultrasonic vibration frequency of 20 kHz. Bearing steel (GCr15) balls of 4 mm in diameter were placed at the bottom of a cylinder-shaped vacuum chamber and resonated by the vibrating head. The sample was fastened on the vacuum chamber and the plastic deformation on the surface layer was induced by impact of the flying balls. Because the sample surface was plastically deformed with high strains and high strain rates from multiple directions, on the surface created a deformed layer with a grain size gradient. Therefore, it lends the opportunity to enhance the

reactive diffusion processes of Fe and Si. In this work, the treatment duration lasted for 20 min, 40 min and 60 min at ambient temperature, respectively. No detectable contamination was introduced into the surface layer during the SMAT.

2.2. Electroless siliconizing in the molten salt

The SMAT samples were cut into small coupons with dimensions of $5 \times 5 \times 3$ mm and degreased with 95% ethanol in an ultrasonic water bath for 10 min, rinsed with running deionized water and dried in air at ambient temperature. Electroless siliconizing was conducted at the compound salt of NaCl, KCl, NaF, Na_2SiF_6 and Si with mole ratio of 36:36:18:5:5, whose lowest liquidus temperature is 900 K. As-SMAT samples were immersed into the melt salt solution and held for 1 h, 3 h and 5 h at 923 K and 973 K, respectively. After siliconizing, the samples were picked up from the molten salt and cooled rapidly in air. The salt adhered on the surface of the samples was removed in water, ethanol and acetone in order, and then dried in air and weighed with an electronic balance with 0.1 mg accuracy.

2.3. Microstructural characterizations

Microstructure development of SMATed samples was examined by an Axiovert25CA (Zeiss) optical microscope. Phase composition of the siliconizing samples was examined by TD-3500 X-ray diffraction with $\text{Cu K}\alpha$ radiation ($\lambda = 0.15418$ nm). The average grain size was calculated from line broadening by using the Scherrer and Wilson method [45]. Cross-sectional morphology of samples was performed by scanning electron microscopy (JSM-6700F) equipped with EDS. H-800 transmission electron microscope (TEM) was used to examine the microstructures of the surface layer. The plane view on the surface layer of TEM foils was obtained first by polishing the sample mechanically on the untreated side until it was about 30 μm thick; followed by ion milling using a Gatan PIPS with a small incident angle.

3. Results

3.1. Microstructure characterization of the SMATed Fe

To investigate the evolution of microstructure, the cross sectional characterization of the pure iron subjected to SMAT for various durations are shown in Fig. 2. According to linear intercept method, the average grain size of original pure iron heat treatment at 1203 K for 120 min is approximately 100 μm . It is found that microstructures of the treated surface layer differ markedly from those in the CG matrix. Grains in the surface layer are significantly refined and grain boundaries could not be identified as in the matrix. The thickness of the deformed layer varies from ~ 30 μm to ~ 100 μm with the treatment duration increasing from 20 min to 60 min. The structure in the severely deformed layer could govern interdiffusion along the short-circuit diffusion paths [46,47].

XRD patterns of the annealed Fe and SMATed for 40 min are shown in Fig. 3. The phase composition is α -Fe, and no new phase determines from the XRD results of SMATed samples. After SMAT, the diffraction peaks of SMAT sample display a visible broadening of Bragg reflections relative to the CG sample, attributed to the grain refinement and the presence of a high level of microstrain. Average grain size can be calculated by the Scherrer–Wilson equation, in which the average crystallite size is derived from the breadths at half maximum intensity of the measured peaks. The calculated result shows that the average crystallite size of as-SMAT sample is approximately 18 nm. It is a clear evidence of microstructure refinement induced by SMAT. The large density of point defects and dislocations in the deformed layer may enhance the atomic mobility [48].

The TEM morphologies of the topmost surface layer in the SMATed sample for 40 min are demonstrated in Fig. 4, (a) bright-field image

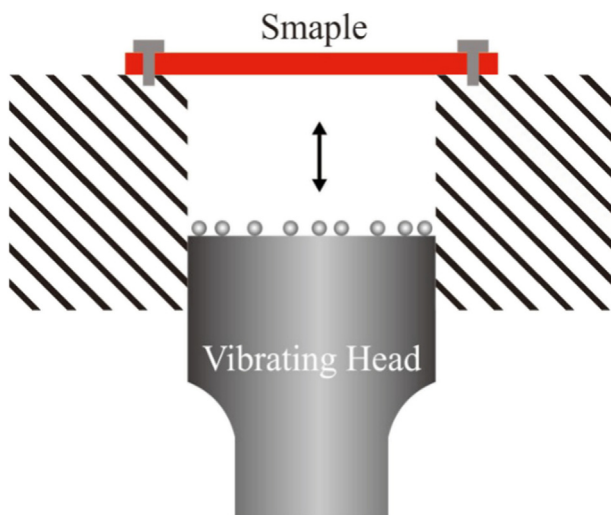


Fig. 1. Schematic illustration of the SMAT apparatus.

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