

PRODUCTION AND CHARACTERIZATION OF SILISIDE COATING ON MIRRAX ESR ALLOY BY PACK CEMENTATION METHOD

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Abstract

The pack-cementation technique was used to manufacture iron (Fe) silicides on Mirrax steel in this study. In an open atmospheric furnace, pack siliconizing process was performed using silicon (Si), ammonium chloride (NH₄Cl), and alumina (Al₂O₃) at 900-950-1000°C and with variable deposition periods of 2-4-6 h. Utilizing scanning electron microscopy-energy dispersive X-ray spectroscopy, the appearance and structure of silicide layers were examined. It was noted that the silicide layers, which were smooth, uniform, and lacked notable porosity, had good bonding with the matrix. By using X-ray diffraction analysis, dominant phase is Fe₃Si was determined. The coating layer thickness increased with process time and temperature. Additionally, the activation energy for the development of the siliconizing layer is determined. Due to the creation of a layer with a smoother morphology, less porosities and oxygen impurities, and an appropriate thickness silicon layer have been obtained.

Keywords: Pack siliconizing, coating, kinetic, activation energy.

1. INTRODUCTION

The remarkable ability of Mirrax steels to maintain strength at elevated temperatures, along with their favorable qualities against wear and corrosion, accounts for their widespread use. The primary use of this steel is in the polymer mould steels [1]. Generally speaking, coating deposition can enhance surface characteristics including hardness, wear, and corrosion resistivity without affecting the ductility of the metals and alloys.

Physical vapor deposition (PVD) and chemical vapor deposition (CVD) are the most often used techniques for coating steel surfaces. Halide activator pack cementation (HAPC) is one CVD technology [2-4]. Pack cementation is an inexpensive, simple-to-use technique that may produce coatings on components of various sizes and complex shapes. It is possible to use the pack siliconizing process to various metals and alloy compositions. In contrast to other surface treatment methods, the pack cementation method is primarily used to protect steel due to its straightforward processing route and quick processing time, which lowers the cost of the coating [5,6]. It can also be used to fabricate the coating on large, complex segments and geometries. The coating powders, filler material, and activator are combined during HAPC, and the cemented powders are heated at a high temperature to allow the halide compounds to activate them and react with the steel surface to form various coating layers. HAPC has the potential to significantly enhance the surface qualities of steels [2,6].

The process of rapidly creating Fe-Si intermetallic compounds results in the production of a stable, extremely hard layer that is protective. The process parameters must be optimized in order to achieve the necessary layer thickness in a fair amount of industrial time. Pack siliconizing steels exhibits the capacity to improve wear

strength and high temperature oxidation, while also enhancing the mechanical properties of the surface with excellent bulk ductility retention. Achieving optimal adherence of the coating layer to the substrate is dependent upon the production and uniform distribution of precipitation along the coating layer, which is a crucial aspect in the fabrication of hard compounds on the substrate surface [2], [3], [7].

The current study examines the production and growth mechanisms of the silicon coating relatively in a low temperature and duration time, comparing with the previous studies than that of in the literature by pack siliconizing method on the Mirrax ESR steel. In addition that, It was aimed to create a silicide-based coating on Mirrax steel and by this way improve its surface properties via the intermetallic-based coating obtained on the surface of the steel.

2. MATERIALS AND METHOD

Commercial Mirrax ESR steel is used as the substrate material, and mass spectrometry was used to determine the alloy's composition. **Table 1** lists the final chemical composition of the alloy. For the pack siliconizing process, experimental samples of "1 cm × 1 cm" were used. Following conventional metallographic process, cross-sections of the specimens were polished and acetone cleaned. The halide activator was NH_4Cl , and the inert filler was Al_2O_3 . Si source was Si in this paper. The pack were then heated in a furnace. The treating temperature of the process was in 900-1000°C setting the treating time from 2 to 6 h. The heating rate in the furnace employed was slow ($10^\circ\text{C}\cdot\text{min}^{-1}$) The main mechanism can be seen in schematic representation of pack siliconizing in (**Figure 1**).

X-ray diffraction analysis was used to characterize the presence of the silicides that developed on the Mirrax substrate (XRD). For the purpose of characterizing the coating layer of the test samples, a Cu K α radiation source with a wavelength of 1.541 Å across a range of 20° to 90° was used in a Rigaku X-ray diffractometer (DMAX 2200). The dispersion of the phases in the coating layer was examined using an energy dispersive spectroscopy (EDS) to ascertain the distribution of alloying components and equipment, as well as an electron microscopy (SEM, Model JEOL JSM-6060, Japan).

Kinetic investigations were carried out using steel samples to ascertain silicon's activation energy during the siliconizing process. First, the slope of the square of the layer thickness (x^2)-siliconizing time (t) curves was used to calculate the diffusion coefficients for each process temperature. The activation energy of the siliconizing process was calculated as 205 kJ/mol based on the slope of the $\ln D-1/T$ curve, which was obtained after the diffusion coefficients of the siliconizing process were determined for each sample.

Table 1 Chemical Composition of Mirrax ESR alloy

Element (wt%)	C	Si	Cr	Mn	Ni	V	Fe
Mirrax ESR	0.25	0.35	13.35	0.55	1.35	0.35	Balanced

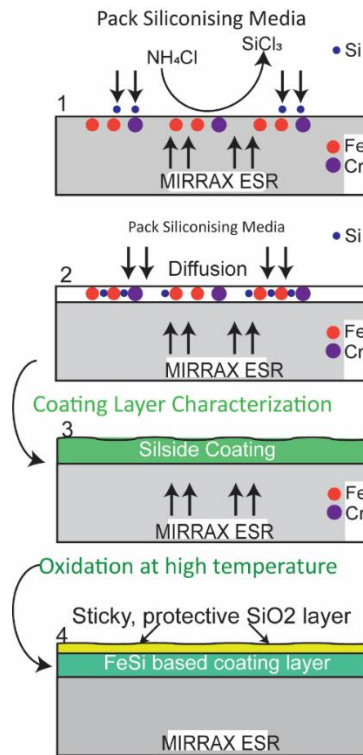
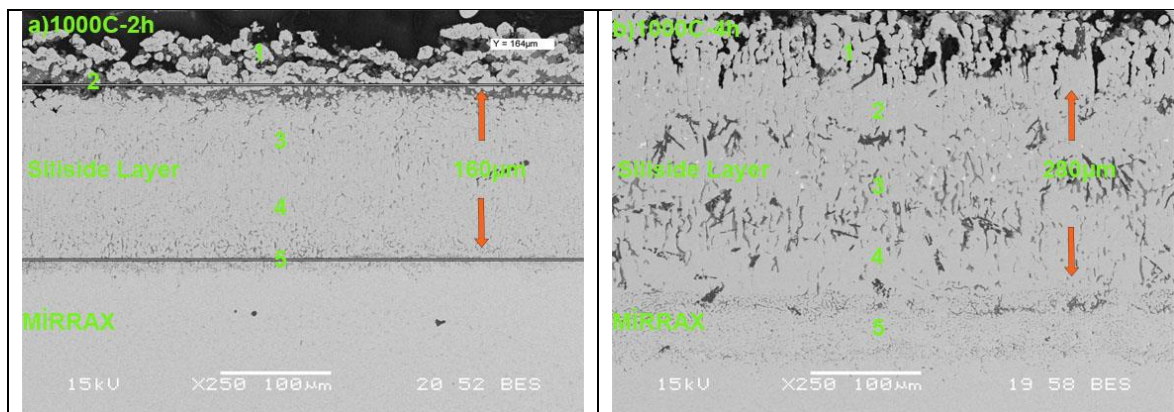


Figure 1 Schematic representation of Silicide Coating Process “[own study]”

3. RESULTS AND DISCUSSION

The SEM analysis belongs to microstructure of the cross-section results in **(Figure 2)** are supported by the EDS analyzes given in **Table 2**, and it is clearly seen that a very thick silicide layer is formed on the Mirrax steel. It is observed that the layer thickness increases rapidly with increasing processing time. However, during the 2-hour processing period, the accumulations did not condense on the upper surface of the coating and a dark area rich in oxide was observed on top surface. The oxide formed on the surface during the siliconization process is defined as SiO_2 , and it can be said that this oxide is a stable oxide such as Al_2O_3 and Cr_2O_3 [8]. **Figure 2b**, Point No. 1 has the largest silicon concentration among the other points, according to the EDS data, and this is because it has the highest silicon accumulation and less Cr deposition for 4 hours. Contrary this, surprisingly, at point 2, Cr amount is relatively high concentration as 9wt%. However, for 6 h process time for point 1 Cr deposition also increased when it is compared with 4 hours. And a homogeneous distribution of the elements was obtained for 6 hours. **Table 2** provides further information. The chemical composition of the coating on the cross-section, as obtained by EDS line scan, is explained in **(Figures 2a, b, and c)**.



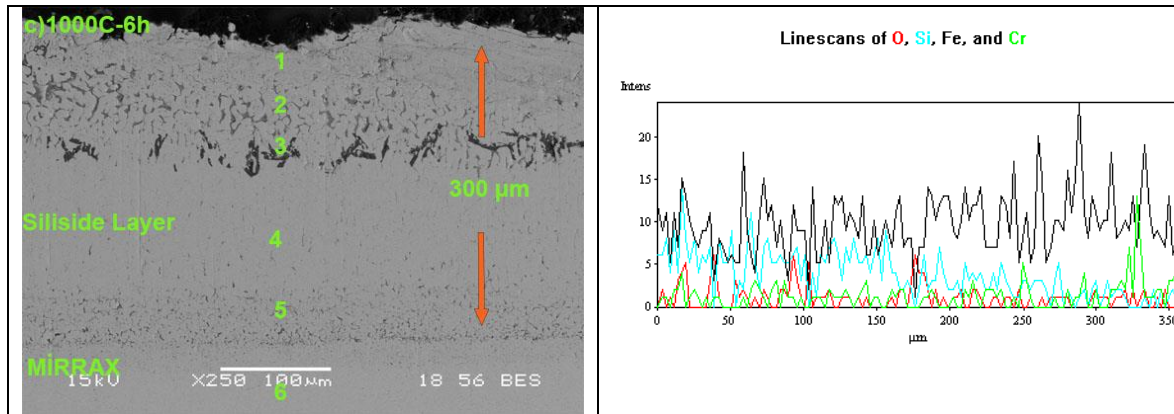


Figure 2 SEM-EDS analyses of of Silicide Coating Process applied for a) 1000°C-2h, b) 1000°C-4h, c) 1000°C-6h

Table 2 SEM-EDS Analyses of Siliconised Mirrax Esr alloy

Element (wt%)	Fe	Si	Cr	Ni
1000-2h (Fig.1a)				
1	82.580	12.272	3.373	1.775
2	39.928	32.937-O-22.355	3.304	1.476
3	83.672	10.454	3.635	2.238
4	84.364	9.315	4.560	1.761
5	86.960	0.670	11.652	0.718
1000-4h (Fig.1b)				
1	80.662	15.815	1.913	1.610
2	74.946	14.624	9.017	1.413
3	81.720	14.355	2.160	1.765
4	86.213	9.780	2.493	1.514
5	90.859	3.392	3.241	2.507
1000-6h (Fig.1c)				
1	80.837	12.958	4.076	2.128
2	82.845	11.417	3.876	1.862
3	84.003	10.242	4.525	1.230
4	85.647	8.710	4.207	1.436
5	86.888	7.022	4.020	2.070
6	87.331	4.933	5.755	1.591

According to XRD Analyses results of coated Mirrax alloy is shown in **(Figure 3)**. To this analyses the main phases are detected as Fe₃Si and FeSi. EDS analyses is supported this result.

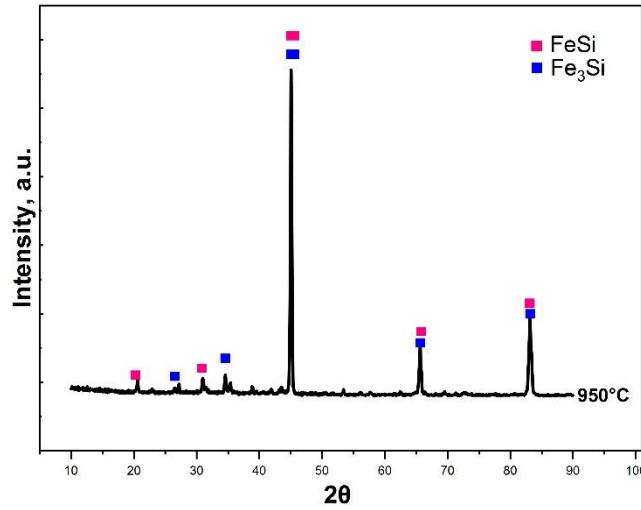


Figure 3 XRD analyses of Si coated Mirrax sample

The thickness of silicon layers varies between 130 and 300 μm , upon the temperature and duration of siliconization. There is no doubt that the base steel's alloying element, siliconizing temperature and time, and siliconizing duration are all directly related to the thickness of the silicide layer. As is well knowledge, the substrate and pack compositions of the process can have an impact on thermochemical coatings such as boronizing, aluminizing, chromizing [9], siliconizing [2], etc.

Kinetic investigations were carried out using steel samples to ascertain silicon's activation energy during the siliconizing process. First, the slope of the square of the layer thickness (x^2)-siliconizing time (t) curves was used to calculate the diffusion coefficients for each process temperature. The activation energy of the siliconizing process was calculated as 205 kJ/mol based on the slope of the $\ln D-1/T$ curve, which was obtained after the diffusion coefficients of the siliconizing process were determined for each sample. The relationship between siliconizing time and silicide layer thickness is almost parabolic, as seen in (Figure 4).

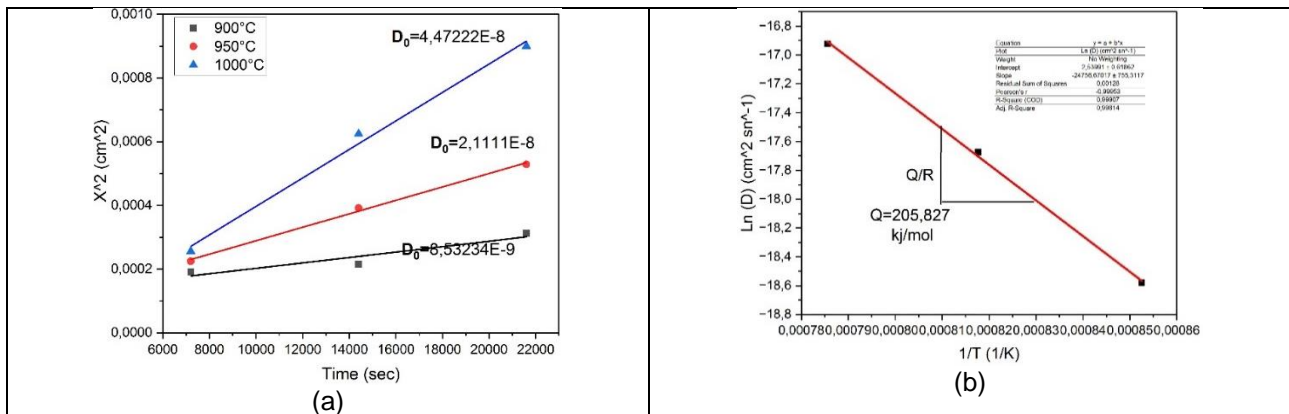


Figure 4 (a) The siliconized Mirrax steel's silicide layer thickness squared against treatment time, and (b) The siliconized Mirrax steel's growth rate constant against temperature

4. CONCLUSION

The paper can be summarized as follows:

Siliconizing process is applied in this study at 950 to 1000 degrees for 2, 4, 6 hours, it was seen that the increasing temperature was effective in the thickness of the coating layer.

The main phase is detected as Fe_3Si on the coating layer.

Activation energy (Q) for the present study was determined as 205 KJ. mol⁻¹. The growth rate constant (D) ranged from 8,53125.10⁻⁹ to 4,47222.10⁻⁸ cm².s⁻¹.

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