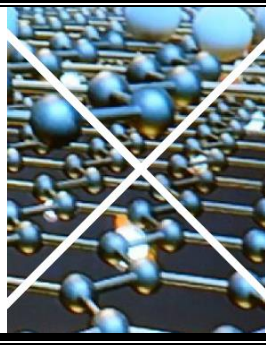
TIJMET, Vol. 0004 (2021) 109-116

The International Journal of

Materials and Engineering Technology

ISSN: 2667-4033
TIJMET ID: 210402003 (8 pages)
Type of the Paper: Research Article
Received: 18.11.2021 Revised: 07.12.2021 Accepted: 20.12.2021 Published (Online): 22.12.2021
Journal homepage: http://dergipark.gov.tr/tijmet



STRUCTURAL AND SURFACE PROPERTIES OF GLASS POWDER COATED Cr STEELS

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Abstract

The most common used glasses are soda-lime-silica glasses, which contain alkaline and alkaline earth oxide materials, such as silicon, sodium, aluminum oxides, *etc*., and these soda-lime-silica glasses are used in different applications such as windows, bottles, *etc*. Glass powder is acquired by grinding of waste glass cracks and applied as an additive in structural materials due to its compositions. In this study, the effect of glass powder on the surface and structural properties of Cr steel sheets has been investigated. Cr steels coated with dehydrated glass powder have been annealed at 950°C temperature in the furnace for various hours and cooled to room temperature in the furnace. The surface and structural properties of these coated steel sheets have been investigated by optical microscopy images, scanning electron microscopy images, and energy dispersive x-ray spectroscopy data. The crystal structures of these sheet samples have been recorded with x-ray diffraction patterns. As a result, glass powder on the surfaces of Cr steel sheets has not melted homogeneously and surface appearances of these steel sheets have been obtained as waved textures.

Keyword: Cr steel, glass powder, heat treatment, structural property, surface property.

1. Introduction

The glass includes non-crystalline silicate structures consisting of oxides, such as CaO, MgO, and Al_2O_3, and has an amorphous structure [1, 2]. This amorphous structure plays a role in the corrosion resistance of materials [2]. By milling of waste glass cracks, the glass powder is obtained and used as additive or coating materials in the structural materials due to its compositions

How to cite this article:
Acar, A.N., Kaya D., Eksi, A.K., Ekicibil, A., Structural and Surface Properties of Glass Powder Coated Cr Steels, The International Journal of Materials and Engineering Technology, 2021, 4(2): 109-116.

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[3, 4]. Waste glass powder consists of high silica constituents which provide high thermal stability, resistance to air conditions, hydrophobic behavior, low surface tension, and high oxidation resistance. The glass powder coating provides to enhance thermal stability, resistance to air conditions, and corrosion resintance of materials [5]. In literature; there are studies about physical and corrosion resistance properties of surfaces of steels coated with glass/glass powder [5-9]. Q195 steel wire was drawn via crucible filled with metallic alloys prepared by arc-melting pure metal elements in a Ti-gettered argon atmosphere and fastly cooled to metallic glass coating. It was observed that metallic glass coating consisted of metallic glass alloy and other intermetallics and metallic glass coating played on the enhancing of corrosion resistance of these steel wires [7]. Metallic glass prepared by arc-melting pure metal elements in an argon atmosphere was applied to surfaces of Q195 steel wires with continuous coating processes. It was observed that applied bulk metallic glass coating had a positive role in improving the corrosion resistance of steel wires [8]. Metallic glass alloy film of alloy prepared by vacuum melting technique deposited on the 316L austenitic stainless steel substrates with DC magnetron sputtering technique. The authors observed that the surface morphology of this film presented a smooth surface on the SEM images of this film [9]. Also, these processes play important roles in the evaluating of waste glass cracks for industrial applications and environmental aspects. Cr steels are one of the most used materials for blading materials in steam turbines to induce high corrosion resistance and high melting point [10, 11]. In our study; dehydrated glass powder consisting of silica, alumina, *etc.* coated to the surface of Cr steel sheets (2 mm of thickness) annealed at high temperature in furnace for 1, 2, and 3 hours. Glass powder film on the surfaces of these steel sheets occurred. These film layers on these sheets were examined on the surface and structural properties of these structures.

2. Material and Method

Scanning electron microscope (*SEM*) image and energy-dispersive x-ray spectroscopy (*EDS)* spectrum of Cr steel sheet with a thickness of 2 mm, which is used in this study, are presented in Figure 1 and Table 1, respectively. The chemical composition of glass powder which was acquired from Çinikoop (Kütahya, Turkey) was given in Table 2. Before the glass powder was applied to surfaces of Cr steels, the glass powder was dehydrated in an oven at 100°C temperature for 24 h [4]. Differential scanning calorimetry (*DSC*) measurement of the glass powder was presented in Figure 2 [12]. When glass powder having an amorphous structure is heated to high temperatures, it is considered that the first peak shows the exothermic reaction occurred at relatively low temperatures and refers to structural softening of the amorphous phase (at approximately 200-250°C temperature range) [13]. At 697°C and approximately 900°C temperatures, two endothermic peaks are related to crystalline phase throughout heat treatment and these indicate that glass powder composition includes crystalline phases [14]. Exothermic reaction at 950°C temperature shows the crystallization temperature of glass powder. At a higher temperature than 950°C temperature, glass powder begin to melt [15]. We selected 950° as annealing temperature from this graphs [12]. These findings have been confirmed by the X-ray diffraction (*XRD*) patterns [14].

Table 1. The EDS spectrums of Cr steel.

Element	Weight %	Atomic %
CrK	17.71	18.85
FeK	74.24	73.57
NiK	8.05	89.05

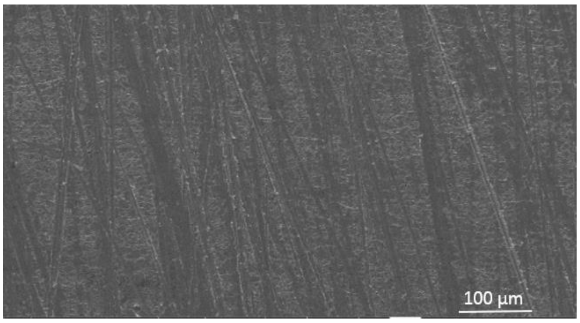

Figure 1.The SEM image of Cr steel

Different surface properties of Cr steel sheets were prepared after ground and polished. One of the sheet surfaces was not coated with glass powder as a control sample and the other sheets were coated with dehydrated glass powder with a thickness of 2 mm. The coated steel sheets were heated to 950°C temperature and annealed for 1, 2, and 3 hours in a furnace. Following we cooled the samples to room temperature in the furnace atmosphere. The surface and microstructural behaviors of coated and non-coated steel sheets have been imaged with an optical microscope (*OM*) and SEM, and also, the composition and the crystal structures of the samples were determined by EDS data. SEM images and EDS spectrums of non-coated and coated samples were obtained from FEI Quanta 650 Field Emission SEM device hıgh vacuum and 30.00 kV EHT. XRD patterns of all samples were recorded using PANalytical EMPYREAN XRD with a scan range of 20° to 90°, a voltage of 45 kV, tube current of 40 mA, Cu of anode material (λ=1.54Å).

3. Results and Discussions

Figure 3(a) presents the non-coated Cr steel sheet after being grounded, polished and clean and the coated Cr steel sheets with dehydrated glass powder at 950°C temperature for 1 hour (b), 2 hours (c), and 3 hours (d). After heat treatment at 950°C temperature, the glass powder is melted and glass-ceramic layers on the surfaces of these steel sheets are formed and permeated. As increasing of waiting time from 1 hour to 3 hours at 950°C temperature in the furnace, a smaller porous appearance on the melted glass powder on the surface of the steel sheet occurred (seen in Figure 3(c) and (d)). Also, the melted glass powder dispersed to all surfaces of this sample. In Figure 3(b), it was observed that melted glass powder was penetrated the surface of the steel sheet more than other coated steel sheet surfaces. Generally, on the images of these sheet samples, the glass powder on the surfaces of Cr steel sheets had not completely melted and surface appearances of these steels sheets had been obtained as waved textures.

Table 2. The chemical composition of glass powder (wt %)

Chemical Composition (wt %)			
SiO_2	68.92	**MgO**	4.07
Al_2O_3	1.81	**Na_2O**	16.00
Fe_2O_3	0.10	**K_2O**	0.42
TiO_2	0.06	**Other**	0.49
CaO	8.19		

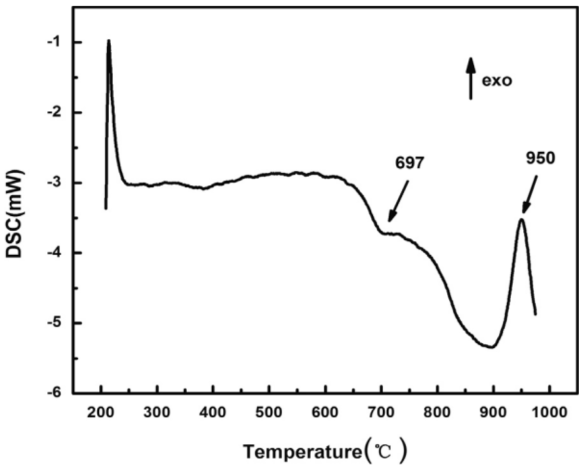

Figure 2. The DSC of glass powder [12]

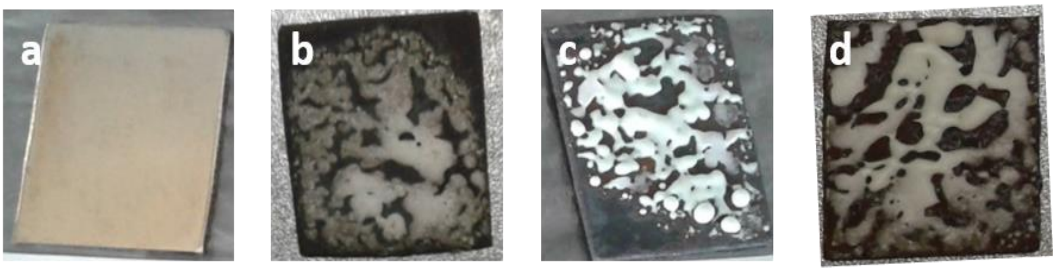

Figure 3. The photographs of non-coated Cr steel sheet (a) and Cr steel coated with dehydrated glass powder at 950°C temperature for 1 hour (b), 2 hours (c) and 3 hours (d)

In this study, the Cr steel sheet is a typical stainless steel sheet. Figure 4 gives the OM images of non-coated Cr steel sheet (a) and Cr steel coated with dehydrated glass powder at 950°C temperature for 1 hour (b), 2 hours (c, d), and 3 hours (e, f) with a 50X magnification. Figure 4(a) presents the OM image of a non-coated Cr steel sheet. The microstructure of the non-coated Cr steel sheet includes ferrite surrounded with austenitic-martensitic matrix [16]. Surfaces of all coated steel had been covered partly glassy layer. In Figure 4(b), a porous appearance on the melted glass powder on surface of the Cr steel sheet was observed. For Figures 4(c-d and e-f), an oxide layer formation was observed on the surface of the Cr steel sheet and glass-ceramic is formed via melting of the glass powder. These ceramics were dispersed, waved appearances on the surfaces of these steel sheets. We observed that the surfaces of these steel sheets had been covered partly by a glassy layer.

Figure 5 presents the SEM images of non-coated Cr steel sheet (a,b) and Cr steel sheet coated with dehydrated glass powder at 950°C temperature for 1 hour (c, d), 2 hours (e, f), and 3 hours (g, h). When annealing temperature is increased to 950°C, cracking and oxide accumulation on the surfaces of the steel sheets are observed. In Figure 5(e and f), cracking on the surface of steel sheet is clear. At this temperature, the primary glass powder begins to melt on the surfaces of the steel sheets. The surfaces of steel sheets are covered with a denser molten layer. The layer is enhanced by viscous flow densifying the amorphous phase structure [17]. While porous appearance occurs, the surface of the steel is isolated to contact with oxygen. The crystalline phase forms and enhances within glass powder melting, as high temperatures continue to rise. The composition of the amorphous phase starts to reduce and the molten film changes to ceramic film [17]. The external diffusion of an iron element is impeded by the crystalline phase. This case presents in Figures 5(c-h). It is considered that porous oxide layers and cracking on the surface of the Cr steel sheet resulted from significant degradation of steel (Figure 5(g and h)) [18].

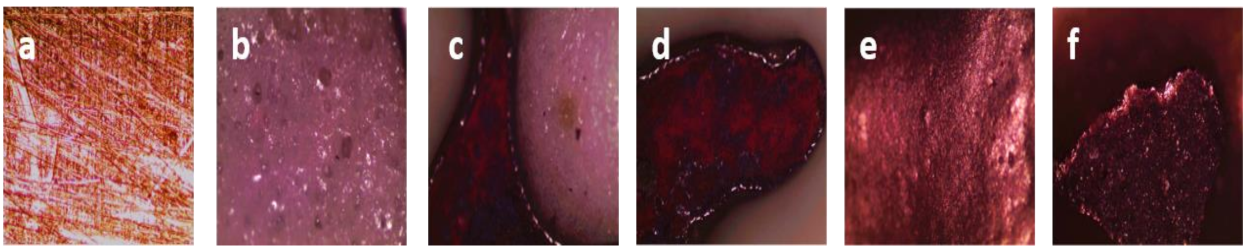

Figure 4. OM images of non-coated Cr steel sheet (a) and Cr steel coated with dehydrated glass powder at 950 °C temperature for 1 hour (b), 2 hours (c, d) and 3 hours (e, f) with a 50X magnification

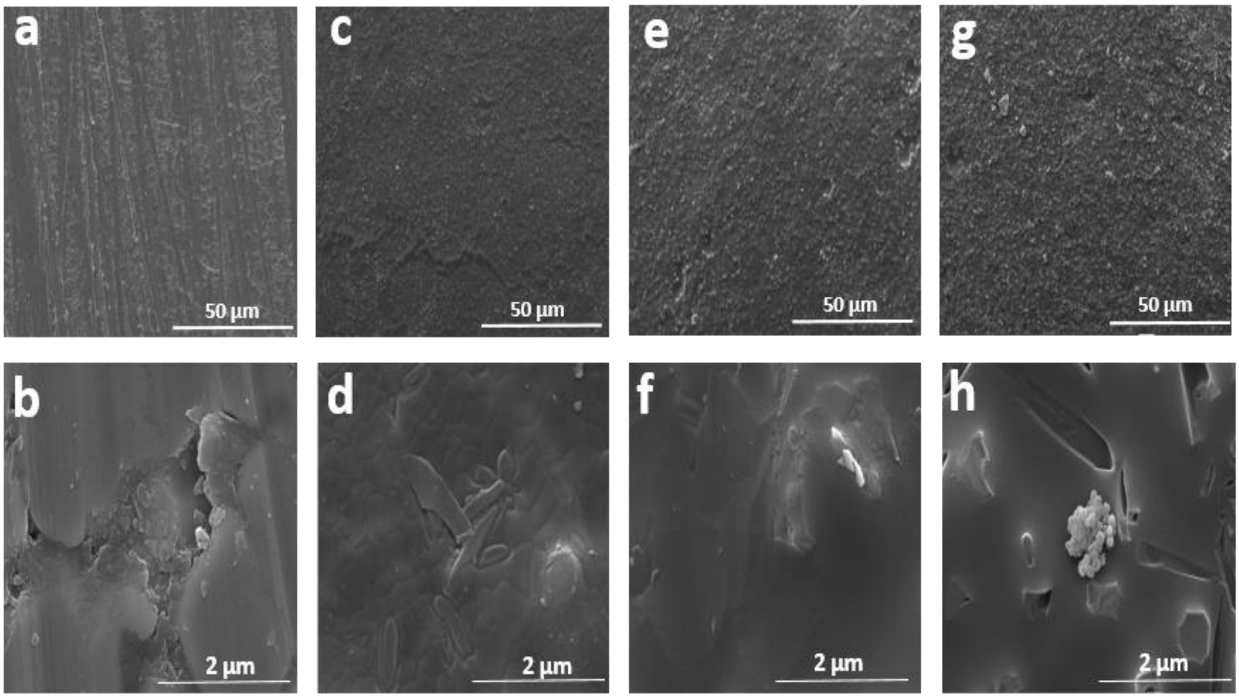

Figure 5: The SEM images of non-coated Cr steel sheet (a, b) and Cr steel sheet coated with dehydrated glass powder at 950 °C temperature for 1 hour (c, d), 2 hours (e, f) and 3 hours (g, h)

Table 3 shows elemental analysis with EDS for Cr steel sheet coated with dehydrated glass powder at 950°C temperature for 1, 2, and 3 hours. When the waiting time in the furnace is increased, the detention effect of the covering of external diffusion of Fe ions forms [10]. Silicon, sodium, and calcium are detected in EDS spectrums. These elements are the most plenty of chemical components in the glass powder. Hence, the effects of these elements on the surfaces of steel may be recognizable [19].

Figure 6 presents the XRD Pattern of the non-coated Cr steel sheet. On the pattern of this sheet, Fe γ (FCC (Face centered cubic) crystal structure) peaks were recorded on the 43.46°, 50.60°, and 74.54° degrees and Cr (BCC (body centered cubic) crystal structure) peaks were recorded at 44.38°, 64.48°, 81.83° degrees. With the heat treatment, the glass powder starts to melt at almost 650°C and crystalizes at around 800-900°C temperatures, and nucleates at approximately 950°C temperature [12, 14].

Table 3. The EDS spectrums of Cr steel sheet coated with dehydrated glass powder at 950 °C temperature for 1 ,2, and 3 hours.

	1 h		**2 h**		**3 h**	
Element	**Weight (%wt)**	**Atomic (%)**	**Weight (%wt)**	**Atomic (%)**	**Weight (%wt)**	**Atomic (%)**
O	34.36	48.67	34.83	48.83	32.05	45.79
Na	2.29	2.26	9.52	9.29	8.77	8.73
Mg	2.39	2.23	4.85	4.47	5.5	5.17
Al	0.41	0.34	1.01	0.84	1.02	0.86
Si	51.23	41.33	37.7	30.1	39.67	32.29
Ca	8.63	4.88	9.71	5.43	11.07	6.31
Cr	0.69	0.3	2.37	1.02	1.93	0.85

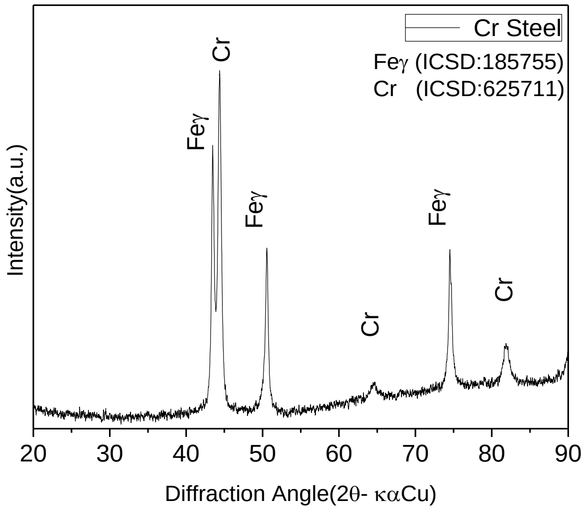

Figure 6. The XRD Pattern of non-coated Cr steel sheet.

Figure 7 shows XRD patterns of Cr steel sheets coated with glass powder at 950 °C temperature for 1, 2, and 3 hours. The surface of all coated steel sheets shows that the structures have corundum (Al_2O_3) having a hexagonal crystal structure (ICSD: 89662), calcium oxide having wurtzite type and hexagonal crystal structure (ICSD:161831), chromium oxide (Cr_2O_3) having an orthorhombic crystal structure (ICSD: 29130), magnetite (Fe_3O_4) having spinel-type and cubic crystal structure (ICSD:183977), magnesium oxide having a cubic crystal structure(ICSD:157526), sodium(Na) having a cubic crystal structure (ICSD:53753) and cristobalite (SiO_2) having a tetragonal crystal structure (ICSD: 153886), and diopside ($CaMgO_6Si_2$) having a monoclinic crystal structure (ICSD: 1223). The formation of the cristobalite phase is related to reaction enthalpy. This reaction enthalpy is represented by the endothermic reaction which is changed the absorbed heat in form of thermal energy to kinetic energy to form ionic diffusion in the materials. Hence, when the temperature increases, crystalline phases form [1]. Small-sized crystals of diopside phases are embedded in a glassy matrix [20].

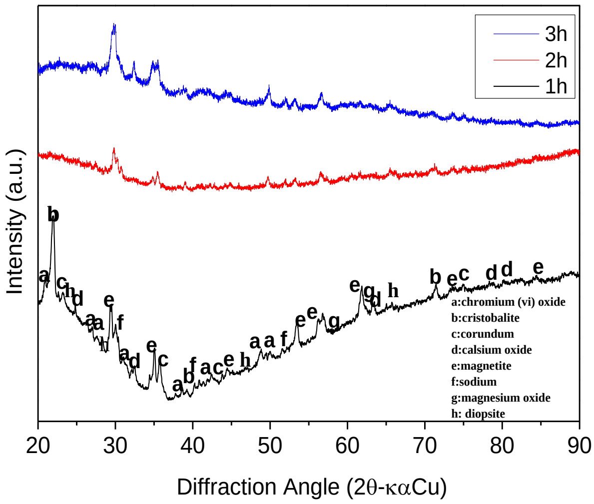

Figure 7. The XRD Patterns of Cr steel sheet coated with dehydrated glass powder at 950 °C temperature for 1 ,2, and 3 hours.

Magnetite peaks have a high degree of crystallinity showed with sharp peaks on the Cr steel sheets magnetite phase possibly crystallized throughout cooling to room temperature from melting temperature, and succeeding heat treatment for crystallization was not sufficient [21]. A higher amount of calcium, sodium, magnesium, silicon oxides is observed in the melted glass powder. These oxides can avoid crystallizing of magnetite via the formation of solid solutions by iron oxides [21]. Increasing the annealing time from 1 h to 3 h resulted in disappear some phases in the structure. This is because a long time led to form single grains and crystal structures so that there is a reduction occurred metastable phases in the mixture. Here, we observed dominant magnetite, chromium oxide, sodium, and corundum phases.

4. Conclusion

Cr sheet metal coated with dehydrated glass powder by heat treating at 950°C temperature for 1, 2, and 3 hours and cooled to room temperature in the furnace. The following results are given;

1. Glass powder melting retention occurred on the surfaces of all Cr sheet metal. Glass powder on the surfaces of Cr steel sheets has fully not melted homogeneously and waved appearances on the surfaces of these steel sheets have been obtained.
2. With increasing temperature and waiting time, surfaces of Cr steel sheet metals coated with glass powder had become porous.
3. Crystoballite, diopside, corundum, magnetite phases, and magnesium and calcium oxides on the surfaces of all coated Cr steel sheets were determined. The higher amounts of calcium, sodium, magnesium and silicon oxides are observed in the melted glass powder.

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