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Thin Siliconized Films For Protection Of Carbon Steel Alloys

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ABSTRACT

Hexa-cyclohexoxy-disiloxane (HCHODSO) was synthesized and its purity was confirmed by FT-IR invistigation. The specimens of carbon steel alloy type B grad 39 were considered for the present work. The specimen surfaces were prepared by different grade of emery paper and painted by airless method with HCHODSO. The painting specimens were thermally treated at 550,575,600,625,650 and 675°C for 25 minutes. The physico mechanical, structure, electrical and thermal properties of various specimens were investigated using different techniques, which include SEM, XRD, EDX, USV, polarized micrography and microhardness analyzer. The results revaled that the thin siliconized film thickness measured 4±1 μm and the optimum condition was reached by heating the steel specimens covered by siliconized film at 625°C.

KEYWORDS

HCHODSO;
SEM;
XRD;
EDX;
USV;
Polarized micrography;
Microhardness analyzer.

INTRODUCTION

The carbon steel alloy equipments used in petroleum, petrochemical and other heavy duty industries are subjected to corrosion environments. These equipments are corroded and quickly damage. These problems are affected economically on the production [1-11]. The organosilicon compounds are used for combat the surface of petroleum equipments from corrosion phenomena and lowering the cost[3-23]. One of these methods are depended on the pyrolysis of organosilicon compounds at high temperature on the surface of equipment [1-7,10-16], and the other are depended on the curing cases[8-18]. The mechanism of

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the formed films are based on the interaction of two process; metal in the form of organosilicon compound coatings was brought into contact with the surface of carbon steel, followed by gradual diffusion of silicon into the lattice of carbon steel at high temperature[1-23].

In the present work, the capability of HCHODSO to produce stable films on the surface of carbon steel alloy specimens at temperatures 550, 575, 600, 625, 650, and 675 ºC are investigated. This is to achieve optimum coat that can be formed at net concentration of HCHODSO. The suitability of the formed films as anti – corrosion coating could be judged by physical properties, visual inspection, dry film thickness (DFT), scanning electron microscope (SEM), ultrasonic vibration (USV), hardness, X- Ray diffraction (XRD) as well as light and polarized optical microscope.

EXPERIMENTAL

Material

Tetrachlorosilane, cyclohexanol, diethyl ether, benzene, xylene, DMF and N, N- dimethylaniline all analar grade and were supplied by Aldrich Co. unused carbon steel alloy specimens type BG 39 used in petroleum equipments were taken for the present study.

Preparation of HCHODSO

The HCHODSO was prepared according to Clark Krishnasamy[24] reaction. The product structure was confirmed by FT-IR and refractive index investigation.

Techniques

Infra-red analysis (IR)

The IR spectra of the prepared HCHODSO were measured by using FT-IR spectrometer model 1650 Perkin Elmer. The wave numbers of the IR bands of different functional groups were determined in the range of 4000- 600 cm^{-1}.

Refractive index measurement

The refractive index data was carried out by Abbe refractometer model carlzeiss JEAN. The measurements were recorded under ambient pressure and temperature.

Preparation the surface of carbon steel alloy specimens with coatings[26]

Specimens of carbon steel alloy were cut as regular edged cuboids with dimensions 3.0 × 1.0 × 0.9 cm. Each specimen was cleaned, polished with 600, 500,400,320&200 grades emery paper, rinsed with water, degreased with ethyl alcohol/acetone, weighted and finally stored under vacuum after wrapping it with adhesive thin paper.

Each specimen was coated with net concentration of synthesized HCHODSO. The coated specimens were then thermally treated at temperatures 550, 575, 600, 625, 650 and 675°C, for 25 minutes under atmospheric pressure.

Analysis techniques

Visual inspection

The visual inspection for all physical properties of the formed films was carried out to investigate the effect of temperature on the coating by HCHODSO compound.

Ultrasonic vibration technique(USV)[27]

The degree of coherence of the films at the surface of the specimens was determined by applying the USV technique using an ultrasonic cleaner model Astrason 7E.

Physical and mechanical properties

Wet film thickness (WFT), dry film thickness (DFT), hardness, adhesion, burnishing, thermal stability, scribtor, electrical conductivity and peeling tests were carried out according to the standard methods[27-34].

Scanning electron microscope (SEM)

SEM model JEOL-T2000 linked with simultaneous multi-element analyzer 860/500 dual disc Energy Dispersive System (EDS) was used to study the morphology and structure of the formed HCHODSO films. The morphological micrographs were exposure by a camera model JEOL-T20-CSI at magnification depending on the purpose of investigation. EDS spectra were analyzed to evaluate the relative concentration of the deposited silicon on

the specimens surface.

X-ray diffraction (XRD)

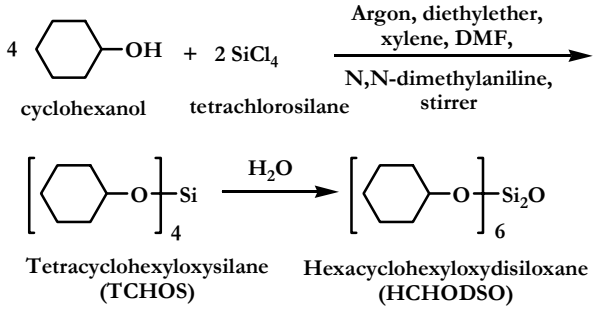
The XRD was used to identify the phases of silicon, which have been formed after thermal treatment, and their ratios as well as the microstructure of the formed phases. X-Ray diffractometer Philips 1976 model 1390 was used to produce a diffractive pattern for the optimium formed film at 625ºC. The pattern was analyzed by applying finks procedure using the most- three d's values and scherrer formula[36-37].

Polarized microscope

The polarized microscope model Versament, type 2, Union 7609 connected by camera model Nilsson FX-35 was used to measure the orientation of the formed grains and to determine the color interaction of formed phases with each other for the formed film at 625ºC. The coated specimen is etched in concentrated HNO_3 / ethanol (1:1 v/v) mixture.

RESULT AND DISCUSSION

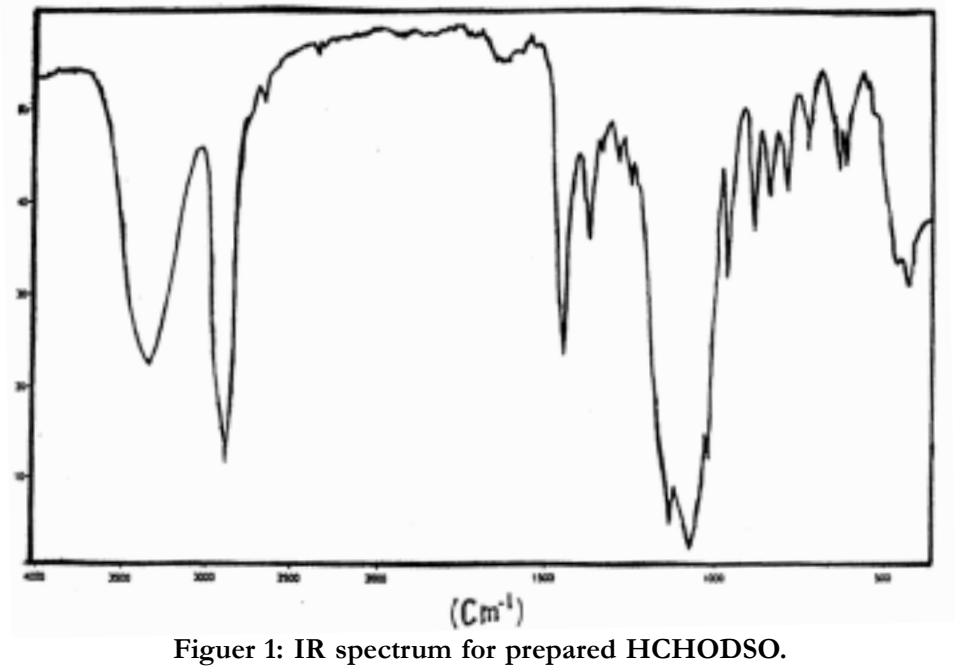
The product compound of HCHODSO is obtained by the reaction of cyclohexanol with tetrachlorosilane under specific conditions according to the following equation:



The HCHODSO compound is purified by fractional distillation and confirmed by FT- IR and RI.

Figure (1) illustrates the characteristics bands of IR spectra for the structure of HCHODSO. The band 3417 cm^{-1} is characteristic the stretching vibration of Si-O-Si bond & band at 2956 and 1406 cm^{-1} for CH_2 aliphatic. The bands 1097 and 1161cm^{-1} and bands 997,839,799,753 and 643 cm^{-1} are characteristic the stretching vibration of Si-O & C-H linkage.

TABLE 1 illustrates the physical properties of the formed films from pyrolytic decomposition of HCHODSO on the surface of carbon steel alloy at 550, 575, 600, 625, 650 and 675ºC for 25 minutes. Moreover, the normalized weights of the formed films per unit area are not affected by the difference in pyrolytic temperature. The visual inspection and



Figuer 1: IR spectrum for prepared HCHODSO.

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TABLE 1: The physical measurement of the formed films from hchodso before and after pyrolytic decomposition at temperature ranging from 550 to 675 ºC & 25 minutes.

Exposure Temp. (ºC)	Wt. of metal specimen (g)	One surface area of specimen (cm^2)	Wt. of specimen after coated with HCHODSO (g)	Wt. of HCHODSO (g)	Normalized wt. of HCHODSO per unit area (WFT) (mg/cm^2)	Wt. of the formed films (g)	Normalized wt. of formed dry films per unit area (WFT) (mg/cm^2)
550	3.8971	2.6289	3.9118	0.0147	5.5917	0.0019	0.7227
575	3.8557	2.6005	3.8708	0.0141	5.4220	0.0017	0.6537
600	3.9073	2.7713	3.9222	0.0149	5.3231	0.0021	0.7578
625	3.8615	2.5991	3.8854	0.0139	5.3401	0.0017	0.6541
650	3.8895	2.6137	3.9038	0.0143	5.4712	0.0018	0.6887
675	3.8779	2.6179	3.8920	0.0141	5.3860	0.0019	0.7258

TABLE 2: Visual inspection of the formed siliconized films from pyrolytic decomposition of hchodso on the surface of specimens at 550 to 675 ºc & 25 minutes.

Pyrolytic temp (ºC)	% of silicon	Visual description								
		Color of the formed films	Lamination on the surface film	Bare Area	Adhesion	Dullness	Staining	Splashing	Electrical Insulation	Coherence
550	8.4	Top layer pale pink inner layer pal violet (glassy)	Apparently one layer thick (homogeneity)	No bare area appeared	The top layer is removed	Harden & lastering glssy	Nothing	few splashed pieces appear on the film surface	High electrical insulating above 24v	The formed films high compact & coherency
575					The formed film compact with specimen surface			"		
600								Very few splashed pieces appear on the surface		
625										
650										
675					"					

the studying of the physico-mechanical properties of the formed dry films are given in TABLES (2-5). These included the color, measuring and calculating the WFT & DFT for the formed wet and dry films, lamination, bare area, adhesion, dull-ness, staining, splashing electrical conductivity, coherence, pull, scribtor, burnishing, peel, thermal cycling tests and micro hardness.

The physical and mechanical properties of the formed dry films TABLES 1-6 are dependent on the pyrolytic decomposition temperature. Moreover the most efficient properties are clear at 625ºC, which is advanced by applying the USV technique, where the properties of the formed dry film at 625ºC are not affected.

SEM analysis of the formed siliconized films

The surface morphologies and cross-section SEM for the formed siliconized films by pyrolytic decomposition of HCHODSO at 550, 575,600, 625, 650 and 675ºC are visually examined figures (2a,b –7a,b). The texture morphologies are indicated the orienta-

TABLE 3: Physical properties adhesion of the formed siliconized films on the surface of specimens at 550 to 765 ºC & 25 minutes.

Temp. (ºC)	Si %	Adhesion Testing				
		Burnishing test BS 6670-86	Pull of test qualitative	Scribetor test BS 4292	Pell test Bs 6670-86	Thermal cycling test Bs 6670-86
550		Partially blisters in limited regime		Failure the surface layer	Peeling 1-3 square	Partially cracking and blistering
575				,,	,,	
600		Partially blisters in limited regime	No failure at the formed films substrate interface	No failure within the scribing squares	No – peel value is recorded	Partially cracking and blistering
525	8.4	Partially blisters in limited regime				low cracking and blistering
650						low cracking and blistering
675		Yellow stain on the rupping tissue paper on the top layer only				No visible defects
						No visible defects

TABLE 4: Microhardness measurements of the formed siliconized films by vecker method of hchodso at on the surface of specimens 550 to 675 ºC & 25 minutes.

Pyrolytic temp. (ºC)	Hardness measurements							
	Before etching				After etching			
	V.H.NO.	Average of V.H.NO.	D.aveage (μm)	V.depth (μm)	V.H.NO.	Average of V.H.NO.	D.aveage (μm)	V. Depth (μm)
550	1- 211.0 2- 211.5 3- 209.5	210.67	67.2	517	191.0 190.5 191.5	191.0	63.13	9.81
575	1- 211.5 2- 209.0 3- 212.0	210.83	67.71	933	189.5 190.5 190.6	190.17	66.57	9.85
600	1- 209.0 2- 211.0 3- 209.0	209.67	66.90	79	189.5 190.5 191.0	190.33	66.93	9.75
625	1- 211.0 2- 209.0 3- 212.0	210.67	67.27	99	193.5 192.5 192.5	192.67	67.01	9.97
650	1- 211.5 2- 213.0 3- 211.0	211.83	67.39	9.979	189.5 190.0 190.5	190.0	65.5	9.77
675	1- 210.5 2- 211.0 3- 209.5	310.33	67.33	9.97	189.5 190.0 189.5	189.67	67.15	9.83

tion, boundaries, shape, segregation and striation for each film separately. The cross- section SEM is indicated the regularities, homogeneity splashing, voids and compatibility of the formed siliconized films with surface of substrata.

Figure (2a) is representing the texture morphologies of the formed siliconized films at 550ºC. The results reveal that the grains boundaries are not appeared; the film is showed as past and more than one layer. This indicates that the formed siliconized film is laminated i.e. not sufficient. These phenomena are clear in cross-section SEM figure (2b). The micrographs have voids and lamination area between the surface of substrate and the formed siliconized film.

Figures (3a and 4a) representing the texture morphologies of the formed siliconized film at 575 & 600ºC respectively. These films are showed as con-

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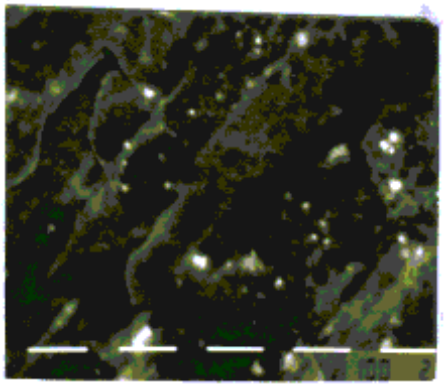

Figure 2a: SEM micrograph of the formed siliconized film at 550°C & 50X.

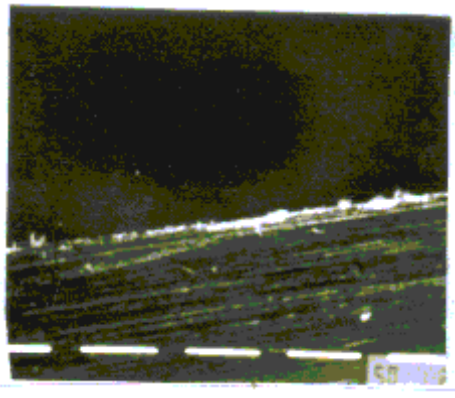

Figure 2b: Cross-selection SEM micrograph of the formed siliconized film at 550°C & 50X.

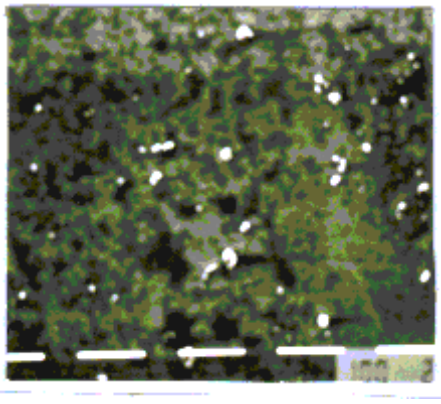

Figure 3a: SEM micrograph of the formed siliconized film at 575°C & 50X.

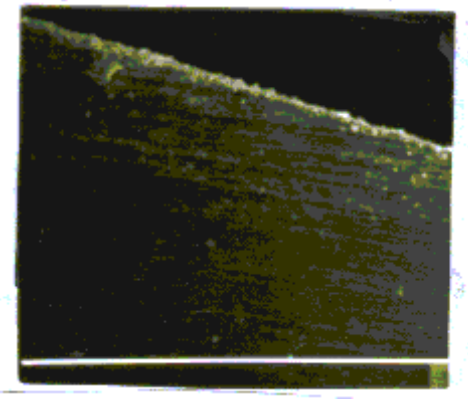

Figure 3b: Cross-section SEM micrograph of the formed siliconized film at 575°C & 150X.

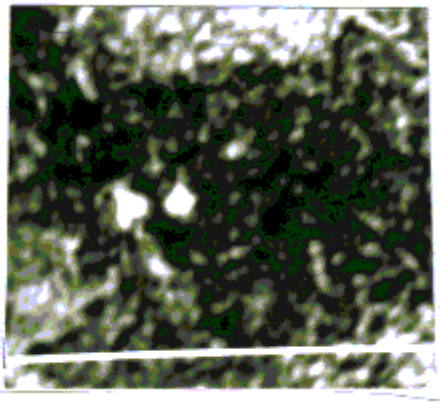

Figure 4a: SEM micrograph of the formed siliconized film at 600°C & 150X.

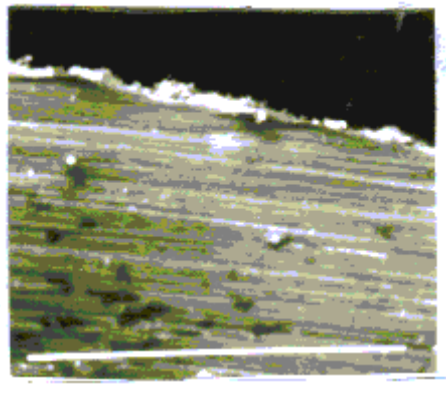

Figure 4b: Cross-section SEM micrograph of the formed siliconized film at 600°C & 120X.

densate white spots. These spots are not connected with each other aggomelation at some area. These phenomena are indicated that the formed films at 575 & 600°C are not sufficient.

The cross-section figures (3b and 4b) have some voids aggomelation of white spots, these phenomena are indicated that the cross-section of the formed siliconized films are not homogenty and compact with the surface of substrate.

Figure (5a) shows the surface morphologies SEM micrograph of the formed siliconized film at pyrolytic temperature 625°C. This shows a crystalline

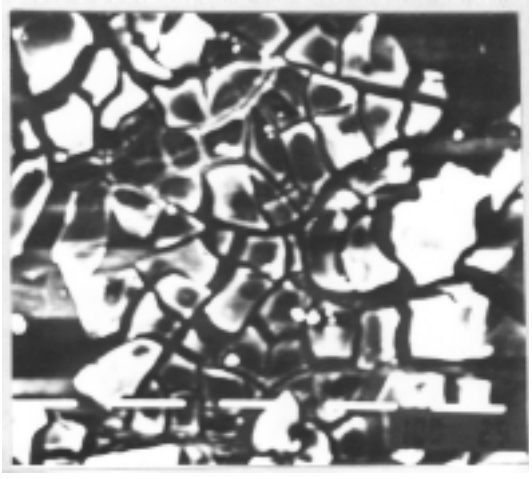

Figure 5a: SEM micrograph of the formed siliconized film at 625°C & 50 X.

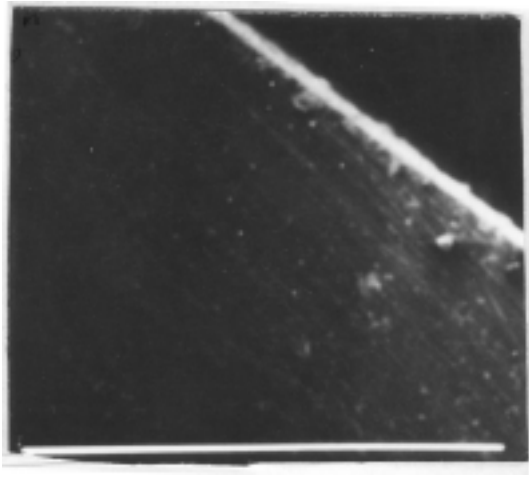

Figure 5b: Cross-section SEM micrograph of the formed siliconized film at 625°C & 120 X.

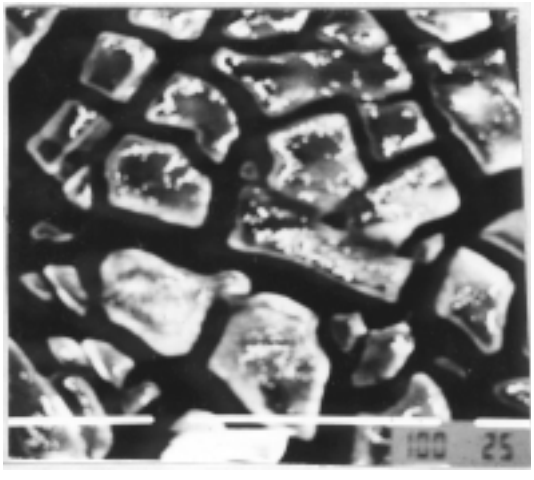

Figure 6a: SEM micrograph of the formed siliconized film at 650°C & 100 X.

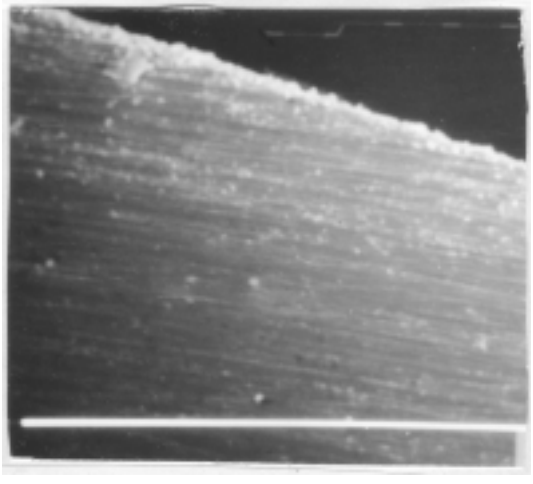

Figure 6b: Cross-section SEM micrograph of the formed siliconized film at 650°C & 150 X.

Figure 7a: SEM micrograph of the formed siliconized film at 675°C & 150 X.

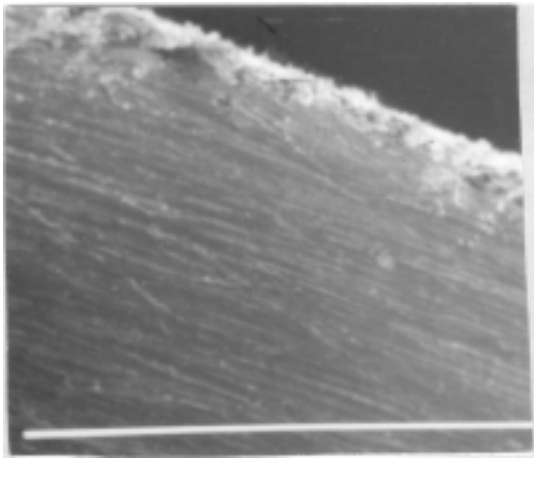

Figure 7b: Cross-section SEM micrograph of the formed siliconized film at 675°C & 150 X.

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TABLE 5: Measured and calculated the dft of the formed siliconized films by pyrolytic decomposition of HCHODSO at temperature 550 to675 °C & 20 minutes by using elecometer 300 FB brosh.

Operating parameters		Measured practical dry film thickness (DFT) (μm)	Calculated dry film thickness (DFT) Mg/Cm²
% of Si	Exposure Temperature °C		
8.4	550	3.7517	0.7227
	575	3.8991	0.6537
	600	4.0987	0.7578
	625	4.0073	0.6541
	650	3.8193	0.0887
	675	4.2105	0.7258

shape, orientation, equidistrbution of grains boundaries, striation, and segregation. Figure(5b) represents the cross-section SEM of the formed siliconized film at 625°C, which is free from any voids on films/metal interface and the splashing phenomena, is not observed. i.e the formed films is sufficient.

Figures (6a, 7a, 6b and 7b) represent the surface morphologies of the formed siliconized film and cross-section SEM, at 650 & 675°C respectively.

The grain boundaries are bigger, striation, segregation phenomena increased by raising the pyrolytic decomposition. The cross- section SEM shows that the formed film is non- homogeneous, not continuous and has splashing phenomena.

These results indicate that the composition of the formed siliconized films are influenced by pyrolytic temperature, i.e. crystalline shape, crystallinity distribution symmetry, which are more advanced at 625°C. This is the optimized temperature of pyrolytic decomposition of HCHODSO on the surface of carbon steel alloy specimen to form the best siliconized film.

EDS system

Figure (8) represents the EDS diagram of the formed siliconized film at pyrolytic decomposition of HCHODSO at optimized temperature 625°C on the surface of substrata.

TABLE 6: The microstructure of the formed siliconized films on the surface of specimen at optimized temperature 625°C.

No.	Mineralogical name	Chemical formula	Specific most four's value (A°)				Reference of inorganic card set	Crystalline phase percentages at optimized temperature 650°C	Microstructure analysis		
									Preferred orientation	Particle size (A°)	Crystallinity order
A	Amorphous	SiO_2	-	-	-	-	-	54.90	--	38.31	Smicrystalline
B	Faylite	Fe_2SiO_4 Iron silicate	2.5	2.83	1.78	3.55	4 – 484	27.83	<222>	391.13	Orthorhombic
C	Wustite	FeO Iron oxide Ferrous oxide	2.48	2.15	2.49	1.52	6 – 615	6.91	<220>	422.71	Cubic
D	Maghemite	Fe_2O_3 Ferric oxide	2.5	1.48	2.95	1.61	4 – 755	0.21	<110>	241.63	Cubic
E	Ferisillicite	FeSi Iron silicide	2.0	1.82	1.19	3.16	1 – 1271	6.95	<321>	607.46	Cubic
F	α-cystballite	SiO_2 Silicon dioxide	4.05	2.49	2.84	4.05	4 – 379	0.027	<101>	407.11	Tetragonal
G	β-cystballite	SiO_2 Silicon dioxide	4.15	2.53	1.64	4.15	4 – 359	3.12	<220>	626.78	Cubic
H	Tridymite	SiO_2 Silicon dioxide	4.3	4.08	3.81	4.3	3 - 227	0.05	<442>	535.95	Orthorhombic

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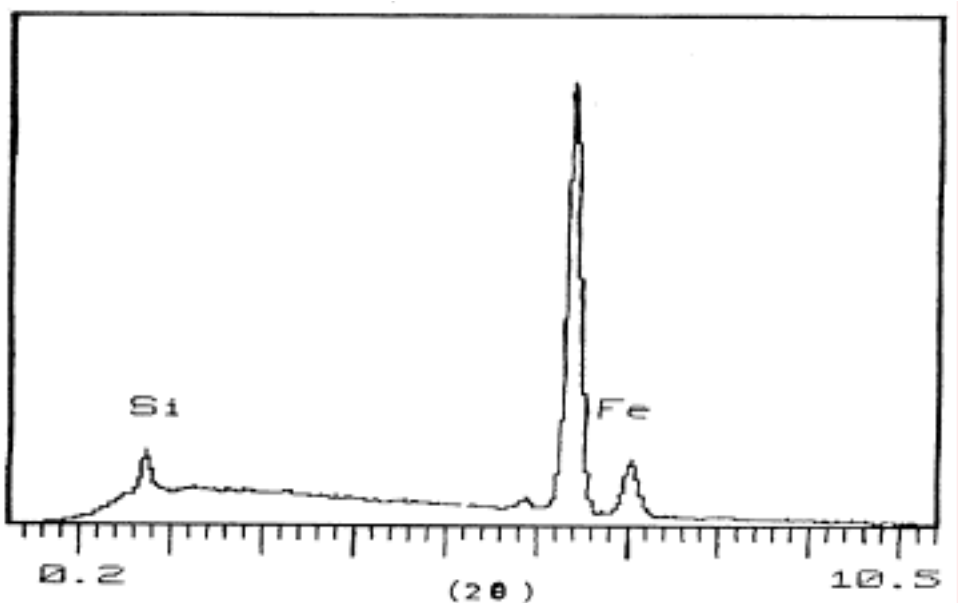

Figure 8: EDS diagram analysis of the formed siliconized film by pyrolytic decompotion of HCHODSO at 625°C.

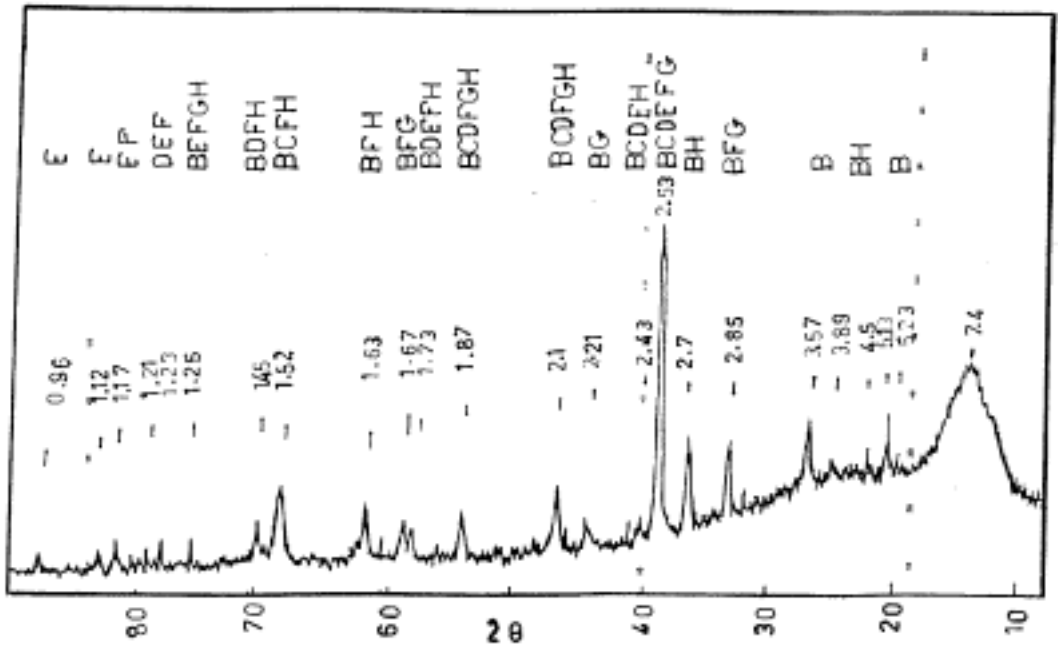

Figure 9: Typical XRD diagram of the formed siliconized film of pyrolytic decomposition of HCHODSO at 625°C

X-Ray diffraction

The formation of siliconized film at 625°C is led to more distribution and crystalline, was examined by XRD. The pattern of XRD, figure (9), analyzed and detects the formed phases (amorphous/crystalline) by Fink's procedure as mentioned in Cullity[36].

A quantitative sense, average size and orientation of crystaline or semi-crystalline grains of formed phase at 625°C are calculated by using XRD pattern

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Figure 10a: YMD SEM micrograph of the formed siliconized film at 625°C & 75 X represent growthing of the silicon phases.

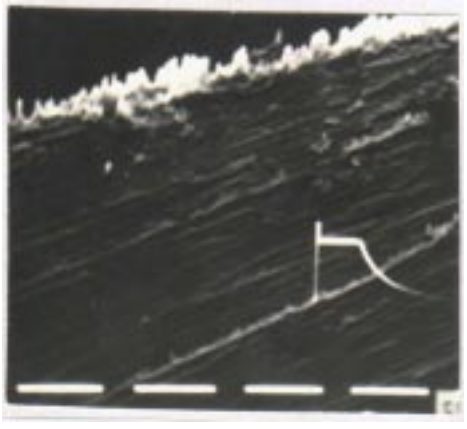

Figure 10b: Cross-section YMD SEM micrograph of the formed siliconized film at 625°C & 50 X represent growthing of the silicon phases.

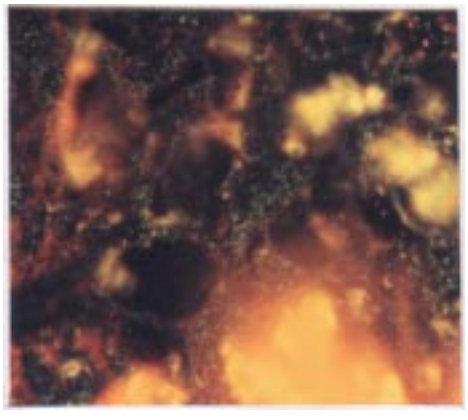

Figure 11a: Polarized bright view the microstructure of the formed siliconized film at 625°C & 350 X.

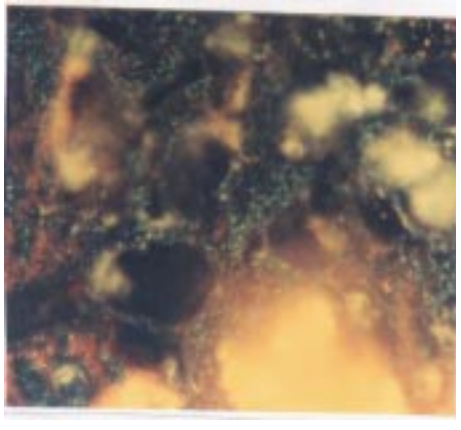

Figure 11b: Polarized light view the microstructure of the formed siliconized film at 625°C & 350 X.

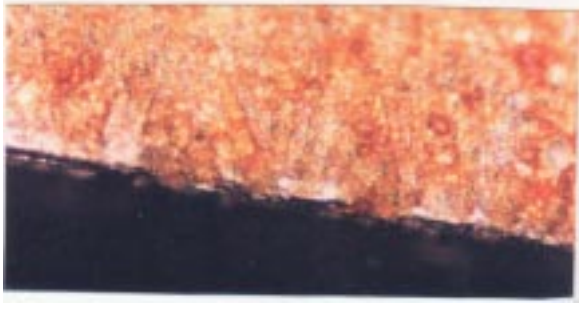

Figure 12: Microstructure cross-section view of the formed siliconized film at 625°C by bright polarized microscope at; a) the surface and b) the white spots at the hardly scratched and polished area underneath indicates the rooting phenomena.

and recorded in TABLE (6). The crystalline size is determined from line width by applying Scherre formula as mentioned by Azaroff [37] and stander inorganic cards.

The formed phases by pyroltic decomposition of HCHODSO at 625°C on the surface of carbon steel alloy specimen are recorded in TABLE (6). The peak at 2 θ ranging between 8° and 18° characterizes the amorphous SiO_2 phase. From the microcrystalline studies, the amorphous SiO_2 is intended to be semi crystalline (psedoamorphous), while the specific peaks for crystalline phases are spotted at 2 θ ranging between 20° and 96° , which are equivalent to 5.23A° - 0.83 A° respectively.

Figure (10a and b) are indicated the YMD dia-

gram for surface morphology and cross-section of the formed siliconized film at 625ºC, which are illustrated the growthing of silicon phases internal the surface of specimens and the formed siliconized film as rooting phenomena.

Polarized morphology

Figures (11a and b) are represented the polarized & light photographic of the interference color, granular and shaplles distribution of the form siliconized film at 625ºC and magnified at 400 x for the view surface.

Fox absorption colors, shaplles and orientation crystals consist mainly of pale yellowish–brown interface. That is the first order interference color for the granular textured micrograph. The pale yellow color at the edges of grains is converted into grainsh at the center, which is due to faylite mixed with Wustite. Also the faylite phase shows only a pale green absorption color. This was supported by SEM and XRD techniques. The gaps between the well boundaries aggregated grains almost entirely filled with fine grained fayalite and/or the basic fayalite layer is partially covered with aggregated grains from maghemite and/or dispersed with (Crystabolite and Tridymite).

Figure (12) is indicated the diffusion of silicon phases as Ferisilicite phase (FeSi) internal the surface lattice of substrata. This case is called the rooting phenomena.

CONCLUSION

The physical properties of the formed siliconized films by pyrolytic decomposition of HCHODSO on the surface of carbon steel alloy at 550, 575, 600, 625, 650 and 675ºC. The visual examination of the surface texture morphology of micrographs of the formed siliconized films one can conclude the crystallinity, orientation, crystalline order, shape, stration, segregation and distribution.

The cross-section SEM micrographs showed the thickness, homogeneity, splashing, voids and compatibility of the formed siliconized film with the surface of substrata.

The USV technique indicates the stability of the formed siliconized films.

From the all physical measurements, SEM, cross-section SEM, and USV techniques, one can conclude that the optimized siliconized film is formed at 625ºC.

The YMD mode of texture morphology indicated the growthing of the silicon phases internal the form siliconized film and the surface of substrata as rooting phenomena.

The XRD detects the amorphous / crystallinity phases and micro crystalline structure of the formed siliconized film at 625ºC according Scherrer formula.

The EDS analysis indicates the percentage of silicon in the formed siliconized film with respect to substrata.

The polarized micrographs indicate the microstructure crystalline phases color interference reaction between the formed phases and rooting phenomena.

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