PACK CEMENTATION OF HIGH CHROMIUM STEEL FOR HIGH TEMPERATURE APPLICATION IN STEAM CONDITION

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Pengoksidaan keluli kromium (Cr) yang tinggi dalam persekitaran wap boleh menyebabkan ketahanan lapisan kromia (Cr_2O_3) terbantut dan tersejat yang mana akan mengurangkan ketahanan karat bahan dandang. Keadaan ini boleh menyebabkan pengurangan jangka hayat dandang atau kegagalan sewaktu beroperasi. Oleh itu, usaha untuk mengekalkan pembentukan lapisan kromia dalam persekitaran wap ini adalah penting supaya ia boleh digunakan untuk masa yang lebih lama dan pada suhu operasi yang lebih tinggi. Dalam kajian ini, pek penyimenan Cr, yang juga dikenali sebagai proses kromizing, diperkenalkan untuk menyerap Cr ke permukaan sampel keluli T91. Serapan Cr dijangka bertindak sebagai takungan untuk pembentukan lapisan pelindung Cr_2O_3 apabila keluli terdedah kepada pengoksidaan wap suhu tinggi. Sampel keluli diletakkan dalam campuran alumina yang mengandungi campuran paket serbuk Cr sebagai masteralloy, serbuk NH_4Cl sebagai pengaktif, dan serbuk Al_2O_3 sebagai pengisi lengai. Bekas alumina kemudian dipanaskan di dalam relau tiub ke suhu yang ditetapkan untuk proses pek penyimenan. Persekitaran di dalam relau tiub disimpan dengan gas argon lengai yang tulen dialirkan dengan kelajuan 150 mL / min. Parameter yang dikaji dalam kajian ini ialah suhu penyimenan pek pada (600°C, 800°C dan 1050°C) dan komposisi campuran pek pada (20wt.% Cr, 40wt.% Cr dan 60wt.% Cr). Masa untuk proses pek penyimenan ditetapkan selama dua jam. Untuk kesan suhu pek penyimenan, campuran pek adalah malar pada : 48wt.% Cr – 4wt.% NH_4Cl – 48wt.% Al_2O_3. Field emission scanning electron magnetomenter/ energy dispersive X-ray (FESEM / EDX) menunjukkan bahawa Cr mula meresap ke dalam keluli dari 800°C. Cr-karbida dan Cr-nitrida juga terbentuk pada permukaan keluli yang mana kewujudannya disahkan oleh hasil analisa pembelauan sinar X. Pembentukan Cr-karbida disebabkan oleh serapan keluar karbon dari keluli, dan Cr-nitrida juga boleh dibentuk dari serapan keluar N dan juga dari penguraian NH_4Cl. Secara keseluruhannya, pek penyimenan pada 1050°C telah menunjukkan lapisan serapan yang paling homogen dan berterusan berbanding dengan 600°C dan 800°C dengan kedalaman Cr sekitar 40 μm. Dari eksperimen komposisi yang berbeza, tiga komposisi campuran pek yang berbeza dan kandungan pengaktif yang tinggi (20wt.% NH_4Cl) dilakukan pada suhu 1050°C. Dari analisis FESEM / EDX, didapati kepekatan Cr dan NH_4Cl yang tinggi menghasilkan kedalaman Cr yang lebih tinggi. 60wt.% Cr menghasilkan kedalaman Cr sekitar 64 μm dan juga lebih homogen berbanding komposisi lain. Cr-karbida dan Cr-nitrida juga dikesan di dalam dalam kesemua sampel. Pengoksidaan juga telah dilakukan pada 700ºC selama 90 jam di bawah keadaan wap. Telah didapati bahawa Cr_2O_3 masih wujud pada keluli dan kedalaman Cr tidak berubah dengan ketara selepas proses pek penyimenan. Keputusan ini menunjukkan pek penyimenan mampu menjadi takungan kepada pembentukan Cr_2O_3 dalam pengoksidaan wap suhu tinggi, dan kewujudannya boleh digunakan untuk masa pengoksidaan yang lebih lama.

ABSTRACT

The oxidation of high Chromium (Cr) steels in steam environment may cause retardation or evaporation of protective chromia (Cr_2O_3) layer, which reduces its corrosion resistance as boiler materials. This condition may cause reduction of boiler life time or failure during operation. Therefore, the effort to maintain the formation of Cr_2O_3 layer in this steam environment is crucial so that it can be used for longer operating time and at a higher operating temperature. In this research, pack cementation of Cr, also known as chromizing process, was introduced to diffuse Cr into the surface of T91 steel sample. The diffused Cr was expected to act as reservoir for the formation of Cr_2O_3 protective layer when the steel was exposed to high temperature steam oxidation. The steel sample was embedded inside an alumina crucible that contained pack mixtures of Cr powder as a masteralloy, NH_4Cl powder as an activator, and Al_2O_3 powder as an inert filler. The alumina crucible then was heated inside tube furnace to the expected temperature for the pack cementation process. The environment inside the tube furnace was kept inert by flowing pure argon gas at 150 mL/min. The parameters being observed in this research were temperature of pack cementation (600^o-1050^oC) and composition of pack mixture (20wt.% Cr - 60wt.% Cr). The time for pack cementation process was kept constant at two hours. In the effect of pack cementation temperature, the composition of pack mixture was constant at 48wt.% Cr – 4wt.% NH_4Cl – 48wt.% Al_2O_3. Field emission scanning electron/Energy dispersive Xray (FESEM/EDX) analysis showed that Cr started to diffuse into the steel from 800^oC. Cr-carbide and Cr-nitride also formed on the surface of the steel, in which its existence was confirmed by Xray diffraction (XRD) result. The formation of Cr-carbide was due to outward diffusion of carbon from the steel, and the Cr-nitride may also be formed from the outward diffusion of N and also from the decomposition of NH_4Cl. Overall, the pack cementation heated at 1050^oC has showed the most homogeneous and continuous diffusion layer as compared to heating at 600^oC and 800^oC, with Cr-depth about 40 μm. From the different composition experiment, three compositions of pack mixture with different Cr concentration and higher activator content (20wt. % NH_4Cl) were observed at 1050^oC. From FESEM/EDX analysis, it was found that higher concentration of Cr and NH_4Cl produce higher Cr-depth. The 60wt.% Cr produced Cr-depth around 64 μm and also more homogeneous as compared to the other composition. Cr-carbides and Cr-nitride were also detected in all samples. Furthermore, high temperature oxidation was conducted to the pack cemented steels at 1050^oC. The oxidation was performed at 700^oC for 90 hours under the steam condition. It was found that the Cr_2O_3 still existed on the steel, and the Cr depth did not significantly change after the oxidation process. These results showed that Cr from the pack cementation process was able to become reservoir for the formation of Cr_2O_3 in high temperature steam oxidation, and its existence could be used for a longer oxidation time.

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η	Efficiency
^{o}C	Temperature unit in degree Celcius
T_{max}	Maximum temperature
T_{min}	Minimum temperature
Wt.%	Weight percent
Δm	Mass change

LIST OF ABBREVIATIONS

Al_2O_3	Aluminium Oxide
BSE	Back-scattered electron
Cr	Chromium
Cr_2O_3	Chromia
CVD	Chemical Vapour Deposition
EDX	Energy Dispersive X-ray
EPMA	Elemental Probe Microanalyser
Fe_2O_3	Hematite
Fe_3O_4	Magnetite
FESEM	Field Emission Scanning Electron Microscopy
MPa	Mega Pascal
NH_4Cl	Ammonium Chloride
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
XRD	X-ray diffraction

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