



## SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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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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LASER CLADDING OF TOOL STEEL FOR GRAIN BOUNDARY STABILITY

PERPUSTAKAAN UNIVERSITI MALAYSIA PAHANG 9	
No. Perolehan <b>T001946</b>	No. Panggilan KK
Tarikh <b>21 JUL 2022</b>	F345 2021 Thesis

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Thesis submitted in fulfillment of the requirements  
for the award of the degree of  
Doctor of Philosophy

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DECEMBER 2021

*“In the name of ALLAH, the Most Beneficent, the Most Merciful”*

This thesis is specially dedicated to:

Beloved husband, father and mother;

**MOHD. HERZWAN BIN HAMZAH**

**FAUZUN BIN YAHYA**

**SHAHRIAH BINTI HAMZAH**

for your love, trust and support along my journey as a student. You are my source of  
inspiration and spirit for me along my study and life.

## ACKNOWLEDGEMENTS

Foremost, I would like to express my deep gratitude to my research supervisor, Associate Professor Dr. Syarifah Nur Aqida binti Syed Ahmad for the patient guidance, sharing germinal ideas, and constantly support in completing this thesis. My sincerely thanks given to her for the time spent to correct my mistakes and giving useful critiques of my research work. Without her guidance, this thesis would not have been completed.

I also would like to express very special thanks to Senior Lecturer Dr. Izwan Ismail from Manufacturing and Mechatronic Engineering Technology Faculty, Universiti Malaysia Pahang (UMP) and Mr. Abdul Fattah Mohd. Tahir, Lecturer of Automotive Manufacturing Department from Kolej Kemahiran Tinggi MARA Kuantan (KKTM) for their ideas, suggestions, help and training throughout the study. Also not forgotten, I would like to extend my thanks to the staff and technicians of the laboratory of Mechanical and Automotive Engineering Technology Faculty (FTKMA), Central Laboratory, Center of Excellence for Advance Research in Fluid Flow (CARIFF) in UMP and Mechanical and Manufacturing Engineering Faculty in Universiti Tun Hussein Onn Malaysia (UTHM) for their supporting ideas and helped especially in technical aspects during this thesis writing.

I would like to acknowledge main support for the research funding from the Ministry of Education Malaysia under Fundamental Research Grant Scheme (FRGS) with the grant number FRGS/1/2016/TK03/UMP/02/4. I also would like to express my sincere gratitude to Pusat Penyelidikan Dan Inovasi, UMP for granting me under Post Graduate Research Scheme and Ministry of Education Malaysia through MyBrain15 program which supports my study fees. My sincere thanks also go to all friends especially my fellow friends from postgraduate students of FTKMA and College of Engineering, UMP for their help and support along this study. Lastly, I would like to thank anyone who kindly helped me along the experiment and completion of this thesis.

I acknowledge my sincere indebtedness and gratitude to my husband Mohd. Herzwan Hamzah, my parents Fauzun bin Yahya and Shahriah binti Hamzah for their love, dream and sacrifice throughout my life. I cannot find appropriate words that could properly describe my appreciation for their devotion, support and faith in my ability to attain my goals. I am also grateful to my siblings Nursyafira, Muhammad Fadhli and Fariza for their continuous support and understanding that were inevitable to make this research done. I attribute the level of my PhD to my family for their enthusiastic encouragement. Finally, I dedicated this thesis to my beloved son, Muhammad Ali bin Mohd. Herzwan.

## ABSTRAK

Teknologi salutan laser adalah salah satu teknik yang berkesan untuk meningkatkan kestabilan haba keluli. Penambahan zarah WC boleh menghalang pergerakan sempadan bijian dalam mikrostruktur yang seringkali terjadi pada struktur yang diubah pada persekitaran suhu tinggi. Tesis ini membentangkan kajian eksperimen tentang salutan laser menggunakan serbuk tungsten karbida (WC) terhadap keluli karbon tinggi H13 untuk meningkatkan kestabilan mikrostruktur. Matlamat utama adalah untuk menghasilkan lapisan gabungan WC dengan penambahbaikan sifat yang sesuai untuk aplikasi suhu tinggi terutamanya dalam industri *die-casting*. Salutan laser dijalankan dengan menggunakan sistem laser bergelombang 1.06  $\mu\text{m}$ . Eksperimen pertama (DOE 1) telah dijalankan dengan menggunakan sistem laser jenis Nd:YAG manakala eksperimen seterusnya (DOE 2, DOE 3 dan DOE 4) menggunakan sistem laser fiber. Lapisan serbuk WC dicampurkan dengan cecair sodium silikat dan 4% PVA sebagai pengikat dan diletakkan diatas keluli sebelum dilaser. DOE 1 mempunyai 16 sampel untuk kajian taburan zarah WC dalam mikrostruktur lapisan gabungan. Manakala DOE 2 dan DOE 3 pula dijalankan untuk mengkaji perbezaan penggunaan pengikat dan juga kesan penyerapan tenaga laser kepada mikrostruktur. DOE 4 yang terakhir dijalankan dengan rekabentuk eksperimen Box-Behnken yang menghasilkan 17 sampel. Tiga faktor yang digunakan adalah kuasa puncak, frekuensi denyut berulang (PRF) dan kelajuan laser dengan julat masing-masing adalah antara 1.8 hingga 2.0 kW, 15 hingga 30 Hz dan 315 hingga 630 mm/min. Analisis statistik telah dijalankan pada DOE 4 untuk mendapatkan rekabentuk eksperimen yang optimum. Semua sampel dianalisis untuk ujian kekerasan, ketebalan lapisan gabungan WC, perubahan elemen dalam mikrostruktur melalui ujian EDXS dan transformasi fasa melalui ujian XRD. Selain itu, ujian kelesuan haba telah dijalankan mengikut process *die-casting* bagi tujuan kajian kestabilan haba lapisan gabungan WC. Ujian kehausan haba merangkumi proses celupan dalam logam aluminium cair, penyejukan diudara dan yang terakhir adalah penyejukan dalam air pada suhu bilik. Ujian ini dijalankan sebanyak 1000, 3000 dan 5000 kitaran. Sampel ujian dianalisis untuk menentukan kadar kehausan struktur lapisan gabungan WC. Simulasi ujian kehausan haba turut dijalankan dengan perisian ABAQUS untuk kajian taburan haba dan tekanan pada permukaan lapisan gabungan WC. Keputusan DOE 4 menunjukkan taburan zarah WC yang homogen berjaya dicapai dalam mikrostruktur lapisan gabungan WC dengan sifat kekerasan maksima sebanyak 2300 HV. Gabungan zarah WC berjaya meningkatkan kekerasan struktur sebanyak 70%. Analisis EDXS menunjukkan elemen W daripada zarah WC yang terlarut telah meresap ke sempadan bijian lalu menguatkan sistem mikrostruktur. Empat fasa telah dikenalpasti melalui analisis XRD iaitu  $\alpha$ -Fe,  $\gamma$ -Fe, tungsten (W), tungsten karbida (W<sub>2</sub>C) dan qusonkite (WC). Ujian kehausan haba menunjukkan kadar kekurangan jisim disebabkan permukaan terhakis yang bertambah seiring dengan jumlah kitaran. Retakan juga terhasil seiring dengan penambahan jumlah kitaran ujian. Selain itu, empat fasa baru terhasil pada sampel yang telah haus haba adalah NiFeAlO<sub>4</sub>, CoWO<sub>4</sub> and FeWO<sub>4</sub>. Keputusan ini disebabkan pengoksidaan dan resapan atom pada permukaan lapisan gabungan WC. Sifat kekerasan sampel yang haus telah menurun sebanyak 23 %. Simulasi haba menunjukkan parameter kehausan haba mempunyai kesan yang nyata terhadap suhu dan taburan tekanan pada sampel laser lapisan. Analisis statistik menemukan rekabentuk eksperimen yang optimum pada 0.901. Penemuan dalam tesis ini adalah penting untuk penambahbaikan kestabilan haba suatu permukaan pada persekitaran kerja suhu tinggi, terutamanya dalam industri *die-casting*.

## ABSTRACT

Laser cladding is one of the best methods to modify the steel surface for enhanced thermal stability properties. Added carbide particle impeded the grain boundary migration which often occurred in metastable modified microstructure at high working temperature. In this thesis, an experimental study of laser cladding on H13 tool steel with tungsten carbide (WC) particles addition is presented. The aim is to produce WC cladded layer with enhanced properties for high temperature applications specifically in die-casting. The laser cladding was conducted using laser systems of 1.06  $\mu\text{m}$  wavelength. The preliminary experiment (DOE 1) was processed using the Nd:YAG laser while other DOE (DOE 2, DOE 3 and DOE 4) with fibre laser. The cladded layer of WC particles with sodium silicate and 4% PVA binder's agent was preplaced prior to laser processing. DOE 1 consists of 16 samples investigates the powder distribution on cladded layer at different parameter settings. Meanwhile, DOE 2 and DOE 3 investigate the effect of binders to powder distribution ratio on clad layer thickness as well as laser energy absorption of the surface. DOE 4 was developed using Box-Behnken design to study the powder distribution and grain evolution of cladded layer. All samples were characterised for hardness properties, depth of cladded layer, elemental changes in microstructure by EDXS analysis and phase transformation by XRD analysis. Thermal wear test that simulated die-casting environment was conducted to investigate the thermal stability of cladded layer. The thermal wear test involves cyclic heating of cladded samples in molten aluminium alloy at an elevated temperature range of 800-850  $^{\circ}\text{C}$  and quenching in room temperature water bath, for 1000, 3000 and 5000 cycles. Sample characterisation was carried out to measure the properties of thermally worn cladded layer. A thermal simulation was developed using ABAQUS software to study the distribution of thermal stress and temperature within cladded layer. The statistical analysis was conducted for DOE 4 with design optimisation. From the findings, powder distribution within clad layer improvement was achieved in samples of DOE 4. The resulted WC particle distribution in DOE 4 shows significant microhardness increment up to 70 %, due to high hardness carbides within the iron matrix. The EDXS analysis indicates WC particle dissolution in the clad layer where W element diffused to the grain boundary, thus strengthen the iron matrix. Four main phases were detected from XRD analysis namely;  $\alpha$ -Fe,  $\gamma$ -Fe, tungsten (W), tungsten carbide (W<sub>2</sub>C) and qusonkite (WC). From the thermal wear test, mass loss and volume of the eroded surface of cladded samples shows an increment with an increasing number of thermal wear cycles. Crack formation and propagation were observed on the thermally worn cladded samples with the increasing number of thermal wear cycles. Phases like NiFeAlO<sub>4</sub>, CoWO<sub>4</sub> and FeWO<sub>4</sub> were diffracted on the thermally worn sample surface in addition to the existing phases ( $\alpha$ -Fe,  $\gamma$ -Fe and W<sub>2</sub>C) indicating oxidation and atomic diffusion occurred on the surface affected by thermal cyclic. Overall, laser clad samples hardness properties reduction was 23 %. Thermal modelling shows significant effect of thermal fatigue parameter towards temperature and stress distribution on cladded sample. The statistical analysis generated optimised design at 0.901. These findings are significant to enhance surface properties especially thermal stability at high working temperature for dies and high wear resistant applications.

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