THE EFFECT OF PRE - TREATMENT FOLLOWED BY CRYOROLLING ON THE MICROSTRUCTURES, MECHANICAL PROPERTIES AND CORROSION BEHAVIOUR OF Al 5052 ALLOY

by

MUHAMMAD ANAS BIN NORAZMAN

Thesis submitted in fulfilment of the
requirements for the degree of
Master of Science

March 2017

ACKNOWLEDGEMENTS

First and foremost, all praises of Allah, Who has given me time, patience and will to finish the project and to complete this thesis. I would like to take this opportunity to thank School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia for providing sufficient resources, facilities and instruments.

My sincerest gratitude goes to my supervisor, Dr. Anasyida Abu Seman who gave me lots of precious comments, support, and guidance throughout the project. Her excellent supervision and constant encouragement have enabled me to complete my Masters project. I would also like to thank Prof. Dr. Zuhailawati Hussain as my co-supervisor and Dr. Tuti Katrina Abdullah who provided their invaluable suggestion throughout the entire project. Special thanks to Siti Aminah Zakaria and Syarifah Mariam Noraini Sayed Ahmad for their assistance and technical suggestions provided to me throughout the entire project.

I am also grateful to all the technicians, academic and administrative staffs for helping me to carry out my project and permitting me to use all the necessary facilities in finishing my project.

Last but not least, I would like to express my gratitude to my parents and family members for their everlasting love and support. Not forget to mention, thanks to my colleagues and fellow postgraduate students who has given me support throughout the year.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AC	Alternating current
Al	Aluminium
AR	Asymmetric rolling
ARB	Accumulative roll bonding
ASTM	American Society for Testing and Materials
CE	Counter electrode
CGP	Constrained groove pressing
CGPA	Cryorolled grain peak-aged alloy
Cr	Chromium
CR	Cryorolled
CRPA	Cryorolled short annealed plus peak-aged alloy
Cu	Copper
DC	Direct current
DSC	Differential scanning calorimetry
EBSD	Electron back scattered diffraction
ECAP	Equal channel angular pressing
EDS	Energy dispersive X-ray spectroscopy
Fe	Iron
FESEM	Field emission scanning electron microscope
FIB	Focused ion beam
FWHM	Full width at half maximum
Ga	Gallium
HPT	High pressure torsion
ICSD	Inorganic crystal structure database

IGC	Intergranular corrosion
Mg	Magnesium
Mn	Manganese
Ni	Nickel
OM	Optical microscope
RE	Reference electrode
RTR	Room temperature rolling
SCC	Stress corrosion cracking
SCE	Saturated calomel electrode
SEM	Scanning electron microscopy
SFE	Stacking-fault energy
SHE	Standard hydrogen electrode
Si	Silicon
SIB	Strain-induced boundaries
SiC	Silicon carbide
SPD	Severe plastic deformation
TE	Torsion extrusion
TEM	Transmission electron microscopy
UFG	Ultrafine-grained
UTS	Ultimate tensile strength
WE	Working electrode
XRD	X-ray diffraction
XRF	X-ray fluorescence
YS	Yield strength
Zn	Zinc

LIST OF SYMBOLS

Å	Angstrom
B	Line broadening
d	Interplanar Spacing
E_{corr}	Corrosion potential
g/cm^3	Gram per cubic centimetre
g/ml	Gram per milliliter
GPa	Gigapascal
h	Hours
Hv	Hardness Scale (Vickers)
I_{corr}	Corrosion current
k	Dimensionless shape factor
K	Kelvin
kg/m^3	Kilogram per cubic meter
kgf	Kilogram-force
kJ/kg	Kilojoules per kilogram
min	Minutes
mm	Milimeter
mm^2	Square millimeter
mm/min	Millimeter per minutes
mm/year	Millimeter per year
MPa	Megapascal
nm	Nanometer
wt.%	Weight percentage
θ	Scattering angle

λ	Wavelength
μA	Microampere
μm	Micrometer
ºC	Degree celcius
°C/min	Degree celcius per minutes
V	Volt
V/s	Volt per second
%	Percentage

KESAN PRA - RAWATAN HABA DIIKUTI OLEH PROSES PENGGELEKAN PADA SUHU KRIOGENIK TERHADAP MIKROSTRUKTUR, SIFAT MEKANIKAL DAN KAKISAN KEPINGAN ALOI Al 5052

ABSTRAK

Kajian ini bertujuan untuk mengkaji kesan pra-rawatan haba yang berbeza ke atas penggelekan kriogenik aloi Al 5052. Dua jenis pra-rawatan; tanpa rawatan haba, penyepuhlindapan (150°C, 200°C, 250°C, 300°C, 350°C) dan rawatan haba larutan (480°C, 510°C, 540°C) telah dipilih. Aloi Al 5052 telah dipanaskan pada pelbagai proses pra-rawatan sebelum melalui proses penggelekan pada suhu kriogenik. Kemudian, aloi Al 5052 dicelup di dalam cecair nitrogen dan digelekkan pada suhu kriogenik pada pengurangan 30% ketebalan. Aloi Al 5052 juga digelek pada suhu bilik pada pengurangan 30% ketebalan sebagai perbandingan. Butir-butir mengenai ciri-ciri mikrostruktur, sifat-sifat fizikal, sifat-sifat mekanik dan tingkah laku kakisan aloi Al 5052 telah dikaji dengan menggunakan mikroskop optik, mikroskop imbasan elektron, elektron bertaburan pembelauan, elektron mikroskop penghantaran, pembelauan sinar-X, ujian mikrokekerasan Vickers, ujian tegangan Instron dan ujian polarisasi potentiodinamik. Optikal mikrograf mempamerkan bahawa ira memanjang sepanjang arah gelekan untuk semua sampel dan sampel yang digelek pada suhu kriogenik menunjukkan nisbah aspek ira lebih tinggi berbanding dengan sampel yang digelek pada suhu bilik. Nisbah aspek ira meningkat dengan peningkatan suhu bagi kedua-dua pra-rawatan dan nisbah aspek ira yang paling tinggi dicapai pada suhu 300°C bagi pra-penyepuhlindapan dan 540°C bagi pra-rawatan haba larutan. Struktur sampel yang melalui proses penggelekan pada suhu kriogenik untuk kedua-dua pra-rawatan menunjukkan ketumpatan kehelan yang tinggi dan pembentukan ira bersaiz

submikron. Sifat-sifat mekanikal menunjukkan bahawa sampel yang digelek pada suhu kriogenik yang melalui pra-penyepuhlipan pada 300°C menunjukkan nilai kekerasan, kekuatan alah dan kekuatan tegangan muktamad yang tertinggi dengan peningkatan sebanyak 17.3%, 13% dan 15%, masing-masing, berbanding dengan sampel tanpa pra-rawatan haba yang digelek pada suhu kriogenik. Manakala, sampel yang digelek pada suhu kriogenik yang melalui pra-rawatan haba larutan pada suhu 540ºC menujukkan nilai kekerasan, kekuatan alah dan kekuatan tegangan muktamad yang tertinggi dengan peningkatan sebanyak 13.6%, 7.1% dan 4.8%, masing-masing, berbanding dengan sampel tanpa pra-rawatan haba yang digelek pada suhu kriogenik. Sampel yang digelek pada suhu kriogenik yang melalui pra-penyepuhlipadan pada suhu 300°C dan pra-rawatan haba larutan pada suhu 540°C mempamerkan nilai ketahanan kakisan yang tertinggi kerana bacaan arus kakisan, I_{corr} dan kadar kakisan paling rendah dan nilai keupayaan kakisan yang lebih positif, E_{corr}. Sampel yang digelek pada suhu kriogenik yang melalui pra-penyepuhlindapan pada suhu 300°C mempunyai sifat-sifat fizikal, mekanikal dan kakisan yang terbaik diantara semua sampel.

.

THE EFFECT OF PRE – TREATMENT FOLLOWED BY CRYOROLLING ON THE MICROSTRUCTURES, MECHANICAL PROPERTIES AND CORROSION BAHAVIOUR OF Al 5052 ALLOY

ABSTRACT

This research was aimed to study the effect of different pre-heat treatment on cryorolled Al 5052 alloy. Two types of pre-treatments; without pre-treatment, annealing (150°C, 200°C, 250°C, 300°C and 350°C) and solution treatment (480°C, 510°C and 540°C) were selected to study the effect of pre-heat treatment on the properties of cryorolled Al 5052 alloy. Al 5052 alloys were heated at various pre-heat treatments process before cryorolling. Then, the Al 5052 alloys were dipped in liquid nitrogen and rolled at cryogenic temperature (cryorolling) at 30% thickness reduction. The Al 5052 alloy also was cold rolled at the 30% thickness reduction for comparison. The details of microstructural characteristics, physical properties, mechanical properties and corrosion behaviour of Al 5052 alloy were investigated using optical microscope (OM), scanning electron microscope (SEM), electron back scattered diffraction (EBSD), transmission electron microscope (TEM), X-ray diffraction (XRD), Vicker microhardness, Instron tensile test and potentiodynamic polarization test. The optical micrograph revealed that the grains are elongated along the rolling direction and the cryorolled samples showed higher grain aspect ratio compared to cold rolled. The grain aspect ratio increases with pre-heat treatment temperatures for both pre-heat treatment and achieved the highest aspect ratio at 300°C for pre-annealed and 540°C for pre-solution treated. The structure of cryorolled sample for both pre-heat treatment samples showed high density of dislocations and formation of submicron-sized grains. The mechanical properties

showed that the cryorolled sample pre-annealed at 300°C exhibited the highest hardness, yield and ultimate tensile strength values with improvement of 17.3%, 13% and 15%, respectively, compared to non pre-treatment cryorolled sample. In contrast, the cryorolled sample pre-solution treated at 540°C exhibited the highest hardness, yield and ultimate tensile strength values with improvement of 13.6%, 7.1% and 4.8%, respectively, compared to non pre-treatment cryorolled sample. The cryorolled sample pre-annealed at 300°C and cryorolled sample pre-solution treated sample at 540°C exhibited the highest corrosion resistance due to lowest corrosion current, I_{corr} and corrosion rate values and more positive value of corrosion potential, E_{corr}. The cryorolled sample pre-annealed at 300°C displayed an excellent physical, mechanical and corrosion properties compared to other samples.