MICROSTRUCTURE ANALYSIS, MECHANICAL PROPERTIES AND CORROSION BEHAVIOUR OF CRYOROLLED ALUMINIUM 1100 ALLOY WITH DIFFERENT HEAT TREATMENT

by

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LIST OF ABBREVIATIONS

Al	Aluminium
AR	Asymmetric rolling
ARB	Accumulative roll bonding
ASTM	American Society for Testing Materials and Minerals
CE	Counter electrode
CGP	Constrained groove pressing
CP-Al	Commercially pure aluminium alloy
Cr	Chromium
CR	Cryorolling
CRPA	Cryorolled short annealed with peak-aged
Cu	Copper
DC	Direct current
DSC	Differential scanning calorimetry
ECAP	Equal channel angular pressing
EDS	Energy dispersive X-ray spectroscopy
Fe	Iron
FESEM	Field emission scanning electron microscope
FWHM	Full width at half maximum
Ga	Gallium
HPT	High pressure torsion
Mg	Magnesium
Mn	Manganese
NaCl	Sodium chloride
Ni	Nickel
OM	Optical microscope
RD	Rolling direction
RE	Reference electrode
RTR	Room temperature rolling
SCE	Standard calomel electrode
SEM	Scanning electron microscope
SFE	Stacking fault energy

SHE	Standard hydrogen electrode
Si	Silicon
SiC	Silicon carbide
SPD	Severe plastic deformation
TE	Torsion extrusion
TEM	Transmission electron microscope
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
°C	degree Celcius
d	grain size/interplanar spacing
E_{corr}	corrosion potential
g/cm^3	gram per cubic centimeter
g/ml	gram per mililiter
h	hour
Hv	Vickers hardness scale
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/year	milimeter per year
mol/l	mole per litre
MPa	megapascal
nm	nanometer
V	volt
wt. %	weight percentage
θ	scattering angle
µm	micrometer
λ	wavelength

ANALISIS MIKROSTRUKTUR, SIFAT-SIFAT MEKANIKAL DAN KAKISAN GELEKAN KRIOGENIK ALOI ALUMINUM 1100 DENGAN RAWATAN HABA YANG BERBEZA

ABSTRAK

Kajian semasa ini mengkaji mikrostruktur, sifat-sifat mekanikal dan kakisan gelekan kriogenik aloi Al 1100 pada pra-rawatan haba yang berbeza. Sebelum proses penggelekan kriogenik, tiga jenis pra-rawatan haba telah dipilih; tanpa rawatan haba, penyepuhlindapan (200˚C-400˚C) dan rawatan haba larutan (500˚C-580˚C). Sampel pra-rawatan pengepuhlindapan (250˚C) dan sampel pra-rawatan haba larutan (540˚C) menunjukkan nisbah aspek ira yang paling tinggi. Kedua-dua sampel menunjukkan saiz kristalit yang paling kecil (37.53 nm, 46.52 nm) dan terikan kekisi tertinggi iaitu (9.50×10^{3},9.02×10^{-3}) untuk pra-penyepuhlindapan dan pra-rawatan haba larutan. Sampel pra-penyepuhlindapan gelekan kriogenik menunjukkan nilai kekerasan yang tinggi, dan peningkatan kekuatan alah dan kekuatan ketegangan dengan kenaikan adalah 43.44%, 24.64% dan 20.33% masing-masing. Peningkatan kekerasan, kekuatan alah dan kekuatan ketegangan sampel pra-rawatan haba larutan gelekan kriogenik dicapai pada suhu 540˚C dengan kenaikan adalah 16.93%, 1.20% dan 5.6% masing-masing. Kekerasan dan kekuatan tegangan selepas pasca penyepuhlindapan secara beransur-ansur berkurangan, tetapi kemuluran bertambah bagi kedua-dua sampel. Sampel pra-penyepuhlindapan gelekan kriogenik selepas pasca penyepuhlindapan pada 175˚C menunjukkan ketumputan kehelan yang tinggi dengan pembentukan struktur-struktur ira-ira yang kecil. Kedua-dua sampel gelekan kriogenik pra-rawatan haba iaitu sampel pra-rawatan pengepuhlindapan (250˚C) dan sampel pra-rawatan haba larutan (540˚C) masing-masing mempamerkan rintangan kakisan tinggi dengan kadar kakisan 0.0214mm/tahun dan 0.0272mm/tahun, arus kakisan yang terendah 1.30µA dan 2.14 µA dan nilai potensi kakisan lebih positif -0.7970V dan -0.9774V. Secara keseluruhannya, sampel pra-penyepuhlindapan gelekan kriogenik pada 250˚C diikuti rawatan pasca penyepuhlindapan pada 175˚C menunjukkan nilai terbaik (sifat-sifat mekanikal dan kelakuan kakisan).

MICROSTRUCTURAL ANALYSIS, MECHANICAL PROPERTIES AND CORROSION BEHAVIOUR OF CRYOROLLED ALUMINIUM 1100 ALLOY WITH DIFFERENT HEAT TREATMENT

ABSTRACT

The present work investigated the microstructure, mechanical properties and corrosion behavior of cryorolled Al 1100 alloy in various pre-heat treatment. Before subjecting to cryorolling process, three different pre-heat treatment were selected; non pre-heat treatment, annealing (200˚C- 400˚C) and solution treatment (500˚C-580˚C). Pre-annealed sample at 250˚C and pre-solution treated sample at 540˚C showed the highest grain aspect ratio. Both of the samples also showed the smallest crystallite size (37.53 nm, 46.52 nm) and the highest lattice strain (9.50×10^{3}, 9.02×10^{-3}). Pre-annealed cryorolled sample also resulted in higher hardness, ultimate tensile strength and yield strength with an improvement of 43.44%, 24.64% and 20.33% respectively. The improvement of hardness, tensile strength and yield strength were achieved for pre-solution treated cryorolled sample at 540˚C with increment of 16.93%, 1.20% and 5.6% respectively. The hardness and tensile strength after post annealed gradually decreased, but ductility increased for both samples. Cryorolled pre-treatment samples after post-annealed at 175˚C showed a high density of dislocations with formation of new sub-grains structure. Both of the pre-heat treatment cryorolled sample exhibited higher corrosion resistance with the lowest corrosion rate 0.0214mm/year and 0.0272mm/year and corrosion current 1.30µA and 2.14 µA, and more positive value of corrosion potential -0.7970V and -0.9774V for pre-annealed cryorolled and pre-solution treated sample respectively. Overall, the pre-annealed at 250˚C cryorolled sample followed by post-annealed

treatment at 175˚C has shown the best value (mechanical and corrosion behaviour) in general.

CHAPTER ONE
INTRODUCTION

1.1 Research background

Aluminum and its alloys are extensively used for various design, engineering applications, where the high strength to weight ratio is one of the basic criteria. The unique properties of aluminum alloys, such as high specific strength, good formability, high corrosion resistance, and recycling potential, make them ideal for replacing the heavier alloys currently used in vehicles in the automotive industries (Krishna et al. 2015). The advantages of pure aluminum make it particularly appropriate for intricate forming processes by virtue of its high ductility and the ideal ratio of Young's modulus to mass density especially as the grain size is reduced (Hamid et al. 2015). It is well known that about 46% of aluminum alloys used for various applications are in the form of sheets and plates (Krishna et al. 2016). An increased usage of these alloys depends on enhancing their mechanical properties such as strength and toughness further. Many important mechanical properties of materials, including yield strength, hardness and toughness can be improved by refining the grain size. The grain refinement of bulk Al alloys to ultrafine regime can further enhance its mechanical properties.

With the rapid development of ultrafine grained metals, severe plastic deformation (SPD) techniques have become increasingly important in rolling. Apart from the accumulative roll bonding technique (ARB), cryorolling (CR) were also considered as SPD techniques. The ARB required a special processing equipment (in differential speed rolling), and limited size (Liu et al. 2014). In order to overcome this constraint, rolling at cryogenic temperature process in which the low temperature is maintained by liquid nitrogen (Yu et al., 2013) was identified as a viable route to produce a large scale product with ultrafine-grain size. During cryorolling, the suppression of dynamic recovery was found to be the main reason for enhancing the strength of material. These are due to the high density of defects that generated during deformation, which act as the potential recrystallization sites (Krishna et al, 2010). Moreover, cryorolling cause the formation of higher density of dislocations compared with the other SPD method. These dislocations will act as driving force for the initiation of the large number of nucleation sites, which resulted in forming the submicrocrystalline or ultrafine grained material (Panigrahi et al, 2008).

Past research have been reported that cryorolling was successfully produced bulk structure with a combination of high strength and ductility in pure metal such as copper (Wang et al., 2003), nickel (Naidenkin et al., 2014), steel alloy (Kvackaj et al., 2014) and aluminium and its alloy. The studies on the production of ultrafine grained aluminum alloy by cryorolling process have been reported on Al 7075 (Panigrahi et al., 2011a), Al 6082 (Kumar et al., 2015), Al 6063 (Panigrahi and Jayagnathan, 2009; 2011b), Al 6061 (Yu et al., 2013), Al 5083 (Lee et al.,2005; Gopala et al., 2010; Singh et al., 2013), Al 5052 (Chandra et al., 2013), and pure aluminium (Rangaraju et al., 2005; Huang et al., 2010; Marnette et al., 2014).

1.2 Problem statement

Deformation at cryogenic temperature has emerged as a potential method to develop ultrafine-grained (UFG) materials with improved mechanical properties. The formation of UFG microstructures in various Al alloys has been investigated by many researchers. The study on the commercially pure aluminium alloy by cryorolling process also have been reported to increase the hardness and strength as its limited the use of pure aluminium alloy in structural application. The presents study on pure aluminium alloy by cryrolling process included the comparison between room temperature rolling, warm rolling and cryogenic rolling, the different in strain hardening during the deformation process and the effect of cryorolling with post treatment. For example, Marnette et al., (2014) have reported that ultrafine-grained Al 1100 alloy with thickness reduction from 10 mm to 2 mm can be produced with both rolling temperatures; room temperature rolling (27˚C) and cryogenic temperature rolling (below -150˚C). However, at the same deformation rate (80% thickness reduction), CR materials exhibited 30% higher in tensile strength. Moreover, the microstructural and mechanical properties of Al 1100 alloy were observed under different stress conditions by Naga Krishna et al., (2010). They claimed that cryorolled material caused in minimal variation in length over wide ratio which is closed to 1.0 and shear strains (0.01 to 0.08) compared to the conventional rolled sample with variation in length over wide ratio is varies from 1.6 to 2.4.

In other work, the combined cryorolling process with post-treatment were reported by Tsuji et al., (2002), Rangaraju et al., (2005), Sabirov et al., (2008), Sivaprasad et al., (2010), Ralston et al., (2011), Ashtiani and Karami, (2015), Rajat, (2014), and Dasharath and Mula, (2016) on Al 1xxx series alloy. All the research studies showed the improvement in hardness, tensile properties and reduced the grain size after subjecting to plastic deformation at cryogenic temperature. For example, Sabirov et al., (2011) have reported cryorolled then followed by short-annealed at 160˚C formed ultrafine grained structures with an average grain size 0.1-0.4 µm and exhibited high strength (550 MPa). Moreover, Rangaraju et al., (2005) have reported that finer grain after subjected to cryorolling and post-annealing treatment (275˚C)