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- Vaibhav Pandey

DEDICATED

to

Prof. Vakil Singh

&

My Beloved Parents

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Abbreviations

ASTM	**A**merican **S**ociety for **T**esting and **M**aterials
CPE	**C**onstant **P**hase **E**lement
CRSS	**C**ritically **R**esolved **S**hear **S**tress
DT	**D**amage **T**olerance
EBSD	**E**lectron **B**ack **S**cattered **D**iffraction
EDS/EDX	**E**nergy **D**ispersive **X**-ray **S**pectroscopy
EIS	**E**lectrochemical **I**mpedance **S**pectroscopy
FCC	**F**ace **C**entered **C**ubic
FESEM	**F**ield **E**mission **T**ransmission **E**lectron **M**icroscope
GNS	**G**radient **N**ano **S**tructure
GP	**G**uinier **P**reston
HAADF	**H**igh **A**ngle **A**nnular **D**ark **F**ield
HCF	**H**igh **C**ycle **F**atigue
HCP	**H**exagonal **C**lose **P**acking
HPT	**H**igh **P**ressure **T**orsion
HRTEM	**H**igh **R**esolution **T**ransmission **E**lectron **M**icroscope

IFFT	**I**nverse **F**ast **F**ourier **T**ransform
IQ	**I**mage **Q**uality
LCF	**L**ow **C**ycle **F**atigue
MB	**M**icro **B**and
OCP	**O**pen **C**ircuit **P**otential
PA-USSP	**P**eak **A**ging followed by **U**ltra **S**onic **S**hot **P**eening **T**reatment
PA-USSP-SR	**P**eak **A**ging & **U**ltra **S**onic **S**hot **P**eening **T**reatment & **S**tress **R**elieved
PD	**P**otentio **D**ynamic
RPM	**R**otation **P**er **M**inute
SADP	**S**elected **A**rea **D**iffraction **P**attern
SC	**S**econdary **C**racks
SCC	**S**tress **C**orrosion **C**racking
SFE	**S**tacking **F**ault **E**nergy
SSP	**S**ever **S**hot **P**eening
SSRT	**S**low **S**train **R**ate **T**ensile
SSS	**S**upersaturated **S**olid **S**olution
ST-USSP-PA	**S**olution **T**reated & **U**ltra **S**onic **S**hot **P**eening **T**reatment & **P**eak **A**ging
TEM	**T**ransmission **E**lectron **M**icrographs
USSP	**U**ltra **S**onic **S**hot **P**eening
XRD	**X**- **R**ay **D**iffraction

Symbols

°C	Degree Centigrade
μm	Micrometer
nm	Nanometer
α	Alpha
θ	Theta
Wt.%	Weight Percent
kHz	Kilo Hertz
mHz	Mili Hertz
mg	Mili Gram
mm	Mili Meter
ml	Mili Liter
t	Time (h)
A	Area (cm^2)
a	Lattice parameter
B	Line Broadening
D	Average crystallite Size

D_0	Initial Grain Size
k	Temperature dependent rate constant
n	Grain growth constant
R	Ideal gas constant
Q	Activation Energy
g	Gram
cm	Centimeter
h	Hour
s	Second
K	Constant (8.76 x 10^4)
W	mass loss (mg)
KN	Kilo Newton
$>$	Greater than
$<$	Less than
b	Burger Vector
λ	Wavelength
ε	Root mean square of micro-strain
β	Beta
η	Equillibrium precipitate
η'	Non-equillibrium Phase
MPa	Mega pascal
R_a	Average surface roughness

H$_v$	Hardness
T$_t$	Transition temperature
v	Poisson’s Ratio
2N$_f$	Number of reversals to failure
ϵ'_f	Fatigue ductility coefficient
W$'_f$	Plastic strain energy density coefficient
N$_i$	Number of cycles to crack initiation
N$_p$	Number of cycles to crack propagation
I_{corr}	Corrosion current
E_{corr}	Corrosion potential
E_{pit}	Pitting potential
R$_p$	Polarization resistance
β_a	Anodic tafel slope
β_c	Cathodic tafel slope
Y$_0$	Constant phase element impednce
R$_u$	Solution resistance between solution and reference electrode

PREFACE

Aluminium alloys are widely used for structural applications in aerospace, automobile and construction industries. The attractiveness of aluminium alloys is due to their relatively low cost, light weight and high specific strength. However, aluminium alloys have relatively low modulus of elasticity, low elevated-temperature capability ($\leq$130°C), and are susceptible to corrosion in certain environments.

Due to the age hardening Al alloys become susceptible to different forms of corrosion such as stress corrosion cracking, pitting corrosion, intergranular corrosion especially in chloride environment. In these alloys corrosion occurs due to presence of intermetallic particles acting either anodic or cathodic with respect to the matrix. Structural components of aircrafts such as wings experience cyclic loading and undergo failure due to initiation and propagation of fatigue cracks from the surface. Since majority of fatigue cracks initiate from the surface, the microstructure at the surface plays important role on fatigue resistance of such alloys.

It is widely accepted that a gradient microstructure with nanostructured surface layer and coarse-grained interior provides excellent fatigue properties, increasing the resistance against fatigue crack initiation and propagation. The resistance of structural aluminium alloys against corrosion, fatigue and wear is strongly affected by the state of the surface and the different processes used for surface modification such as ultrasonic shot peening (USSP), surface mechanical attrition treatment (SMAT), laser shock peening, shot peening, sand blasting, and sliding wear to improve their performance. USSP involves repeated impact of hard balls on surface of the workpiece to cause work hardening and induce compressive residual stresses in surface region. In comparison with the usual process of shot peening, USSP induces plastic deformation and compressive residual stress to larger depth along with formation of nanostructure in the surface region because of the high kinetic energy associated with the hard balls in this process. USSP produces gradient structure of nano size in the surface region and a fine-grained structure of progressively increasing grain size, up to the substrate. Further, compressive residual stress in surface region of the component/specimen increases the resistance of material against crack initiation as well as retards the rate of crack propagation.

The present study deals with characterization of surface nanostructure developed from the USSP treatment and effect of the nanostructuring on low cycle fatigue and corrosion behavior of the important aircraft grade aluminium alloy 7075 in peak aged condition. The thesis comprises of nine chapters.

Chapter-1 presents a brief introduction along with literature review on properties and applications of the AA7075. It also presents the details of grain refinement processes in metals/alloys. USSP improves both fatigue resistance and corrosion resistance of aluminium alloys. The objectives of present investigation are listed at the end of this chapter.

Chapter-2 deals with details of the experimental procedure of USSP and characterization of the nanostructure in surface region of the AA7075. The 7075 aluminium alloy was procured from M/s Hindalco Industries Limited, Renukot, India, in the form of cylindrical bar of 54 mm diameter and 1000 mm length. The material was studied in retrogression and re-aged (RRA) condition in which the alloy was solution treated at 470°C for 30 min, pre-aged at 120°C for 24 h, followed by retrogression at 200°C for 10 min and subsequent secondary aging at 120°C for 24 h.

Chapter-3 presents the effect of USSP on microstructure modification, surface roughness and microhardness. Peak aged AA7075 mainly constituted fine dispersed precipitates of GP-zones, η' coarse precipitates of η and E-phase ($Al_{18}Cr_2Mg_3$). The samples USSP treated for different durations of 15, 30, 60, 180 and 300 seconds were examined for microstructural changes and phase transformation, if any. The average surface roughness was found to increase with increase in USSP duration. Microhardness was found to be highest in the USSP treated surface region and gradually decreases towards the substrate. Microhardness of the surface region and also the depth of the affected region increased with the duration of the USSP treatment. No phase transformation was observed due to USSP as confirmed by the XRD. Nanostructures of 20, 20, 18 and 16 nm sizes developed in surface region after USSP for durations of 30, 60, 180 and 300 seconds, respectively.

Chapter-4 describes the thermal stability of nanostructured surface layer generated from USSP treatment. The thermal stability and other features such as precipitation of hardening particles, grain growth kinetics and microstructural evolution of the nanostructured surface layer were investigated by annealing USSP samples at different temperatures from 150°C-350°C. Retention of nanostructure was observed at 150°C. Precipitates started to reappear at 200°C, coarsened and finally dissolved at around 350°C. The nanograins resulting from USSP were thermally stable up to 250°C and grain coarsening occurred at higher temperature of 300°C, however, the grain size was less than 100 nm even after annealing at 350°C. The high thermal stability of the nanostructure was due to pinning of the grain boundaries by fine precipitates. Quantitative evaluation of the different strengthening processes showed that grain boundary strengthening from the Hall-Petch relationship and dislocation hardening as per the Bailey-Hirsch relationship were the dominant strengthening mechanisms.

Chapter-5 describes the effect of USSP on low cycle fatigue (LCF) behavior of the AA7075. The LCF samples were USSP treated for 30, 60, 180 and 300 seconds. Strain controlled LCF tests were conducted for the un-USSP and different USSP treated samples, at different total strain amplitudes ($\Delta\varepsilon_t/2$) of ±0.60%, ±0.55%, ±0.50%, ±0.45%, ±0.40% and ±0.38%. In general, fatigue life was increased with decrease in strain amplitude, for the both, un-USSP as well as USSP treated samples. However, the improvement in fatigue life of the USSP treated samples was more prominent at lower strain amplitudes. Enhancement in LCF life was observed by USSP treatment up to the duration of 180 s, however, fatigue life was reduced from longer duration of USSP for 300 s. Pronounced enhancement in LCF life resulted from the USSP treatment for 180 s due to combined beneficial effect of grain refinement in the surface region and the associated compressive stresses without any damage of the treated surface. USSP treatment for 300 s (USSP 300) caused damage on the surface, cracks were developed and fatigue life was reduced.

Chapter-6 presents the role of thermal treatments, pre- and post- USSP to reduce the associated residual compressive stress and the modification of microstructure on low cycle fatigue behavior of the AA7075, at room temperature (RT). The un-shot peened samples are designated as PA-unUSSP. The peak aged samples subjected to USSP are designated as PA-USSP. The USSP treatment was carried out for 180 seconds at constant amplitude of 80μm with hard steel balls of 3 mm diameter. Following the USSP treatment, some samples were subjected to stress relieving treatment at 90°C for 4 h to relieve the residual stress and these samples are designated as PA-USSP-SR. Another set of the specimens in solution treated condition were subjected to USSP for the same duration of 180 seconds and subsequently to peak aging (PA) and these were designated as ST-USSP-PA. The high density of dislocations generated during the USSP promoted nanosize precipitates of the second phase particles during the peak aging treatment. In ST-USSP-PA condition the high density of η' precipitates along with nanograined surface layer resulted in delaying the process of crack initiation and thus led to enhanced LCF life. Decrease in dislocation density and relieving of compressive residual stress was observed after the stress relieving treatment which resulted in decrease in LCF life in the PA-USSP-SR condition.

Chapter-7 presents corrosion behavior of the USSP treated AA7075 in 3.5 wt% NaCl solution. The sample USSP treated for 15s (USSP 15) exhibited lower current density (0.564 mA/cm^2) and higher corrosion potential (-0.695 V) as compared with that of the un-USSP specimen with 1.269 mA/cm^2 and -0.839 V, respectively. The enhancement in corrosion resistance of USSP treated sample was due to rapid development of uniform, homogeneous and effective passive layer on the nanostructured surface coupled with refinement of the coarse precipitates. Also, there was optimum combination of surface roughness, compressive residual stress, and dislocation density in the surface region to produce highest corrosion resistance in the USSP 15 condition.

Chapter-8 deals with the optimization of the USSP duration for enhanced corrosion resistance. USSP was performed for different durations of 5, 10, 15, 20, 25, 30 seconds and samples are designated as USSP 5, USSP 10, USSP 15, USSP 20, USSP 25 and

USSP 30, for the optimization. Among the specimens USSP treated from 5 to 30 seconds, the one USSP treated for 15 seconds (USSP 15) was found to exhibit highest corrosion potential (E_{corr}) and lowest corrosion current density (i_{corr}). The enhanced corrosion resistance of the USSP 15 sample was found to be due to combined effect of surface nanostructure of the matrix, homogeneity and refinement of second phase precipitates. Also slow strain rate tests (SSRT) were performed at constant strain rate of $1 \mathrm{x} 10^{6} \mathrm{s}^{-1}$ to evaluate stress corrosion cracking (SCC) behavior. The tensile strength in SSRT for the USSP treated sample was enhanced significantly and the susceptibility to SCC was reduced as compared to that of un-USSP.

Chapter-9 presents the major conclusions drawn from the present investigation along with suggestions for the future work.