Dedicated to

My Beloved Parents

Shri Giradhar Singh

&

Smt. Sona Devi

Declaration

I hereby declare that the work presented in this Thesis titled ***Effects of Processing Methods on Microstructure-Flow Property Relationships at Ambient and Elevated Temperatures in Binary Al-Si Alloys*** – *Doctor of Philosophy* submitted to the Indian Institute of Technology Jodhpur in partial fulfilment of the requirements for the award of the degree of Doctor of Philosophy, is a bonafide record of the research work carried out under the supervision of **Professor Bhagwati Prasad Kashyap**. The contents of this thesis, in full or in parts, have not been submitted to, and will not be submitted by me to, any other Institute or University in India or abroad for the award of any degree or diploma.

Kuldeep Singh
P18MT001

Certificate

This is to certify that the thesis titled ***Effects of Processing Methods on Microstructure-Flow Property Relationships at Ambient and Elevated Temperatures in Binary Al-Si Alloys***, submitted by *Kuldeep Singh* (P18MT001) to the Indian Institute of Technology Jodhpur for the award of the degree of *Doctor of Philosophy*, is a bonafide record of the research work done by him under my supervision. To the best of my knowledge, the contents of this report, in full or in parts, have not been submitted to any other Institute or University for the award of any degree or diploma.

Bhagwati Prasad Kashyap
Ph.D. Thesis Supervisor

Abstract

Al-Si alloys constitute a widely used group of light metals and alloys. In spite of it traditionally being a well-studied system, no systematic attempts have been made to overcome the strength-ductility limitations existing in these alloys. It becomes even more important to investigate when numerous developments have been witnessed in playing with microstructure control by newer processing methods to enrich our capability of broadening the process-structure-property optimization regimes.

This research investigates the relationship between composition, processing, structure, and properties of binary Al-2Si to Al-30Si alloys at room and high temperatures. The Al-Si alloys were produced by melting and casting in the induction furnace. Microstructure and mechanical property studies were conducted on as-cast, hot extrusion, friction stir processing (FSP), and high-pressure torsion (HPT) processed Al-Si alloys. The minimum grain sizes obtained are 1-2μm on HPT, 2-3μm upon FSP, and 5-8μm on extrusion, which are much smaller than the as-cast grain sizes of 15-115μm.

Irrespective of processing methods, strength was found to increase with decreasing grain size according to the Hall-Petch type relationship, but the strengthening caused by the grain interior and the grain boundary zone was found to be sensitive to the processing method. In contrast to this, at elevated temperatures (450-840K), the strength decreased, whereas elongation increased with decreasing grain size. Superplastic behaviour investigated in FSP material showed elongations of 191-341%, depending on Al-Si composition, with the maximum elongation of 341% corresponding to the Al-12Si eutectic alloy.

The values of parameters of the constitutive relationship for high-temperature deformation were obtained to be as follows. (i) Strain rate sensitivity index (m) was found to be a maximum of ~0.4 for FSPed Al-12Si alloy, whereas it was a minimum of 0.04 for the hypereutectic Al-30Si alloy. The magnitude of m increased with test temperature and generally decreased with increasing Si content for all the processes involved. (ii) The apparent activation energy for deformation (Q_a) was obtained to range from 79.4±11.8kJ/mol to 572±148kJ/mol. The FSP material shows $m \geq 0.4$ and true activation energy Q_t~59.4kJ/mol over the temperature range of 530-650K and 221.5kJ/mol over 650-780K. The mechanism for superplastic deformation is accordingly suggested to be grain boundary sliding and its accommodation by grain boundary diffusion at the lower end of the temperature range, and the same by lattice diffusion at the upper end of the temperature range. Q_t generally increased whereas m decreased with increasing Si content, for which the deformation mechanisms were identified to be dislocation climb creep for low Si-containing Al-Si alloys, but power law breakdown appeared for hypereutectic Al-Si alloys in as-cast and extruded Al-Si alloys. According to the present results on FSPed Al-8Si to Al-30Si, both dislocation-based creep mechanisms ($m \leq 0.3$) and superplasticity-type ($m \geq 0.3$) mechanisms occur over different ranges of temperatures, strain rates, and compositions.

In conclusion, it is possible to make Al-Si alloy stronger by selecting higher Si content, but irrespective of composition, the same can be made to exhibit superplasticity, thus providing an opportunity to extend the applications of Al-Si alloys by proper processing and meeting the service conditions.

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Kuldeep Singh
Ph.D. Student

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List of Symbols

Symbol	Description
Al	Aluminium
Si	Silicon
α-Al	Solid solution of Si in Al
wt.%	Weight Percentage
Na	Sodium
P	Phosphorus
Sr	Strontium
Sc	Scandium
Ca	Calcium
B	Boron
Pb	Lead
g/cc	Gram per centimetre cube
°C	Degree Centigrade
Ω	Ohm
m	Meter
W	Watt
K	Kelvin
HV	Micro Vickers Hardness
Fe	Iron
AA4032	Aluminium, 21%wt Silicon, 0.82%wt Nickel and 0.67%wt Copper
T651	Heat Treatment
ε_{eq}	Equivalent Strain
N	Number of HPT turns
r	Sample Radius
t	Thickness of the Sample
(α+β) Ti	Two-Phase Titanium Alloy
Al7075	Aluminium alloy with major Zinc and Magnesium elements
d_m	Metal Grain Size
d_c	Ceramic Grain Size
µm	Micron
T	Temperature
T_m	Melting Temperature
$\dot{\varepsilon}$	Strain Rate
s	Second
E	Elastic Modulus
b	Burgers Vector
p	Exponent of inverse grain size
m	Strain Rate Sensitivity Index
n	Inverse of Strain Rate Sensitivity Index
Q	Activation Energy for Deformation
k	Boltzmann Constant
$D_0 \exp\left(\frac{-Q}{RT}\right)$	Diffusion Coefficient
A	Material or Mechanism-Dependent Constant
σ	Flow Stress
Q_a	Apparent Activation Energy
Q_t	True Activation Energy
R	Universal Gas Constant
Q_{gb}	Activation Energy for Grain Boundary Diffusion

Q_l	Activation Energy for Lattice Diffusion
Q_{IPB}	Activation Energy for Interphase Boundary Diffusion
kJ/mol	Kilo Joule Per Mol
L	Length
D_T	Diameter at Top Side
D_B	Diameter at Bottom Side
w	Width
t_p	Thickness of the Plate
γ	Shear Strain
θ	Rotation Angle in Radian
180, 2000	Grit Size of Emery Paper (Particles Per Square Inch)
D	Sample Diameter
h	Sample Height
g	Gram
σ_e	Engineering Stress
ε_e	Engineering Strain
P	Load
A_{cs}	Cross-sectional Area of Gage Section
d	Diameter of Gage Section
ΔL	Change in Gage Length
L_0	Initial Gage Length
σ_t	True Stress
ε_t	True Strain
θ_H	Work hardening Rate
θ_S	Work softening Rate
σ_p	Peak Stress
ε_p	Strain Corresponds to Peak Stress
σ_0	Stress at the Onset of Plastic Strain
ε_0	Strain Corresponds to Onset of Plastic Deformation
σ_S	Stress at the End of Softening Part
ε_S	Strain Corresponds to σ_S
K	Strength Coefficient
n	Strain-Hardening Exponent
σ_y	Yield Strength
σ_{HP}	Hall-Petch Parameter (slope)
k_{HP}	Intercept of Hall-Petch Plot
k'	Material Constant
C', D'	Constant
R^2	Correlation Coefficient
d_α	α-Al Grain Size
d_p	Particle Size
f	Volume Fraction
C_{Si}	Silicon wt.%
f_α	α-Al Fraction
f_p	Primary Si Fraction
f_{eu}	Eutectic Fraction
C_t	Compression Test
T_t	Tension Test
ε_f	Percentage Elongation
K_H, n_H	Hollomon Parameters; Strength Coefficient and Strain Hardening Exponent, respectively
K_{Ht}, n_{Ht}	Hollomon Parameters from Tension Test
K_{Hc}, n_{Hc}	Hollomon Parameters from Compression Test

A_{CS}, B_{CS}	Constants Used for As-Cast Samples
Λ	Interparticle Spacing
l	Length of Particle
A_{ES}, B_{ES}	Constants Used for Extrusion Samples
K_{HFS}, n_{HFS}	Hollomon Parameters Used for FSP Samples
A_{FS}, B_{FS}	Constants Used for FSP Samples
A_{HS}, B_{HS}	Constants Used for HPT Samples
A_H, B_H	Constants for Hardening
A_S, B_S	Constants for Softening
HV_d	Microhardness of Deformed Sample
d_{CV}	Cavity Size
ε_f	Fracture Strain
Z	Zener-Hollomon Parameter

List of Abbreviations

Abbreviation	*Full form*
SDAS	Secondary Dendrite Arm Spacing
SPD	Severe Plastic Deformation
FSP	Friction Stir Processing
HPT	High Pressure Torsion
ECAP	Equal Channel Angular Pressing
ARB	Accumulative Roll Bonding
UFG	Ultra Fine Grain
AM	Additive Manufacturing
TPRE	Twin Plane Re-Entrant angle
CEC	Cyclic Extrusion-Compression
RCS	Repetitive Corrugation and Straightening
TE	Twist Extrusion
TCEC	Tube Cyclic Extrusion-Compression
ACEF	Accumulative Continuous Extrusion Forming
MDF	Multi-Directional Forging
SLM	Selective Laser Melting
FSW	Friction Stir Welding
NZ	Nugget Zone
SZ	Stir Zone
TMAZ	Thermo-Mechanical Affected Zone
HAZ	Heat Affected Zone
EBSD	Electron Backscatter Diffraction
TEM	Transmission Electron Microscopy
SEM	Scanning Electron Microscope
BSE	Backscatter Electron
OM	Optical Microscope
FCC	Face Cantered Cubic
HCP	Hexagonal Close Packed
GND	Geometrically Necessary Dislocation
GBS	Grain Boundary Sliding
CDRX	Continuous Dynamic Recrystallization
DDRX	Discontinuous Dynamic Recrystallization
GDRX	Geometric Dynamic Recrystallization
P/M	Powder Metallurgy
LAB	Low Angle Boundary
HAB	High Angle Boundary
FESEM	Field Emission Scanning Electron Microscopy
CS	Cast Sample
ES	Extrusion Sample
FS	FSP Sample
HS	HPT Sample
OES	Optical Emission Spectroscopy
XRF	X-Ray Fluorescence
kN	Kilo Newton
UTM	Universal Testing Machine
GL	Gage Length
GD	Gage Diameter
GW	Gage Width

TL	Total Length
GS	Grain Size
ET	Extrusion Temperature
P-Si	Primary Silicon
UTS	Ultimate Tensile Strength
TYS	Tensile Yield Strength
CYS	Compressive Yield Strength
UCS	Ultimate Compressive Strength
YSA	Yield Strength Asymmetry
CSR	Constant Initial Strain Rate
DSR	Differential Strain Rate
NA	Necking Area
EP	Eutectic Phase
PSN	Particle Simulated Nucleation
SRC	Static Recovery