

國立臺灣大學工學院材料科學與工程學系



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Bachelor's Thesis

多階段固溶熱處理對不同銅添加量 AA7075 鋁合金之
機械性質與顯微結構影響

Effects of Multi-Stage Solid Solution Heat Treatments on
the Mechanical Properties and Microstructures of
Different Cu Additions AA7075 Aluminum Alloys

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張子恩

中文摘要

本研究聚焦於比較不同銅含量之 AA7075 鋁合金經不同固溶熱處理後，機械性質與顯微結構組織之差異。探討採用不同溫度之多階段固溶熱處理，以掃描式電子顯微鏡 (Scanning Electron Microscope, SEM) 觀察鋁合金內部二次相之消融狀態，與傳統單一溫度之單階固溶熱處理之鋁合金差別，並且最佳化固溶製程以達更高機械性質，並且改良高銅含量鋁合金不佳之性質。

經差示掃描量熱法 (Differential Scanning Calorimetry, DSC) 及 X 光繞射分析儀 (X-ray diffraction, XRD) 分析可得，AA7075 鋁合金內主要二次相為 η 相，S 相，及 ω 相，熔融溫度分別為攝氏 481 度，493 度及不固溶。並且不同銅含量鋁合金會改變內部二次相之比率，本實驗則以此溫度為依據設計 475 °C 與 490 °C 之固溶熱處理，以最佳化固溶二次相並防止二次相初熔 (incipient melting)。

透過電子微探儀 (Electron Probe Micro Analyzer, EPMA) 可得知，在經超過二次相熔點之溫度直接固溶會造成初熔之孔洞，並且降低機械性質。以 490 °C 單階固溶會造成 η 相初融留下孔洞。而以低於二次相熔點過多溫度固溶則無法固溶完全，透過研究結果，以 475 °C 固溶會使 S 相殘留，並大幅降低機械性質。

研究發現針對二次相進行二階段固溶設計，分別以 475 °C 和 490 °C 熱處理，可解決二次相殘留與初熔問題，並大幅提升機械性質，並且分別以背向散射繞射儀 (Electron Backscattered Diffraction, EBSD) 和 穿透式電子顯微鏡 (Transmission Electron Microscope, TEM) 觀察發現單階段與二階段固溶在晶粒大小與析出物並無明顯差異，因此對鋁合金做多階段固溶可改善二次相所造成之問題。

最後為改善高銅含量鋁合金之機械性質，新增第三階段固溶熱處理 500 °C 階段，以消融殘餘 S 相，同時增加析出物分布密度，實際可增加高銅鋁合金之強度及

延伸率，透過 TRM 明場、HRTEM 及原子級影像分析可得在經三階段固溶熱處理之頂時效條件下，析出物較前階段有變小趨勢，並且三階段固溶對高銅鋁合金之改善多於低銅鋁合金。綜合實驗結果可得，雖然原本高銅含量鋁合金性質不佳，在經多階段固溶熱處理後，高銅鋁合金性質可達不低於低銅鋁合金程度。

關鍵字：AA7075 鋁合金；二次相；固溶熱處理；掃描式電子顯微鏡；初熔； η 析出物；HRTEM；HAADF；原子級影像

ABSTRACT



This study focuses on comparing the differences in mechanical properties and microstructure of AA7075 aluminum alloys with different copper contents after various solution heat treatments. By employing multi-stage solution heat treatments at different temperatures, the dissolution state of second phases within the aluminum alloy is observed using a Scanning Electron Microscope (SEM), differentiating it from aluminum alloys treated with traditional single temperature solution heat treatments. The process aims to optimize the time of solution treatment to achieve higher mechanical properties, and improve the poor properties of high copper content aluminum alloy.

Through Differential Scanning Calorimetry (DSC) and X-Ray Diffraction (XRD) analysis, it is found that the main second phases in AA7075 aluminum alloys are the η phase, S phase, and ω phase, with melting points of 481 °C, 493 °C, and insoluble, respectively. Different copper contents change the ratio of the second phases, especially S phases. This experiment designs solution heat treatments at 475 °C and 490 °C based on DSC results to optimize the dissolution of second phases and prevent incipient melting.

The Electron Probe Micro Analyzer (EPMA) reveals that solution treatment at temperatures exceeding the melting points of second phases causes incipient melting voids, reducing mechanical properties. Single-stage solution treatment at 490°C causes the η phase to incipiently melt, leaving voids. Solution treatment at temperatures significantly below the melting points of second phases fails to dissolve them completely. The study finds that solution treatment at 475 °C leaves the S phase behind, significantly reducing mechanical properties.

The study discovers that designing two-stage solution treatments for second phases, with heat treatments at 475 °C and 490 °C respectively, can solve the problems of residual

phases and incipient melting mentioned-above, significantly enhancing mechanical properties. Using Electron Backscattered Diffraction (EBSD) and Transmission Electron Microscope (TEM) find no significant differences in grain size and precipitates between single-stage and two-stage solution treatments. Thus, applying two-stage solution treatment to the aluminum alloy can improve problems caused by second phases.

To enhance the mechanical properties of high copper content aluminum alloys, a 500 °C third stage of solution heat treatment was introduced to eliminate the S phase remained and increase the density of precipitates. Finally, it effectively improves the strength and ductility of high copper aluminum alloys. Through analysis using TEM bright field, HRTEM, and atomic-resolution image, it was observed that, under the peak aging conditions of the three-stage solution heat treatment, the precipitates tend to be smaller compared to those in the single-stage and two-stage. Synthesizing the experimental results, it is observed that although the high-copper aluminum alloys initially exhibited inferior properties, through multi-stage solution heat treatments, the properties of high-copper aluminum alloys can achieve levels comparable to or better than those of low-copper aluminum alloys.

Keywords : AA7075 Aluminium alloys; second phases; solid solution treatment; SEM; incipient melting; η precipitates ; HRTEM ; HAADF ; atomic-resolution image

CONTENTS



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國立臺灣大學工學院材料科學與工程學系	i
誌謝	i
中文摘要	vii
ABSTRACT	ix
CONTENTS	xi
LIST OF FIGURES	xv
LIST OF TABLES	xxv
Chapter 1 General Introduction	1
Chapter 2 Literature Review	4
2.1 Introduction to Aluminum Alloy	4
2.1.1 Application of Aluminum Alloy	4
2.1.2 Types of Aluminum Alloy	5
2.1.3 Effect of Adding Different Elements	8
2.1.4 Heat treatment of Aluminium Alloys	10
2.2 Precipitation Hardening	16
2.2.1 Mechanism of Precipitation Hardening	16
2.2.2 Introduction to Precipitates in AA7075 Aluminum Alloy	18
2.2.3 GP zones	18
2.2.4 η' Precipitates	21
2.2.5 η Precipitates	22
2.2.6 Overview of Precipitates	27
2.3 Second Phases	28

2.3.1	Introduction to Second Phases	28
2.3.2	Introduction to Second Phases in AA7000 Series Aluminium Alloys	30
2.3.3	The problem of second phases	30
2.4	The Effect of Adding Cu on 7075 Aluminum Alloy.....	31
2.5	Mutli-Stage Solution Heat Treatment.....	35
Chapter 3	Experiment Procedures and Instructment	40
3.1	Experiment Procedures	40
3.1.1	Materials.....	40
3.1.2	Heat Treatment Designment.....	40
3.2	Experiment Instructment and Specimens Preparation.....	43
3.2.1	Hardness Test	43
3.2.2	Tensile Test.....	43
3.2.3	Differential Scanning Calorimetry (DSC).....	45
3.2.4	X-Ray Diffraction (XRD)	45
3.2.5	Scanning Electron Microscope (SEM).....	45
3.2.6	Energy dispersive X-ray spectroscopy (EDS).....	46
3.2.7	Electron Back-Scattered Diffraction (EBSD)	46
3.2.8	Electron Probe Micro Analyzer (EPMA).....	47
3.2.9	Transmission Electron Microscope (TEM).....	47
Chapter 4	Single-Stage and Two-stage solid solution treatment Experiment	
	Result.....	49
4.1	Introduction.....	49
4.2	Tensile Test	50
4.2.1	As-received Aluminium Alloys.....	50
4.2.2	Single-Stage Solution Treatment.....	51

4.2.3	Two-Stage Solution Treatment.....	57
4.2.4	Two-Stage Solution Treatment with 475 °C 0-15 h + 490 °C 3 h	61
4.2.5	Two-Stage Solution Treatment with 475 °C 0-15 h + 490 °C 6 h	65
4.2.6	Two-Stage Solution Treatment with 475 °C 0-15 h + 490 °C 9 h	69
4.2.7	Two-Stage Solution Treatment with 475 °C 0-15 h + 490 °C 12 h	73
4.2.8	Two-Stage Solution Treatment Organizing the Variation in 475 °C Solution Time with a Fixed 490 °C from 3 to 12 Hours	77
4.2.9	Two-Stage Solution Treatment Organizing the Variation in 490 °C Solution Time with a Fixed 475 °C from 3 to 15 Hours	79
4.2.10	Single-stage Solution Treatment for 475 °C	81
4.2.11	Optimized Solution Treatment Process and Comparison.....	84
4.3	DSC, XRD and Hardness Test Analysis	87
4.3.1	DSC Result.....	87
4.3.2	XRD Result	90
4.3.3	Hardness Test	93
4.4	SEI, BEI, EDS, and EPMA Analysis	95
4.4.1	As-received LCu	95
4.4.2	LCu after 475 °C 9 h Solution Treatment	99
4.4.3	LCu after 490 °C 3h Solution Treatment	103
4.4.4	LCu after 475 °C 9 h + 490 °C 3 h Solution Treatment.....	106
4.4.5	As-received HCu	112
4.4.6	HCu after 475 °C 3 h Solution Treatment.....	116
4.4.7	HCu after 490 °C 3 h solution treatment.....	120
4.4.8	HCu after 475 °C 3 h + 490 °C 3 h Solution Treatment	125
4.4.9	Fracture surface	129

4.5	EBSD	131
4.5.1	LCu.....	131
4.5.2	HCu	134
4.6	TEM.....	136
4.6.1	LCu after 490 °C 3 h Solution Treatment	136
4.6.2	LCu after 475 °C 9 h + 490 °C 3 h Solution Treatment.....	141
4.7	Discussion	145
Chapter 5	Three-Stage solid solution treatment	150
5.1	Introduction.....	150
5.2	Tensile test	151
5.3	DSC, XRD and EPMA Result	154
5.4	TEM Bright Field	158
5.5	HRTEM image.....	159
5.6	HAADF and Atomic-Resolution Image Analysis	161
5.7	Comparison between LCu and HCu	164
Chapter 6	Conclusion	166
Chapter 7	Future Work	168
Reference		169

LIST OF FIGURES



Chapter 2 - Literature Review

Figure 2- 1 Development Timeline Diagram of Aluminum Alloy Airplanes[10, 11].	4
Figure 2- 2 Proportional Usage of structural materials in Boeing Commercial Aircraft [9, 11].	5
Figure 2- 3 The distribution of strength and elongation relationships of commercial AA2000, AA6000, and AA7000 series aluminum alloys [16].	6
Figure 2- 4 The distribution of strength and elongation relationships for AA5000, AA6000, AA7000 series aluminum alloys [17].	7
Figure 2- 5 The strength distribution of different series of aluminum alloys [17].	7
Figure 2- 6 Types of aluminum alloys with different main added elements and their strengthening mechanisms [18].	8
Figure 2- 7 The solubility of different alloying elements at various temperatures [18].	9
Figure 2- 8 CCT (continuous cooling transformation) diagram of 7075 aluminium alloy [24].	11
Figure 2- 9 The first natural aging curve of an Al alloy after quenching at 520 °C, published by Alfred Wilm [27].	13
Figure 2- 10 Mechanical property changes of Al-7.55Zn-1.58Mg-1.39Cu-0.12Zr with different aging temperatures [31].	14
Figure 2- 11 TEM image of peak aging condition of Al-7.55Zn-1.58Mg-1.39Cu-0.12Zr (wt%) (a-c) 100 °C/36h ; (d-f) 120 °C/24h ; (g-i) 140 °C/14h ; (j-l) 160 °C/4h [31].	15
Figure 2- 12 Diagram Illustrating the Transition of Precipitation Hardening Mechanism When Precipitates Reach Critical Size.	17

Figure 2- 13 (a) Diagram of dislocation loops passing around precipitates when they are larger than the critical size (b) Diagram of shearing through precipitates when they are smaller than the critical size [36].	17
Figure 2- 14 Streak-like signals appeared on the Al-Cu alloy [41].	19
Figure 2- 15 HAADF-STEM image of nature aging Al-4.62Zn-0.9Mg (wt%) (a-c) 1 week ; (d-f) 1 month ; (g-i) 3 monthss ; (j-l) 14 months [44].	20
Figure 2- 16 Diagram illusating four variants of η' [53].	22
Figure 2- 17 Diagram illustrating orientation of η [61].	24
Figure 2- 18 HRTEM image of (a) GPII zone (b) η' (c) η FFT of (d) GPII zone (e) η' (f) η [53].	25
Figure 2- 19 (a) HR-TEM image of Al-6.25Zn-2.14Mg-2.23Cu-0.05Fe-0.03Si (wt.%) showing transformation of η' (region 2) to η_2 (region 1) (b)(c) FFT of region 1 and region 2 [53].	26
Figure 2- 20 Major precipitates in different stage of heat treatment.	27
Figure 2- 21 (a) (b) The 2219 aluminum alloy fractures on the surface due to second phase [72].	28
Figure 2- 22 Fracture sufaces of (a) 475 °C/8 h, (b) 475 °C/8 h + 485 °C/4 h and (c) 475 °C/8 h + 485 °C/4 h + 495 °C/2 h in Al-6.22Zn-2.11Mg-2.39Cu (wt.%) alloy.	29
Figure 2- 23 Fracture sufaces of (a) 490 °C/2 h, (b) 505 °C/1 h and (c) 515 °C/1 h in Al-6.22Zn-2.11Mg-2.39Cu (wt.%) alloy.	29
Figure 2- 24 Relation between Cu ratio and second phases in aluminium alloys [88].	31
Figure 2- 25 Tensile curves of aluminum alloys with different Cu contents [88].	32
Figure 2- 26 Mechanism properties of aluminum alloys with different Cu contents [95].	32

Figure 2- 27 Cu-containing second phases at the grain boundaries of different Cu content aluminum alloys (a)2.2% (b)1.8% (c)1.5% (d)1.3% (e)0.8% (Cu wt%) [88].

.....33

Figure 2- 28 Fracture surface morphology of aluminum alloys with different Cu contents under SEM (a, b) 0, (c, d) 1.0, (e, f) 2.0, and (g, h) 3.0 Cu wt% [88].34

Figure 2- 29 Mechanical properties of single-stage solution treatment on (a)(b) Al-7.58Zn-2.12Mg-1.66Cu (wt.%) [102].36

Figure 2- 30 Mechanical properties of multiple-stage solution treatment on (a) (b) Al-8.81Zn-2.23Mg-2.43Cu (wt.%) [111].38

Figure 2- 31 (a) (b)DSC result of Al-6.43Zn-2.21Mg-2.15Cu (wt.%) [110].39

Chapter 3 - Experiment Procedures and Instructment

Figure 3- 1 Illustration of heat treatment used for the experiment.41

Figure 3- 2 Vickers hardness as a function in LCu and HCu specimens of the time ageing at 120 ° C [119].42

Figure 3- 3 The standard of ASTM E8 sample [120].44

Figure 3- 4 The two tensile curve presents in the same tensile test.44

Chapter 4 - Single-Stage and Two-stage solid solution treatment Experiment Result

Figure 4-1 Stress-strain curve of as-received LCu and as-received HCu.51

Figure 4-2 Tensile curves of single-stage solution treatment for LCu on the condition peak ageing at 120 °C.53

Figure 4-3 Tensile curves of single-stage solution treatment for HCu on the condition peak ageing at 120 °C.54

Figure 4-4 Mechanical properties of single-stage solution treatment for LCu and HCu on the condition peak ageing at 120 °C.55

Figure 4-5 Tensile curves of two-stage and single-stage solution treatment for LCu on the condition peak ageing at 120 °C.....	58
Figure 4-6 Mechanical properties of two-stage and single-stage solution treatment for LCu on the condition peak ageing at 120 °C.....	58
Figure 4-7 Tensile curves of two-stage and single-stage solution treatment for HCu on the condition peak ageing at 120 °C.....	59
Figure 4-8 Mechanical properties of two-stage and single-stage solution treatment for HCu on the condition peak ageing at 120 °C.	59
Figure 4-9 Tensile curves of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.....	62
Figure 4-10 Mechanical properties of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.....	63
Figure 4-11 Tensile curves of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.....	66
Figure 4-12 Mechanical properties of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.....	67
Figure 4-13 Tensile curves of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.....	70
Figure 4-14 Mechanical properties of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.....	71
Figure 4-15 Tensile curves of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.....	74
Figure 4-16 Mechanical properties of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.....	75
Figure 4-17 Mechanical properties of two-stage solution treatment with 475 °C as the	

variable for LCu and HCu on the condition peak ageing at 120 °C.....	78
Figure 4-18 Mechanical properties of two-stage solution treatment with 490 °C as the	
variable for LCu and HCu on the condition peak ageing at 120 °C.....	80
Figure 4-19 Tensile curves of single-stage solution treatment with 475 °C for LCu and	
HCu on the condition peak ageing at 120 °C.	82
Figure 4-20 Tensile curves of two-stage and single-stage solution treatment for LCu and	
HCu on the condition peak ageing at 120 °C.	85
Figure 4-21 DSC curves of (a) as-received (b) first stage solution treatment (c) second	
stage solution treatment (d) two-stage solution treatment on LCu.....	88
Figure 4-22 DSC curves of (a) as-received (b) first stage solution treatment (c) second	
stage solution treatment (d) two-stage solution treatment on HCu.	89
Figure 4-23 XRD curves of (a) as-received (b) first stage solution treatment (c) second	
stage solution treatment (d) two-stage solution treatment on LCu.....	92
Figure 4-24 XRD curves of (a) as-received (b) first stage solution treatment (c) second	
stage solution treatment (d) two-stage solution treatment on HCu.	92
Figure 4-25 Hardness distribution of as-received/ first stage solution treatment/ second	
stage solution treatment/ two-stage solution treatment on LCu with peak	
aging.	94
Figure 4-26 Hardness distribution of as-received/ first stage solution treatment/ second	
stage solution treatment/ two-stage solution treatment on HCu with peak	
aging.	94
Figure 4- 27 SEI and BEI of LCu as-received with EDS(red : η ; yellow : S; green, blue :	
ω). 96	
Figure 4-28 EPMA mapping of LCu as-received (brown : η ; orange : S; yellow : ω)...97	
Figure 4-29 WDS elemental quantification of LCu as-received.	98

Figure 4-30 SEI and BEI of LCu after 475°C 9h solution treatment with peak aging with EDS.....	99
Figure 4-31 EPMA mapping of LCu after 475 °C 9h solution treatment with peak aging (orange : S; yellow : ω ; green : Mg_2Si holes).....	101
Figure 4-32 WDS elemental quantification of LCu after 475 °C 9h solution treatment with peak aging.....	102
Figure 4-33 SEI and BEI of LCu after 490 °C 3h solution treatment with peak aging with EDS.....	103
Figure 4-34 EPMA mapping of LCu after 490 °C 3h solution treatment with peak aging (yellow : ω ; green : Mg_2Si ; red : incipient melting).	104
Figure 4-35 WDS elemental quantification of LCu after 490 °C 3h solution treatment with peak aging.....	105
Figure 4-36 SEI and BEI of LCu after 475 °C 9 h+490 °C 3 h solution treatment with peak aging with EDS	106
Figure 4-37 SEI and BEI of LCu after 490 °C 3 h and 475 °C 9 h+490 °C 3 h solution treatment with peak aging on condition of mechanical polishing.	107
Figure 4-38 SEI and BEI of LCu after 490 °C 3 h and 475 °C 9 h+490 °C 3 h solution treatment with peak aging on condition of electrolytic polishing.	108
Figure 4-39 EPMA mapping of LCu after 475 °C 9h + 490 °C 3h solution treatment with peak aging (yellow : ω ; green : Mg_2Si).	110
Figure 4-40 WDS elemental quantification of LCu after 475 °C 9h + 490 °C 3h solution treatment with peak aging.....	111
Figure 4-41 SEI and BEI of HCu as-received with EDS(red : η ; yellow : S; green, blue : ω).	113
Figure 4-42 EPMA mapping of HCu as-received (brown : η ; orange : S; yellow : ω).	

.....	114
Figure 4-43 WDS elemental quantification of as-received HCu.....	115
Figure 4-44 SEI and BEI of HCu after 475 °C 3 h solution treatment with peak aging with EDS (red ,yellow : S; green : ω).	116
Figure 4-45 EPMA mapping of HCu after 475 °C 3 h solution treatment with peak aging (orange : S; yellow : ω ; green : Mg_2Si holes).	118
Figure 4-46 WDS elemental quantification of HCu after 475 °C 3 h solution treatment with peak aging.	119
Figure 4-47 SEI and BEI of HCu after 490 °C 3 h solution treatment with peak aging with EDS (red ,yellow : ω ; green : S).	121
Figure 4-48 EPMA mapping of HCu after 490 °C 3 h solution treatment with peak aging (orange : S; yellow : ω ; green : Mg_2Si ; red : incipient melting).	122
Figure 4-49 WDS elemental quantification of HCu after 490 °C 3 h solution treatment with peak aging place 1.	123
Figure 4-50 WDS elemental quantification of HCu after 490°C 3h solution treatment with peak aging place 2.	124
Figure 4-51 SEI and BEI of HCu after 475 °C 3 h + 490° C 3 h solution treatment with peak aging with EDS (red ,yellow ,green : S).	125
Figure 4-52 SEI and BEI of LCu after 490 °C 3 h and 475 °C 9 h+490 °C 3 h solution treatment with peak aginon condition of mechanical polishing.	126
Figure 4-53 EPMA mapping of HCu after 475 °C 3 h + 490 °C 3 h solution treatment with peak aging (yellow : ω ; green : Mg_2Si).	127
Figure 4-54 WDS elemental quantification of HCu after 475 °C 3 h + 490 °C 3 h solution treatment with peak aging.....	128
Figure 4-55 Fracture surfaces of LCu after (a) 475 °C 9 h (b) 490 °C 3 h (c) 475 °C 9 h +	

475 °C 3 h solution treatment with peak aging.	130
Figure 4-56 Fracture surfaces of HCu after (a) 475 °C 3 h (b) 490 °C 3 h (c) 475 °C 3 h + 490 °C 3 h solution treatment with peak aging.	131
Figure 4-57 EBSD analysis of LCu 490 °C 3 h after solution treatment with peak aging. 132	
Figure 4-58 EBSD analysis of LCu 475 °C 9 h + 490 °C 3 h after solution treatment with peak aging.	132
Figure 4-59 EBSD analysis of LCu 490 °C 12 h after solution treatment with peak aging. 133	
Figure 4-60 EBSD analysis of LCu 490 °C 24 h after solution treatment with peak aging. 133	
Figure 4-61 EBSD analysis of HCu 490 °C 3 h after solution treatment with peak aging. 134	
Figure 4-62 EBSD analysis of HCu 490 °C 6 h after solution treatment with peak aging. 135	
Figure 4-63 EBSD analysis of HCu 475 °C 3 h + 490 °C 3 h after solution treatment with peak aging.	135
Figure 4-64 TEM-BF images of LCu after 490 °C 3 h Solution Treatment with peak aging. 137	
Figure 4-65 HRTEM image of η' precipitate in 490 °C 3 h Solution Treatment with peak aging.	138
Figure 4-66 HRTEM image of η precipitate in 490 °C 3 h Solution Treatment with peak aging.	139
Figure 4-67 HRTEM image of η' precipitate in 490 °C 3 h Solution Treatment with peak aging.	140

Figure 4-68 TEM-BF images of LCu after 475 °C 9 h + 490 °C 3 h solution treatment with peak aging.....	142
Figure 4-69 HRTEM image of η' and η precipitate after 475 °C 9 h + 490 °C 3 h Solution Treatment with peak aging.	143
Figure 4-70 HRTEM image of η' and η precipitate after 475 °C 9 h + 490 °C 3 h Solution Treatment with peak aging.	144
Figure 4-71 Summarize of two-stage solution treatment.	149

Chapter 5 - Three-Stage solid solution treatment

Figure 5-1 Illustration of three-stage solution treatment used for the experiment.	151
Figure 5-2 Tensile curves of three-stage, two-stage, and single-stage solution treatment for LCu and HCu on the condition peak ageing at 120 °C.	152
Figure 5-3 DSC curves of three-stage solution treatment on HCu.	154
Figure 5-4 XRD curves of three-stage solution treatment on HCu.	155
Figure 5-5 EPMA mapping of HCu after 475 °C 2 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging (yellow : ω ; green : Mg_2Si).	156
Figure 5-6 WDS elemental quantification of HCu after 475 °C 3 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging.	157
Figure 5-7 TEM-BF images of HCu after 475 °C 2 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging.	158
Figure 5-8 HRTEM image of precipitate in HCu after 475 °C 2 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging.	160
Figure 5-9 HAADF and EDS mapping of HCu after 475 °C 2 h + 490 °C 3 h + 500 °C 1 h Solution Treatment with peak aging.	162
Figure 5-10 Atomic-resolution image and FFT diffraction of HCu after 475 °C 2 h + 490	

°C 3 h + 500 °C 1 h solution treatment with peak aging.....	163
---	-----



LIST OF TABLES



Chapter 2 - Literature Review

Table 2- 1	Characteristics of Different Series of Wrought Aluminum Alloys [1, 4, 10, 13-15].....	6
Table 2- 2	The effect of adding different alloying elements on AA7075 [1, 4, 14, 20].10	
Table 2- 3	Common Heat Treatment symbols [25, 26].....	12
Table 2- 4	Four variants of η' precipitates in aluminium alloys [53].	21
Table 2- 5	Orientation relationships and morphologies of η precipitates in aluminum alloys [61].	23
Table 2- 6	Main second phases in AA7000 series aluminium alloys [81-87].	30

Chapter 3 - Experiment Procedures and Instructment

Table 3- 1	Chemical composition of aluminum alloys used in the study	40
------------	---	----

Chapter 4 - Single-Stage and Two-stage solid solution treatment Experiment Result

Table 4-1	Mechanical properties of as-received LCu and as-received HCu.	51
Table 4-2	YS of single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.	56
Table 4-3	UTS of single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.	56
Table 4-4	EL of single-stage solution treatment for LCu and HCu with peak ageing at 120 °C	57
Table 4-5	YS of two-stage and single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.	60
Table 4-6	UTS of two-stage and single-stage solution treatment for LCu and HCu with	

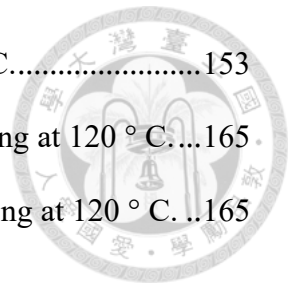
peak ageing at 120 °C.....	60
Table 4-7 EL of two-stage and single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.	60
Table 4-8 YS of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.	64
Table 4-9 UTS of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.....	64
Table 4-10 EL of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.....	64
Table 4-11 YS of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.	68
Table 4-12 UTS of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.....	68
Table 4-13 EL of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.	68
Table 4-14 YS of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.	72
Table 4-15 UTS of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.....	72
Table 4-16 EL of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.	72
Table 4-17 YS of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.....	76
Table 4-18 UTS of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.....	76

Table 4-19	EL of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.....	76
Table 4-20	YS of single-stage solution treatment with 475 °C for LCu and HCu on the condition peak ageing at 120 °C.....	83
Table 4-21	UTS of single-stage solution treatment with 475 °C for LCu and HCu on the condition peak ageing at 120 °C.....	83
Table 4-22	EL of single-stage solution treatment with 475 °C for LCu and HCu on the condition peak ageing at 120 °C.....	83
Table 4-23	Mechanical properties of two-stage and single-stage solution treatment for LCu on the condition peak ageing at 120 °C.....	86
Table 4-24	Mechanical properties of two-stage and single-stage solution treatment for HCu on the condition peak ageing at 120 ° C.	86
Table 4-25	Incipient melting peaks on Figure 4-21(a).....	89
Table 4-26	Hardness comparsion of different solution treatment for LCu on peak ageing.	93
Table 4-27	Hardness comparsion of different solution treatment for HCu on peak ageing.	93
Table 4-28	Average grain size of LCu summarized from EBSD.....	133
Table 4-29	Average grain size of HCu summarized from EBSD.	135
Table 4-30	Analyzed result of precipitates in LCu of single-stage and two-stage solution treatmrent with peak aging.	142

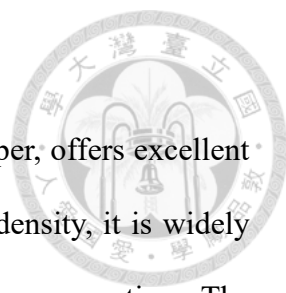
Chapter 5 - Three-Stage solid solution treatment

Table 5-1	Mechanical properties of three-stage, two-stage, and single-stage solution treatment for LCu on the condition peak ageing at 120 °C.	153
Table 5-2	Mechanical properties of three-stage, two-stage, and single-stage solution	

	treatment for HCu on the condition peak ageing at 120 °C.....	153
Table 5-3	Mechanical properties of LCu on the condition peak ageing at 120 ° C....	165
Table 5-4	Mechanical properties of HCu on the condition peak ageing at 120 ° C. .	165

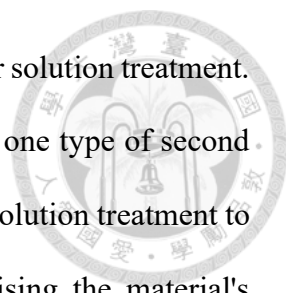


Chapter 1 General Introduction



The 7075 aluminum alloy, containing magnesium, zinc, and copper, offers excellent strength and corrosion resistance. Due to its high strength and low density, it is widely used in the aerospace and automotive industries to lower fuel consumption. The strengthening mechanism of the AA7075 alloy is primarily through precipitation strengthening. Solution heat treatment of the wrought material dissolves alloying elements such as zinc, magnesium, and copper back into the matrix, followed by aging to precipitate nanoscale particles, achieving high mechanical properties. Past researchers believed that increasing the copper content in aluminum alloys could enhance the mechanical properties of the alloys. However, the aluminum alloy contains various micrometer-scale second phases after forging. Increasing the copper content can lead to an increase in copper-containing second phases, such as the S phase, in the alloy. Unlike nanoscale precipitates, most second phases decrease the strength and ductility of the aluminum alloy, and dissolving them is necessary to achieve better mechanical properties. Previous solution heat treatments often maintained a single temperature to dissolve these second phases back into the matrix.

Nevertheless, melting points of second phases vary from each other, taking a single-step solution heat treatment presents two main drawbacks. Firstly, excessively high solution temperatures can directly melt some second phases, causing incipient melting, which research has shown to reduce mechanical properties. Thus, avoiding incipient melting is a crucial aspect of heat treatment for aluminum alloys. Secondly, if the solution temperature is too low for the melting points of second phases, or the duration of the time is insufficient, the alloying elements from second phases that strengthen through precipitation cannot effectively dissolve back into the aluminum matrix, resulting in a diminished precipitation strengthening effect. If a material contains only one type of



second phase, it would be feasible to select an optimal temperature for solution treatment. However, in copper-containing aluminum alloys, there is more than one type of second phase present. This complexity requires a more tailored approach to solution treatment to effectively dissolve all types of second phases without compromising the material's properties. To prevent incipient melting and ensure the effective dissolution of second phases, this experiment plans to design a multi-stage heat treatment with varied solution temperatures, allowing the second phases with different melting points to sequentially dissolve back into the aluminum alloy matrix.

To reach higher mechanical properties, past research has studied multi-stage solution heat treatments to improve the mechanical properties of materials, but previous literature often involved excessively long solution times, leading to insignificant benefits. Extending the solution treatment duration indefinitely is impractical in application due to the associated costs. This approach not only increases energy consumption but also affects productivity by occupying equipment for longer periods, ultimately leading to higher production costs. Moreover, if the issues regarding second phases were effectively resolved, both the elongation and strength of the material should see significant improvements; nonetheless, prior studies have not extensively covered elongation. In the automotive industry, 7000 series aluminum alloys are not used because they have insufficient ductility, so it is necessary to enhance ductility. Therefore, this study aims to optimize the solution time, considering both the enhancement of material strength and elongation.

This thesis is divided into seven chapters: The first chapter provides a general introduction to aluminum alloys, and motivation of this research. The second chapter offers a detailed review of previous studies on aluminum alloys, including the evolution of precipitates and the impact of second phases and incipient melting. The third chapter

describes the instruments used in this experiment and the preparation methods for samples. In the fourth chapter, the solution process is explored as a variable to investigate the differences in internal microstructure and mechanical properties, covering from surface particles and grain size to the influence of precipitates. The fifth chapter describes the effect of adding third-stage solution treatment. The sixth chapter systematically organizes the conclusions of this experiment. The seventh chapter discusses potential future research directions based on this experiment, and the finally lists the references for this thesis.

Chapter 2 Literature Review

2.1 Introduction to Aluminum Alloy

2.1.1 Application of Aluminum Alloy

Aluminum, being the third most abundant element in the earth, is one of the most widely used metals [1] due to the high strength and low density of its alloys [2, 3]. However, because of its poor mechanical properties of pure aluminum [4], the history of aluminum alloy applications does not as long as other copper and iron alloys. Until the early 20th century, German scientists developed the first practically applicable hardened aluminum alloy naming "duralumin," [5] which is the beginning of human use of aluminum alloys. Since then, during World Wars I and II [6], over ninety percent of aluminum was employed in warplanes. Due to its lightweight and high-strength characteristics, aluminum alloys still make up a significant proportion of materials in the aerospace field [7-9], shown in Figure 2-1 and Figure 2-2, demonstrating the importance of aluminum alloys.

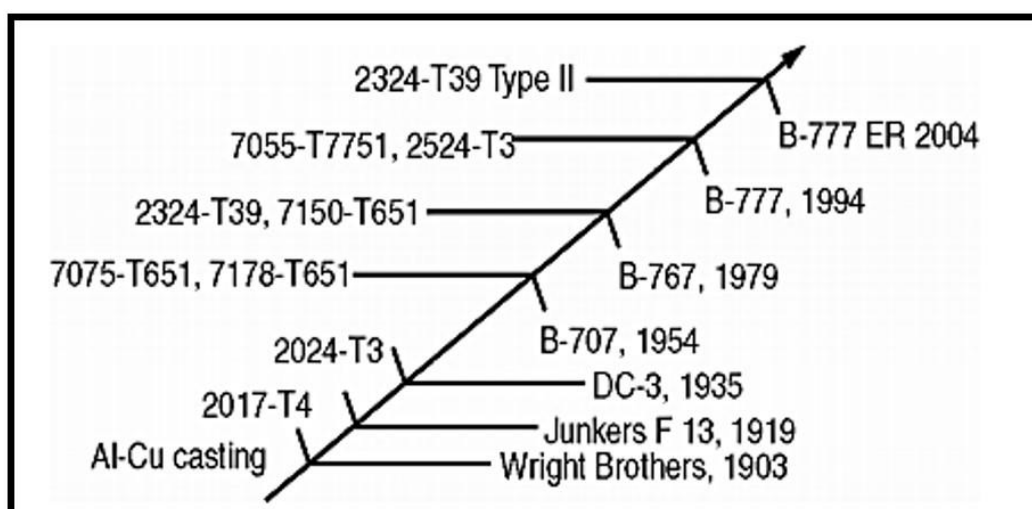


Figure 2- 1 Development Timeline Diagram of Aluminum Alloy Airplanes [10, 11].

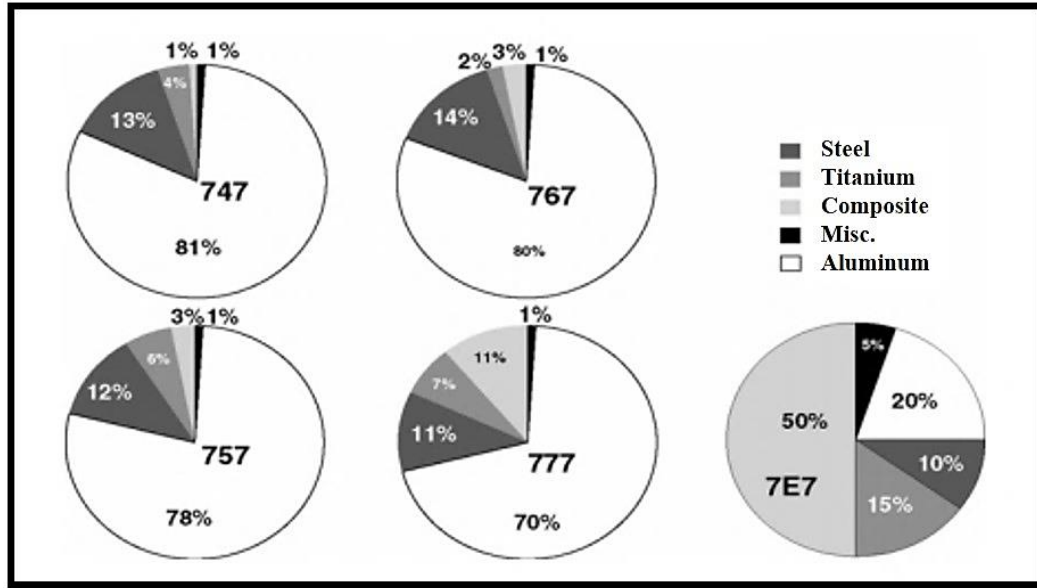


Figure 2- 2 Proportional Usage of structural materials in Boeing Commercial Aircraft [9, 11].

2.1.2 Types of Aluminum Alloy

Owing to the low strength of pure aluminum, scientists had tried to add various alloying elements [12, 13] to the aluminum matrix in order to improve its mechanical properties throughout the history of aluminum alloy development. As a result, most of the aluminum used today exist are alloys, which can be further divided into wrought and cast types. Based on the different types of elements added, wrought and cast aluminum alloys are named with four-digit and three-digit numbers, respectively, where the first digit represents the type of alloying element added. The major industrial application is wrought aluminum alloys, which can be further divided into heat-treatable alloys, whose main strengthening mechanism is precipitation hardening, represented by AA2xxx, AA4xxx, AA6xxx, AA7xxx series, and non-heat-treatable alloys, whose main strengthening mechanisms are work hardening and grain refinement, represented by AA1xxx, AA3xxx, AA5xxx series. Table 2-1 summerize the characteristic of different series aluminum alloy. The naming follows the classification by the American Aluminum Association, and the alloy used in this study is the Al-Zn-Mg-Cu, AA7075 heat-treatable aluminum alloy. In

Figures 2-3 and 2-4, the strength of different series of aluminum alloys can be observed, with the 7xxx series having good strength but lower ductility, thus improving the ductility of the 7075 aluminum alloy is the focus of this experiment.

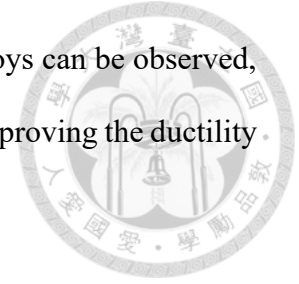


Table 2- 1 Characteristics of Different Series of Wrought Aluminum Alloys [1, 4, 10, 13-15]

Aluminum Series	Composition	Characteristics	Heat treatable
AA1xxx	Al > 99.0 wt%	High electrical and thermal conductivity	
AA2xxx	Al-Cu	High strength Low corrosion resistance	V
AA3xxx	Al-Mn	Good formability	
AA4xxx	Al-Si	Good wear resistance	V
AA5xxx	Al-Mg	Good corrosion resistance and weldability	
AA6xxx	Al-Mg-Si	Good formability	V
AA7xxx	Al-Zn-Mg-Cu	High strength and fatigue resistance	V
AA8xxx	mixed	Miscellaneous	

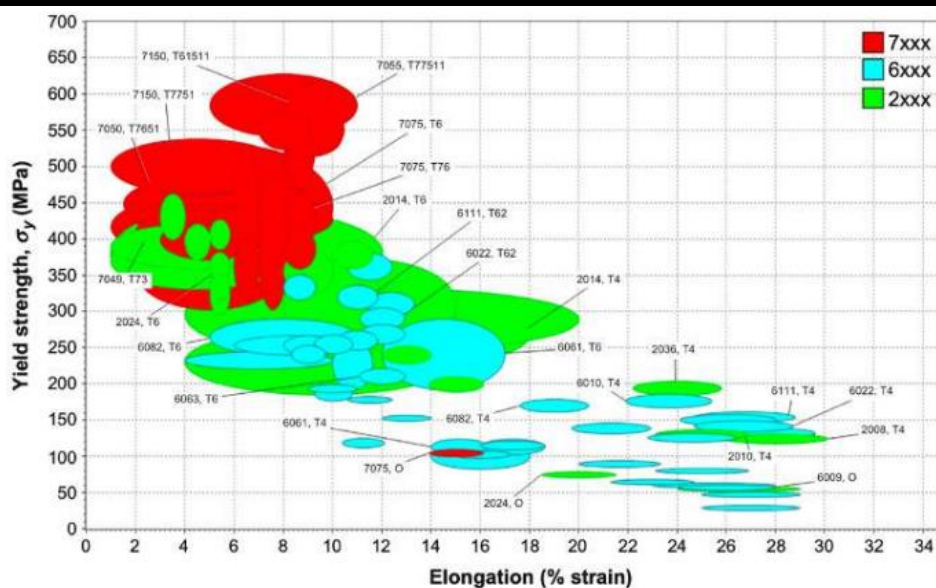


Figure 2- 3 The distribution of strength and elongation relationships of commercial AA2000, AA6000, and AA7000 series aluminum alloys [16].

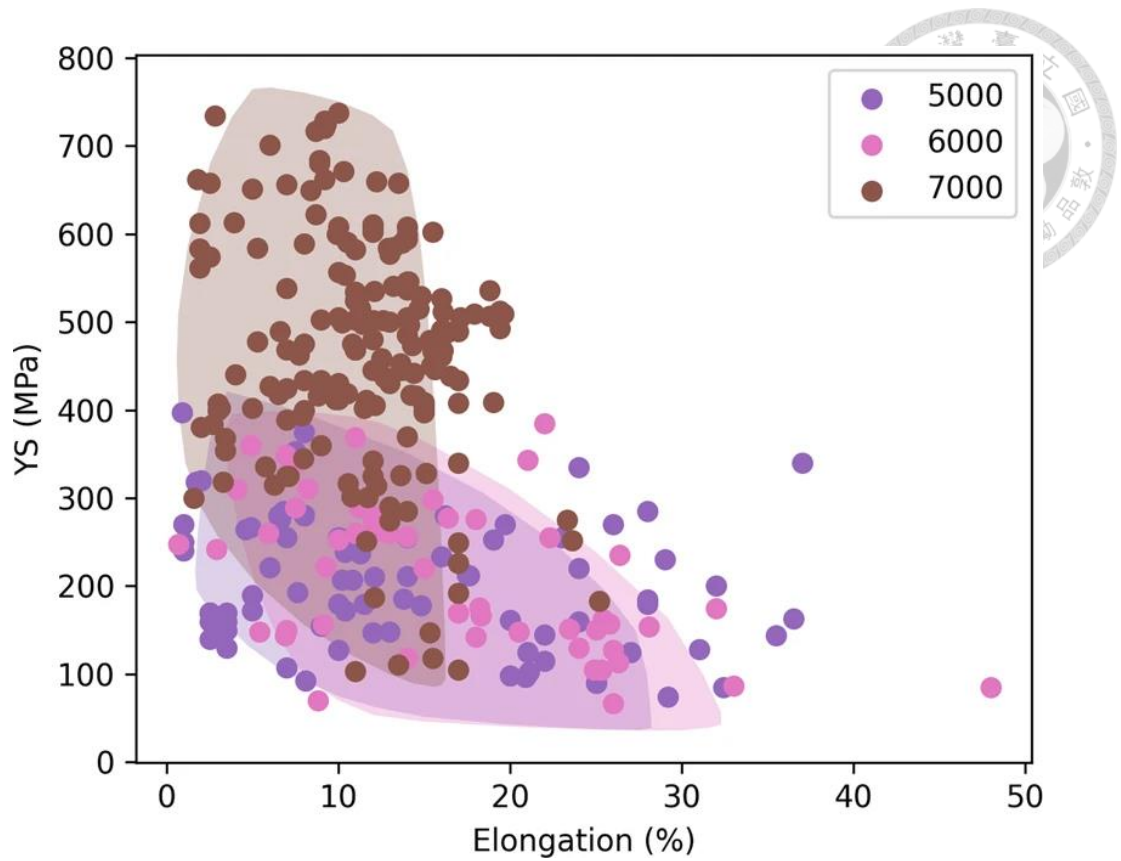


Figure 2- 4 The distribution of strength and elongation relationships for AA5000, AA6000, AA7000 series aluminum alloys [17].

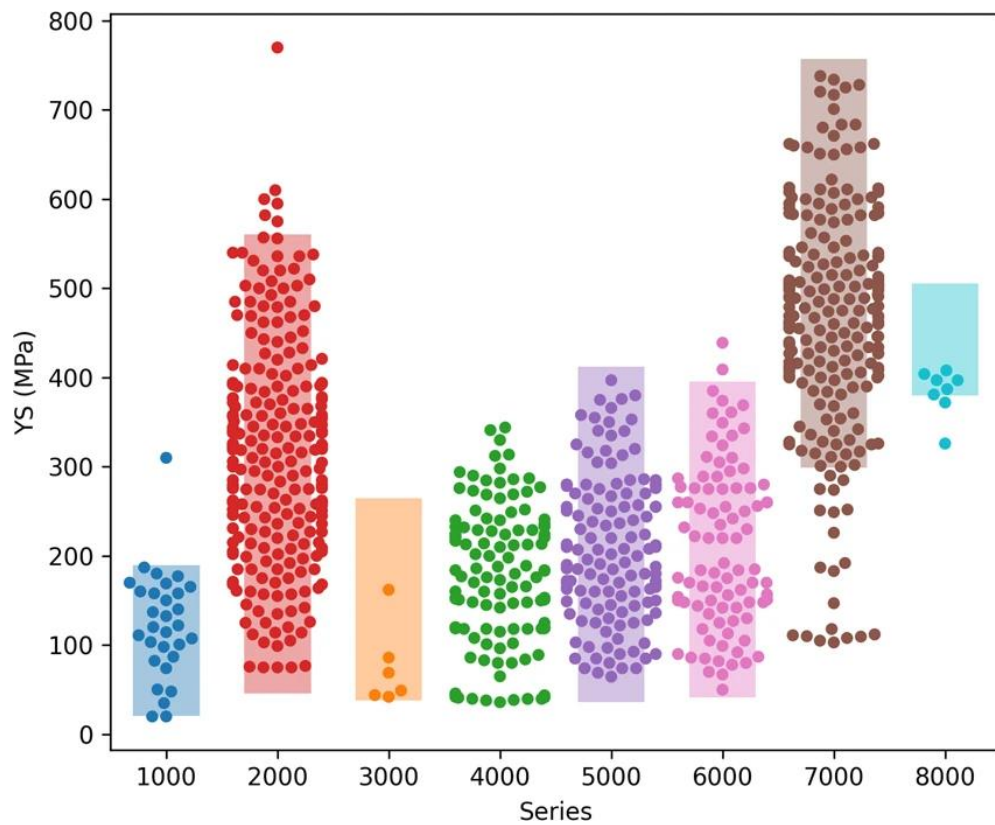


Figure 2- 5 The strength distribution of different series of aluminum alloys [17].

2.1.3 Effect of Adding Different Elements

During the development of aluminum alloy history, a large number of elements were added to aluminium matrix to improve mechanical properties [18]. The incorporation of different elements affects various strengthening mechanisms, resulting in different classifications, shown in Figure 2-6, such as Al-Cu, Al-Zn-Mg, Al-Si, Al-Mn alloys, which correspond to the different series of aluminum alloys mentioned previously.

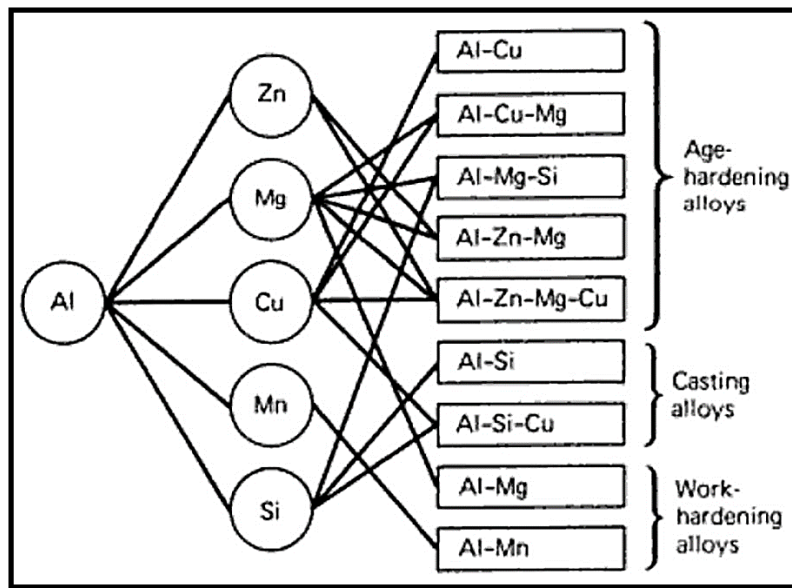


Figure 2- 6 Types of aluminum alloys with different main added elements and their strengthening mechanisms [18].

The AA7075 aluminum alloy used in this experiment added elements such as Zn, Mg, Cu. Moreover, the AA7000 series aluminum alloy is a heat-treatable type. For heat treatment, the alloying elements must be dissolved back into the matrix to achieve precipitation strengthening. The Figure 2-7 shows the solubility of different alloying elements at various temperatures.

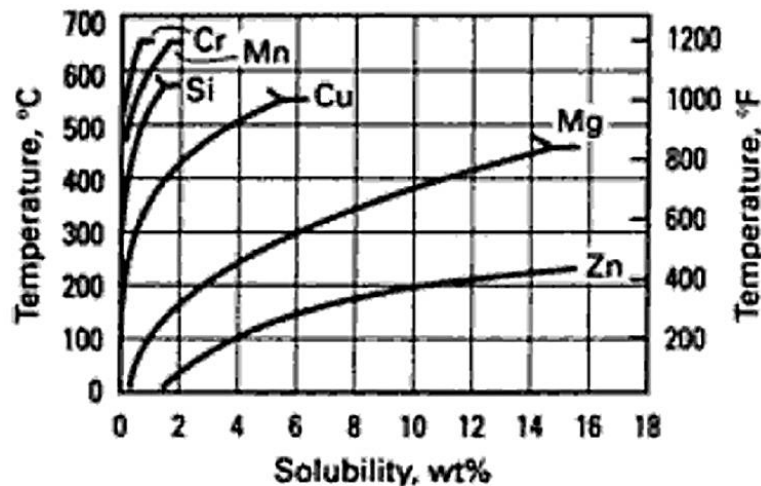


Figure 2- 7 The solubility of different alloying elements at various temperatures [18].

On top of that, within the AA7075 aluminum alloy, adding different alloying elements has various effects on precipitation strengthening. The addition of Zn and Mg promotes the formation of the precipitate η (MgZn_2), its precursor η' , and GP zones, which are the main strengthening precipitates, enhancing the strengthening effect[4, 14]. The inclusion of Cu strengthens the yield strength of aluminum. Studies have shown that a low copper content can improve strength and elongation. Cu facilitates the nucleation of alloy elements forming GP zones and η' , and prevents the transformation of metastable phases, such as η' , into stable phases [12, 14]. However, an excessive amount of copper can lead to the formation of second phases [19], reducing elongation, which will be detailed later. Other second alloying elements, such as Mn, Cr, Si, are summarized in the Table 2-2 below.

Table 2- 2 The effect of adding different alloying elements on AA7075 [1, 4, 14, 20].

Element	Effect
Zn	1. Promotes the formation of Zn clusters and GP zones, facilitating the generation of the strengthening phase η'. 2. High Zn content addition detrimentally affects SCC resistance.
Mg	1. Promotes the formation of Mg clusters and GP zones, facilitating the generation of the strengthening phase η'. 2. High Mg content addition detrimentally affects SCC resistance.
Cu	1. Increases the stability of η'. 2. Accelerates the rate of precipitation hardening.
Zr	1. Raises the recrystallization temperature to inhibit recrystallization, aiding in grain refinement. 2. Formation of Al_3Zr assists in grain refinement, and providing nucleation points for η'.
Si	1. Improves fluidity and casting properties. 2. Reduces quench sensitivity.
Cr	1. Increases corrosion resistance and strength. 2. Formation of $\text{Al}_{12}\text{Mg}_2\text{Cr}$ dispersoids worsens quench sensitivity.
Mn	1. Inhibits recrystallization, aiding in grain refinement and work hardening. 2. Softens the boundaries of iron-based second phases.

2.1.4 Heat treatment of Aluminium Alloys

In the strengthening of heat-treatable aluminum alloys, the method of heat treatment is a key factor for mechanical properties because precipitation hardening is applying to strengthen the aluminum alloy, making solution heat treatment, quenching, and aging heat treatment all important. This experiment focuses on changing the solution factors, which will be introduced in detail later. Regarding quenching, metal heat treatment is not only influenced by the heating process but also by the rate of cooling, which is another variable affecting mechanical properties [21]. After solution treatment, quenching can be classified according to the rate of cooling as water quenching, oil quenching, air cooling,

and furnace cooling. Generally speaking, as the rate of quenching cooling increases, the concentration of vacancies rises, meaning more nucleation points, leading to an increase in strength. The quenching method used in this experiment is water quenching, directly cooling from the solution temperature to room temperature to achieve higher mechanical properties. Additionally, in aluminum alloys, 400 °C is a favorable growth temperature for second phases [22, 23]. If the cooling rate is too slow, too many second phases will grow at 400 °C, reducing the material's strength and ductility. Figure 2-8 is the Continuous Cooling Transformation diagram (CCT) for 7075 aluminum alloy.

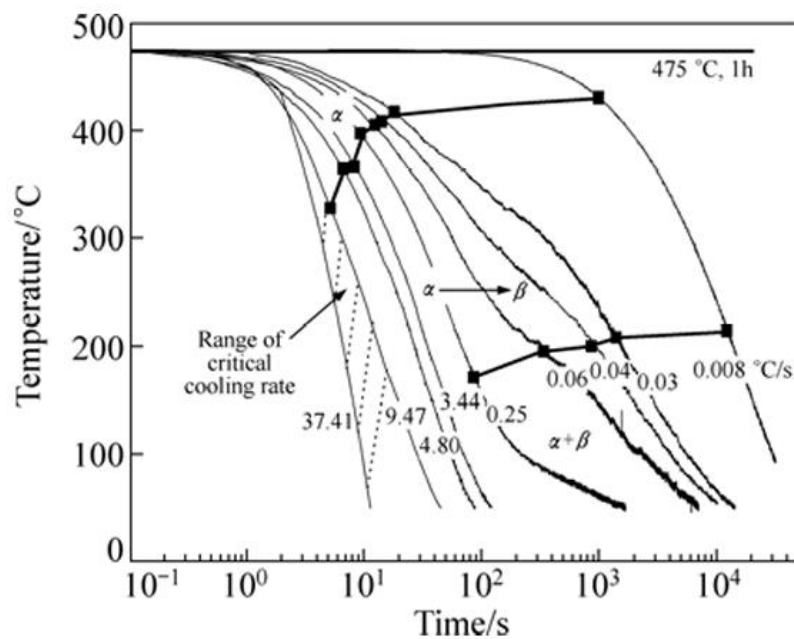


Figure 2- 8 CCT (continuous cooling transformation) diagram of 7075 aluminium alloy [24].

In addition to quenching, aging heat treatment is the most common method to change mechanical properties of metals. Table 2-3 is the symbol reference table for aging heat treatments.

Table 2- 3 Common Heat Treatment symbols [25, 26]

Term	Heat Treatment Method
T1	Cooled from an elevated temperature shaping process and naturally aged.
T2	Cooled from an elevated temperature shaping process, cold worked, and naturally aged.
T3	Solution heat treated, cold worked, and naturally aged.
T4	Solution heat treated and naturally aged.
T5	Cooled from an elevated temperature shaping process, and artificially aged.
T6	Solution heat treated and artificially aged.
T7	Solution heat treated and overaged.
T8	Solution heat treated, cold worked, and artificially aged.
T9	Solution heat treated, artificially aged, and cold worked.
T10	Cooled from an elevated temperature shaping process, cold worked, and artificially aged

In the Table 2-3, T4 denotes natural aging, originating from scientist Alfred Wilm in 1906, who mimicked the quenching heat treatment used in steel. He used an Al-3.5 to 5.5Cu-1Mg-1Mn (wt%) alloy solution followed by quenching, but the strength achieved was unsatisfactory, only about 70HV. However, as shown in Figure 2-9, the strength of the aluminum alloy showed a trend of increasing over time when left at room temperature [27]. This represents the earliest form of T4 heat treatment for aluminum alloys, where precipitates slowly form and grow at room temperature, ultimately achieving precipitation strengthening effects. Nonetheless, T4 heat treatment requires a long period of time and is overly time-consuming, so aluminum alloys often undergo T6 artificial aging [28] to perform precipitation hardening heat treatment.

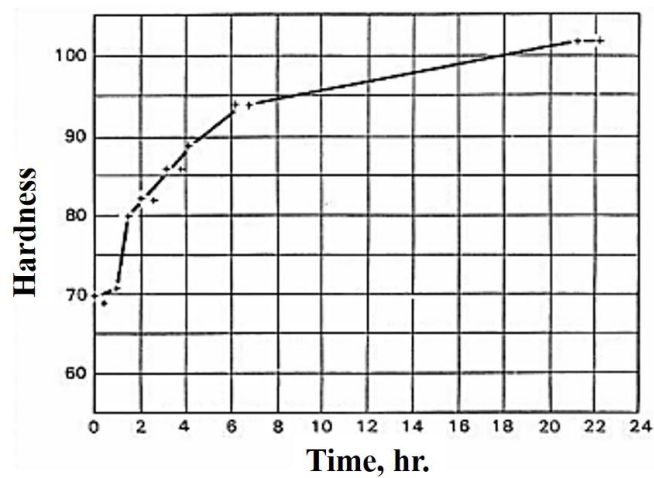


Figure 2- 9 The first natural aging curve of an Al alloy after quenching at 520 °C, published by Alfred Wilm [27].

Due to the time-consuming nature of T4 natural aging, in industrial processing, the aging temperature is increased to T6 for artificial aging. T6 artificial aging is the most commonly used heat treatment method. Natural aging involves leaving the material at room temperature after quenching for the precipitates to naturally nucleate and grow, whereas T6 artificial aging involves maintaining the temperature at 95-200 °C for 8-48 hours after quenching until the optimum strength is achieved, known as peak aging. As the environmental temperature increases, the diffusion rate of solute atoms rises, making it easier to overcome the free energy barrier for cluster formation, thereby forming GP zones, which then transform into metastable phases such as η' , ultimately leading to the formation of η for precipitation strengthening effects. With the increase in aging temperature, the time required to reach peak aging also shortens, as the increased temperature accelerates the diffusion rate of solute atoms, reducing time costs. As the aging time progresses, the material's strength generally increases, peaks, and then decreases, while ductility shows a trend of decreasing and then leveling off [29-31].

Figure 2-10 shows the mechanical property changes of Al-7.55Zn-1.58Mg-1.39Cu-0.12Zr with different aging temperatures. From the figure, it is evident that there is no significant peak aging before 120 °C, and the strength noticeably decreases after 140 °C, with peak aging strength decreasing as the aging temperature rises. This is because the mechanism of precipitation strengthening is closely related to the size and distribution of precipitates, which will be discussed in detail in the next section. The attached Figure 2-11 is a TEM photo of peak aging, showing coarsening of the precipitates as the aging temperature increases.

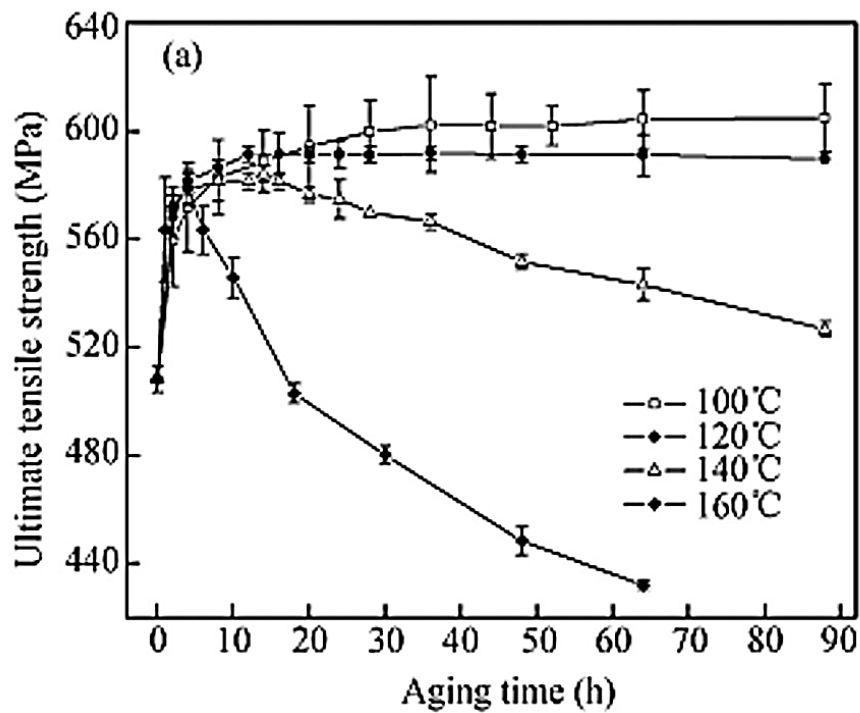


Figure 2- 10 Mechanical property changes of Al-7.55Zn-1.58Mg-1.39Cu-0.12Zr with different aging temperatures [31].

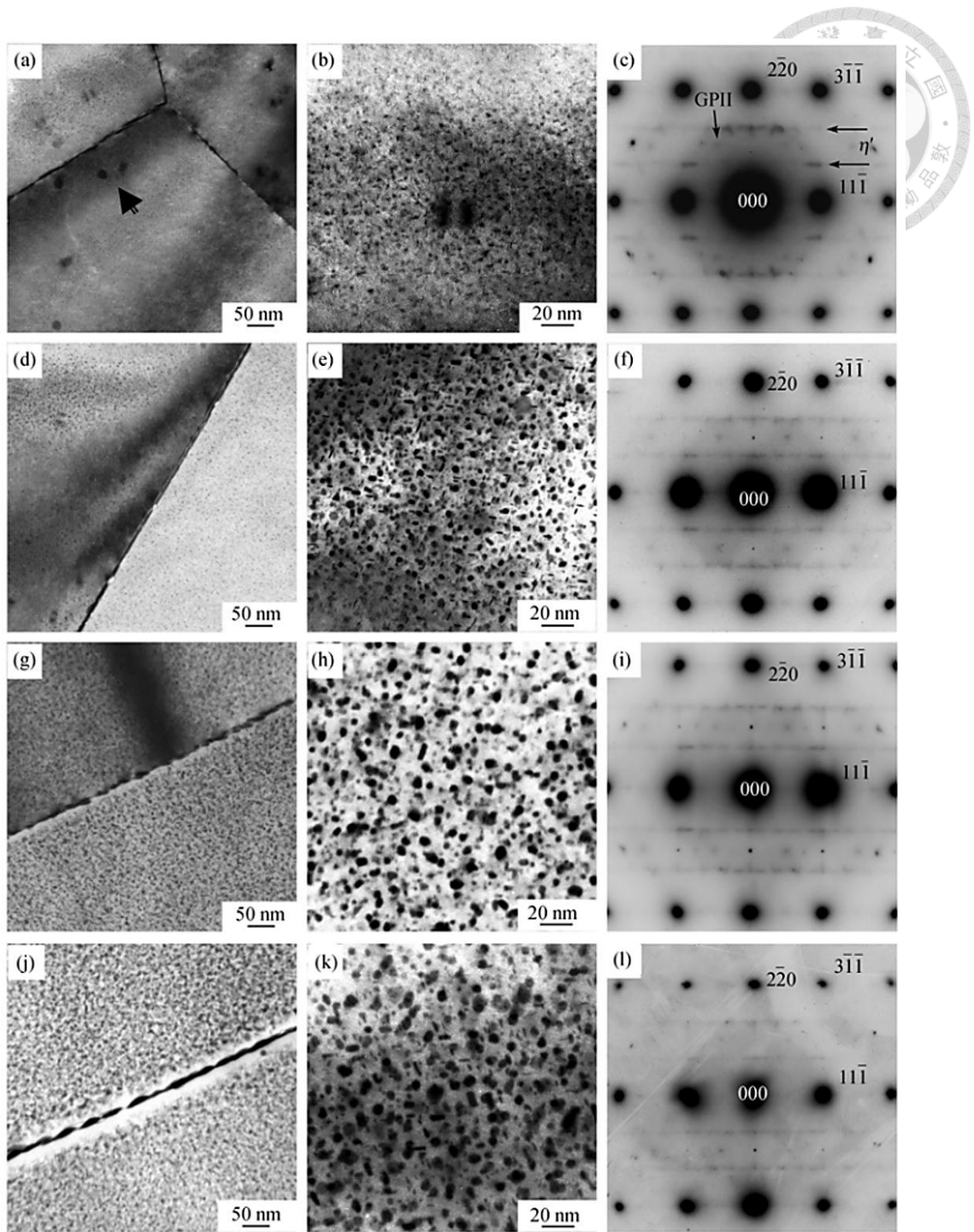


Figure 2- 11 TEM image of peak aging condition of Al-7.55Zn-1.58Mg-1.39Cu-0.12Zr (wt%) (a-c) 100 °C/36h ; (d-f) 120 °C/24h ; (g-i) 140 °C/14h ; (j-l) 160 °C/4h [31].

2.2 Precipitation Hardening

2.2.1 Mechanism of Precipitation Hardening

In heat-treatable aluminum alloys, precipitation strengthening is the primary mechanism of enhancement. Within this mechanism, the size and distribution of precipitates are particularly crucial [32]. The theory of precipitation strengthening mainly focuses on the interaction between dislocations and precipitates because precipitates can block the movement of dislocations, thereby increasing material strength. A widespread distribution of precipitates can better obstruct dislocations. Under the same volume fraction of precipitates, materials with a broader distribution of precipitates will exhibit better precipitation strengthening effects. The ability of precipitates to block dislocations is directly proportional to the size of the precipitates [33-35], requiring greater stress to shear the precipitates and allow dislocations to pass during the initial formation of precipitates. The size of precipitates is positively correlated with material strength, as indicated by Equation 2-1. However, once precipitates exceed a certain size, dislocations cannot shear through them and will instead pass around the precipitates in the form of dislocation loops [36]. At this point, an increase in the size of precipitates no longer strengthens the material but instead can reduce strength, as shown in Equation 2-2, which also explains why strength decreases after peak aging as discussed in the previous section. Figure 2-12 is the equation function and Figure 2-13 is the illustrating diagram of different size precipitates. Therefore, based on the theory of precipitation strengthening, achieving an optimal size of precipitates while maintaining a widespread distribution is a method to enhance material strength.



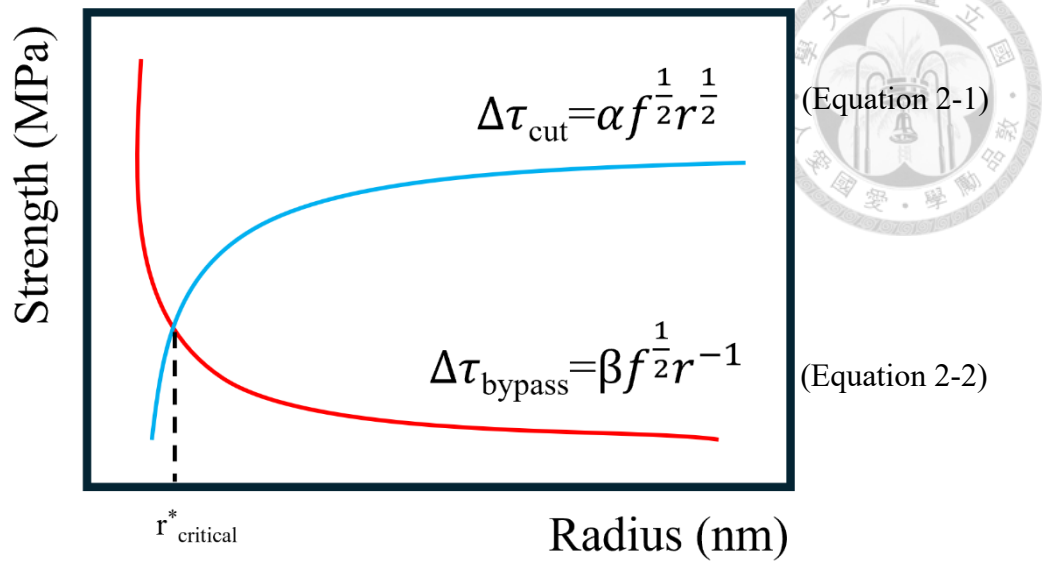
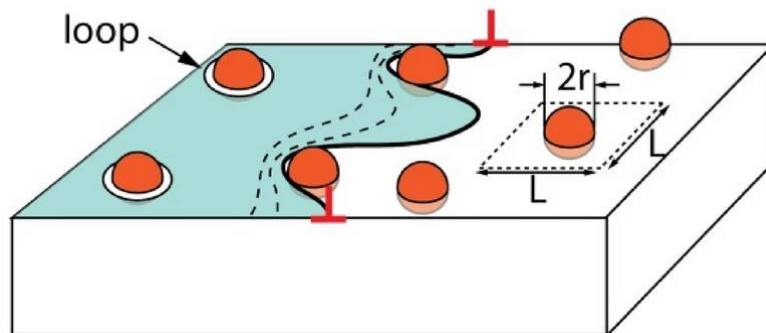


Figure 2- 12 Diagram Illustrating the Transition of Precipitation Hardening Mechanism When Precipitates Reach Critical Size.

(a) Orowan Mechanism



(b) Cut-Through Mechanism

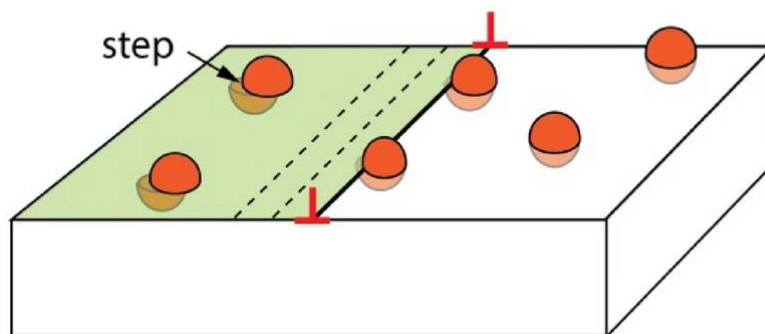


Figure 2- 13 (a) Diagram of dislocation loops passing around precipitates when they are larger than the critical size (b) Diagram of shearing through precipitates when they are smaller than the critical size [36].

2.2.2 Introduction to Precipitates in AA7075 Aluminum Alloy

In the AA7075 aluminum alloy, the precipitates mainly consist of η and η' as the primary precipitates [37]. However, before the formation of η and η' , there are other precursors that can also achieve precipitation strengthening effects, especially η' , which is crucial. The T6 artificial aging heat treatment is aimed at accelerating the formation rate of η' . These precipitates range in size from a few atoms to several nanometers [38], different in scale from the second phases mentioned later. During the solution treatment of aluminum alloys, the aim is to diffuse impurity and solute atoms from second phases uniformly into the matrix, forming a supersaturated solid solution (SSSS). During aging, solute atoms in the matrix cluster due to high-temperature diffusion to form embryos. As the solute atoms increase, coherent GP zones form, followed by semi-coherent η' , and finally, the incoherent stable phase η [39, 40]. These precipitates will be introduced in more detail in the following section.

2.2.3 GP zones

The GP zone is a coherent metastable phase formed by the clustering of solute atoms during natural aging after solution treatment of aluminum [41, 42]. It was simultaneously discovered by André Guinier [41] and George Dawson Preston [42] in 1938 and named after the initials of their surnames. At that time, signals distinct from the aluminum alloy matrix were found in the diffraction patterns of Al-Cu alloys, as shown in Figure 2-14. These signals originated from copper and appeared one hour after solution treatment, becoming more pronounced over time. This is the GP zone formed by the clustering of solute atoms during natural aging.

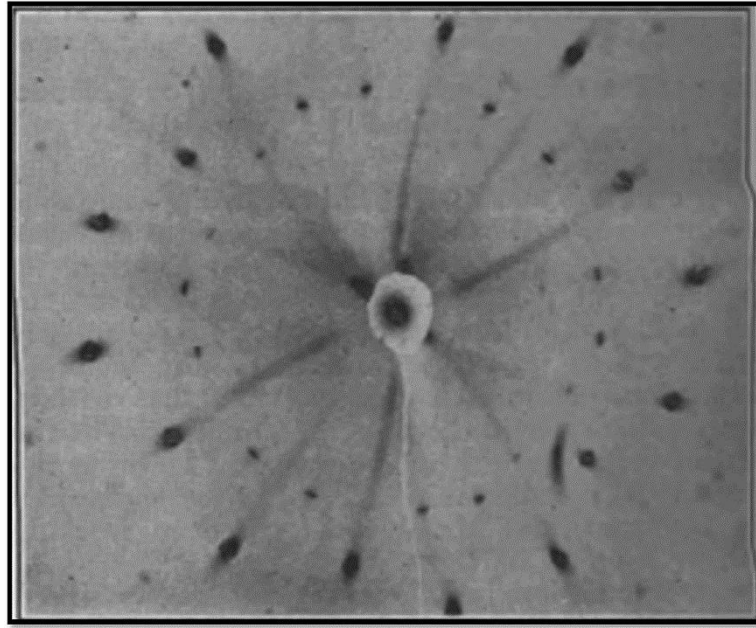


Figure 2- 14 Streak-like signals appeared on the Al-Cu alloy [41].

Subsequent research has revealed the presence of two types of GP zones in the AA7000 series aluminum alloys, named GPI and GPII zones [40, 43]. The GPI zone, characterized by a spherical morphology with a diameter less than 3 nanometers [39, 44-49], is orderly arranged on the $\{100\}_{\text{Al}}$ planes through a coherent interface. GPI zones primarily form at room temperature and the temperatures used for artificial aging [50], representing the earliest form of solute atom cluster precipitates. They are the reason for the gradual increase in material strength during T4 natural aging. GPII zones, differing from GPI zones, have a plate-like morphology, with sizes ranging from 3 to 6 nanometers and thickness of several angstroms, growing on $\{111\}_{\text{Al}}$ [39, 49, 51]. They mainly form at temperatures higher than room temperature, around 70 degrees Celsius, and research indicates a positive correlation with the concentration of defects after solution treatment and quenching. Although both GPI and GPII zones are types of GP zones, they are generally considered different precipitates. STEM images of both types are shown in the Figure 2-15.

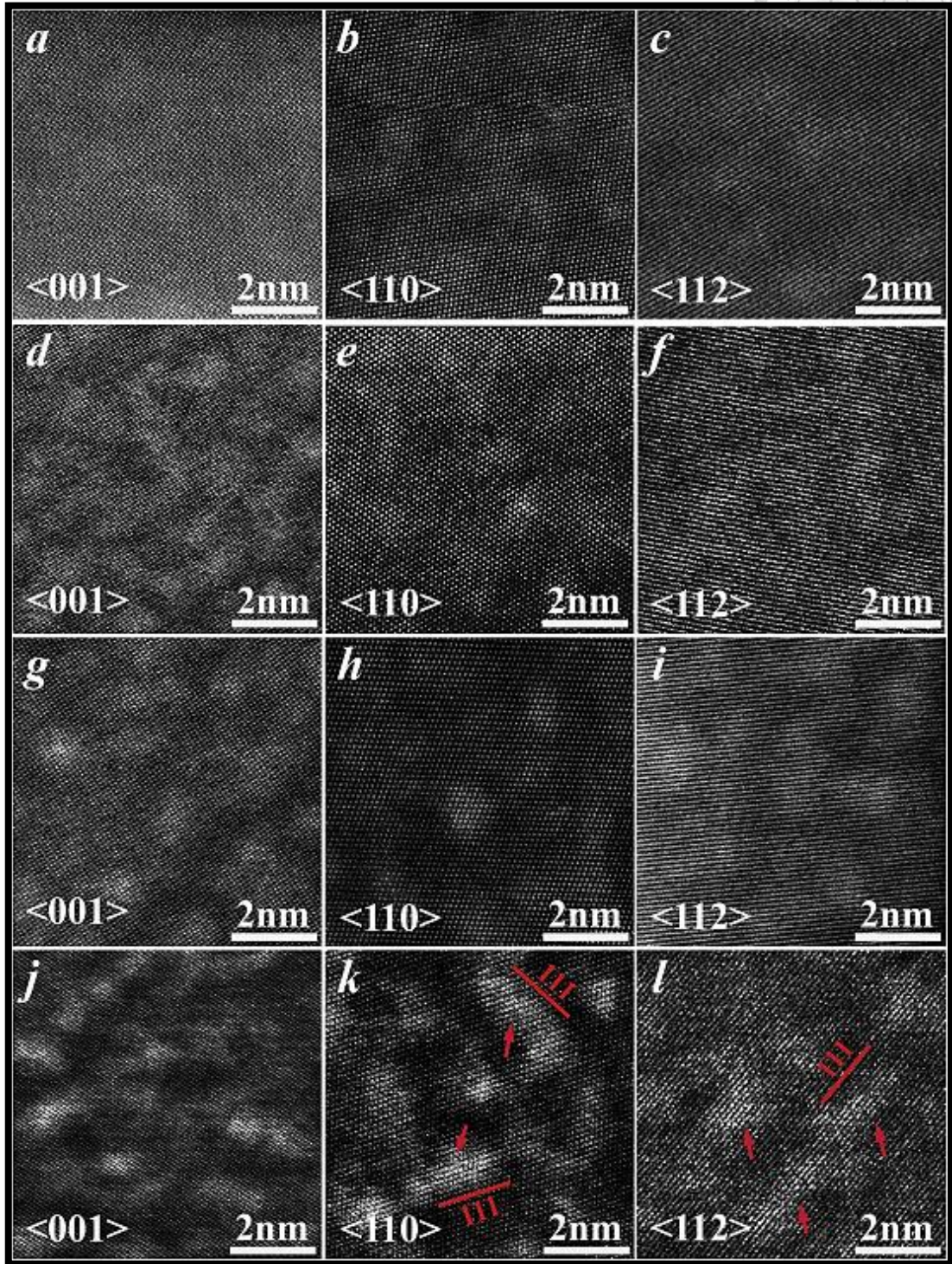


Figure 2- 15 HAADF-STEM image of nature aging Al-4.62Zn-0.9Mg (wt%) (a-c) 1 week ; (d-f) 1 month ; (g-i) 3 monthss ; (j-l) 14 months [44].

2.2.4 η' Precipitates

As the GP zone undergoes further clustering of solute atoms and the Zn/Mg ratio gradually increases (less than 1.5) [38, 52, 53], it transitions into the metastable phase η' [39, 54], considered the primary precipitation strengthening phase in AA7000 series aluminum alloys. The interface between the η' phase and the aluminum matrix becomes aemicoherent [53]. η' is thought to be hexagonal [55-57], with lattice constants calculated using the Vienna Ab-initio Simulation Package (VASP) as $a=0.504\text{nm}$ and $c=1.303\text{nm}$ [58]. There are a total of four variants of η' orientation relationships [53], as shown in the Figure 2-16 and Table 2-4 below. The orientation relationship of η' exhibits unique characteristics; when observing η' , at most, only two types of edge-on η' can be seen, presenting a plate-like morphology, while non-edge-on η' can only be observed in projection.

Table 2- 4 Four variants of η' precipitates in aluminium alloys [53].

η'	Orientation relationship	
1	$(0001)_{\eta'} // (\bar{1}\bar{1}1)_{Al}$	$[10\bar{1}0]_{\eta'} // [110]_{Al}$
2	$(0001)_{\eta'} // (\bar{1}\bar{1}\bar{1})_{Al}$	$[10\bar{1}0]_{\eta'} // [110]_{Al}$
3	$(0001)_{\eta'} // (111)_{Al}$	$[10\bar{1}0]_{\eta'} // [\bar{1}10]_{Al}$
4	$(0001)_{\eta'} // (11\bar{1})_{Al}$	$[10\bar{1}0]_{\eta'} // [\bar{1}10]_{Al}$

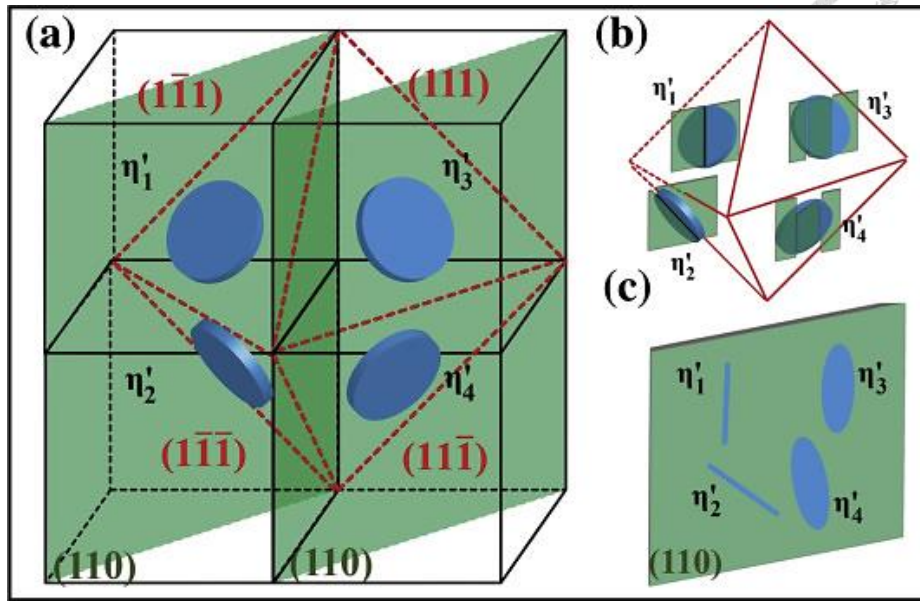


Figure 2- 16 Diagram illustrating four variants of η' [53].

2.2.5 η Precipitates

Upon continued heating and clustering of solute atoms, when the Zn/Mg ratio reaches a certain level [38, 53, 55], it transitions into the stable phase η precipitates, with the composition of MgZn_2 [40, 57, 59]. Both η and η' are hexagonal crystal structures, but η and the aluminum alloy matrix form an incoherent interface [40, 54, 57, 59, 60]. According to first-principles calculations, the lattice constants are $a=0.504\text{nm}$ and $c=0.828\text{nm}$. η can be further divided into 15 types based on different orientation relationships with the aluminum alloy matrix [61], as shown in the Table 2-5 and Figure 2-17 below. Additionally, research indicates that the precipitates are primarily dominated by η_2 , believed to be transformed from η' , as both share the same orientation relationship with the aluminum alloy matrix. Figure 2-18 and Figure 2-19 show the HRTEM and FFT of GPII, η' and η . The precipitates η mentioned in this section and the second phase η discussed in the following section differ; both are composed of MgZn_2 , but they differ in size and quantity, and while precipitates enhance the mechanical properties of the material, second phases decrease them.

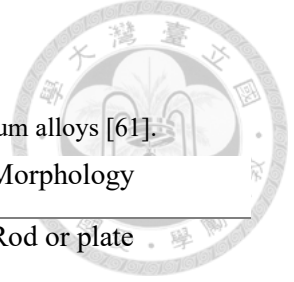


Table 2- 5 Orientation relationships and morphologies of η precipitates in aluminum alloys [61].

Variants	Orientation relationship		Morphology
η_1	$(10\bar{1}0)_{\eta_1} // (001)_{Al}$	$[2\bar{1}\bar{1}0]_{\eta_1} // [110]_{Al}$	Rod or plate
η_2	$(0001)_{\eta_2} // (1\bar{1}\bar{1})_{Al}$	$[10\bar{1}0]_{\eta_2} // [110]_{Al}$	Hexagonal or rounded plate
η_3	$(0001)_{\eta_3} // (1\bar{1}\bar{1})_{Al}$	$[2\bar{1}\bar{1}0]_{\eta_3} // [110]_{Al}$	Hexagonal or triangular plate
η_4	$(0001)_{\eta_4} // (110)_{Al}$	$[2\bar{1}\bar{1}0]_{\eta_4} // [1\bar{1}\bar{1}]_{Al}$	Rod
η_4'	$(0001)_{\eta_4'} // (110)_{Al}$	$[1\bar{1}\bar{0}0]_{\eta_4'} // [001]_{Al}$	Hexagonal plate or prism
η_5	$(\bar{1}2\bar{1}0)_{\eta_5} // (1\bar{1}1)_{Al}$	$[30\bar{3}2]_{\eta_5} // [110]_{Al}$	Rod
η_6	$(\bar{1}2\bar{1}0)_{\eta_6} // (1\bar{1}1)_{Al}$	$[20\bar{2}1]_{\eta_6} // [1\bar{1}2]_{Al}$	Rod
η_7	$(\bar{1}2\bar{1}0)_{\eta_7} // (1\bar{1}1)_{Al}$	$[10\bar{1}4]_{\eta_7} // [110]_{Al}$	Rod
η_8	$(\bar{1}2\bar{1}0)_{\eta_8} // (1\bar{1}2)_{Al}$	$[0001]_{\eta_8} // [31\bar{1}]_{Al}$	Rod
η_9	$(11\bar{2}0)_{\eta_9} // (001)_{Al}$	$[1\bar{1}00]_{\eta_9} // [110]_{Al}$	Hexagonal prism
η_{10}	$(0001)_{\eta_{10}} // (1\bar{1}\bar{1})_{Al}$	$[2\bar{1}\bar{1}0]_{\eta_{10}} // [1\bar{3}4]_{Al}$	Not indicated
η_{11}	$(0001)_{\eta_{11}} // (110)_{Al}$	$[10\bar{1}0]_{\eta_{11}} // [1\bar{1}\bar{1}]_{Al}$	Rod
η_{12}	$(0001)_{\eta_{12}} // (1\bar{1}3)_{Al}$	$[2\bar{1}\bar{1}0]_{\eta_{12}} // [110]_{Al}$	Plate
η_{13}	$(0001)_{\eta_{13}} // (042)_{Al}$	$[2\bar{1}\bar{1}0]_{\eta_{13}} // [100]_{Al}$	Rod
η_{14}	$(0001)_{\eta_{14}} // (51\bar{3})_{Al}$	$[2\bar{1}\bar{1}0]_{\eta_{14}} // [112]_{Al}$	Plate

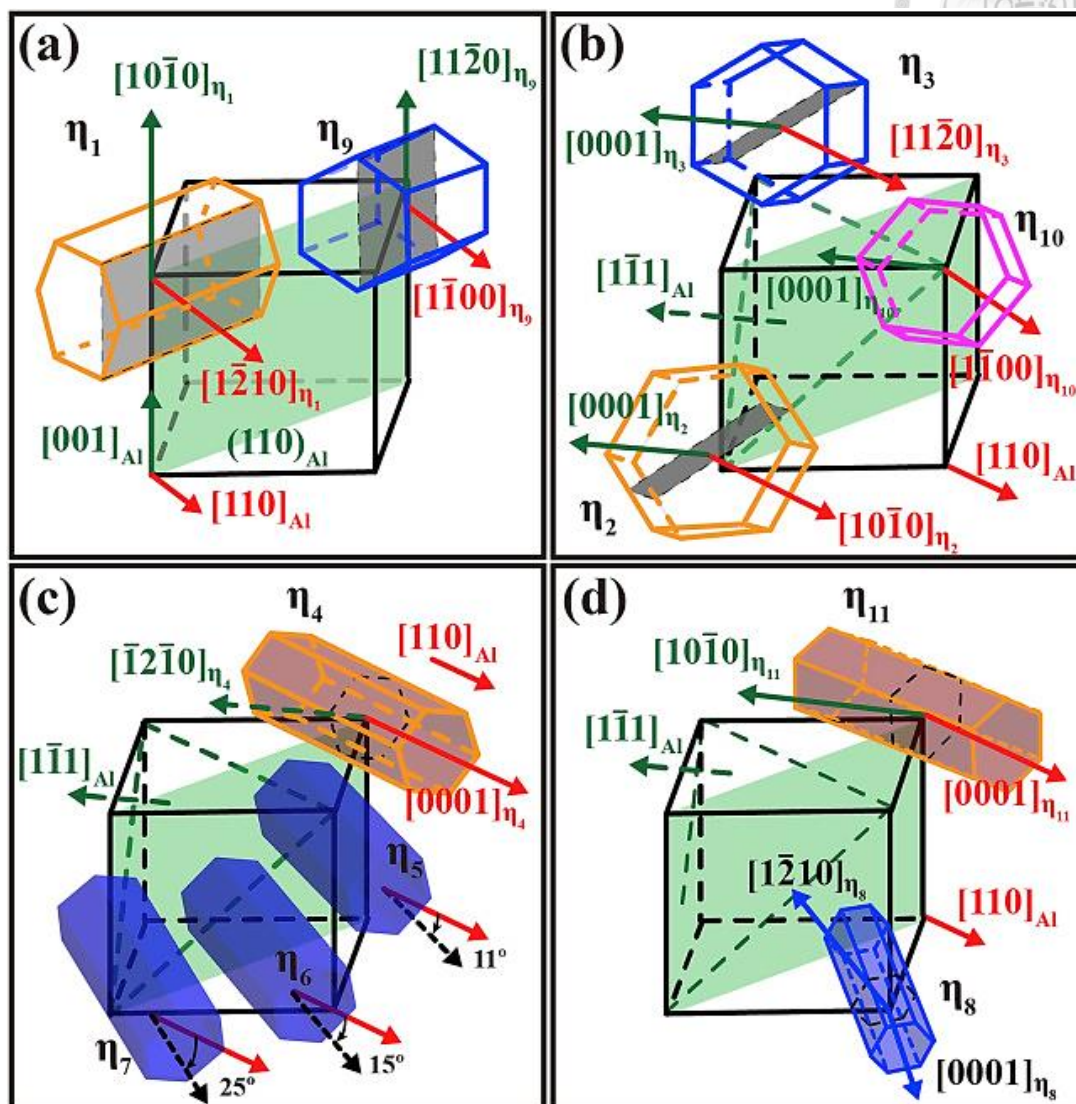


Figure 2- 17 Diagram illustrating orientation of η [61].

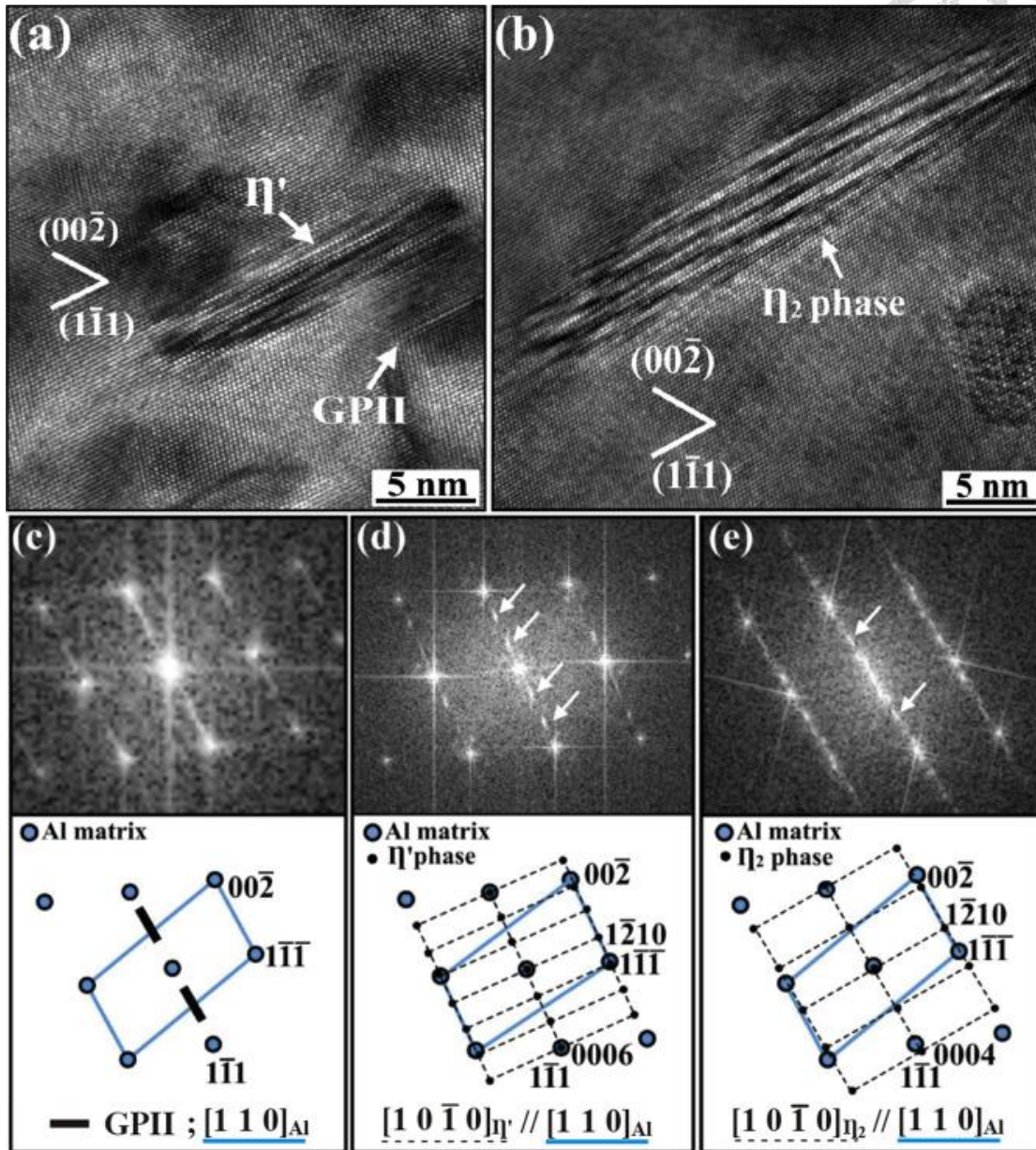


Figure 2- 18 HRTEM image of (a) GPII zone (b) η' (c) η FFT of (d) GPII zone (e) η' (f) η [53].

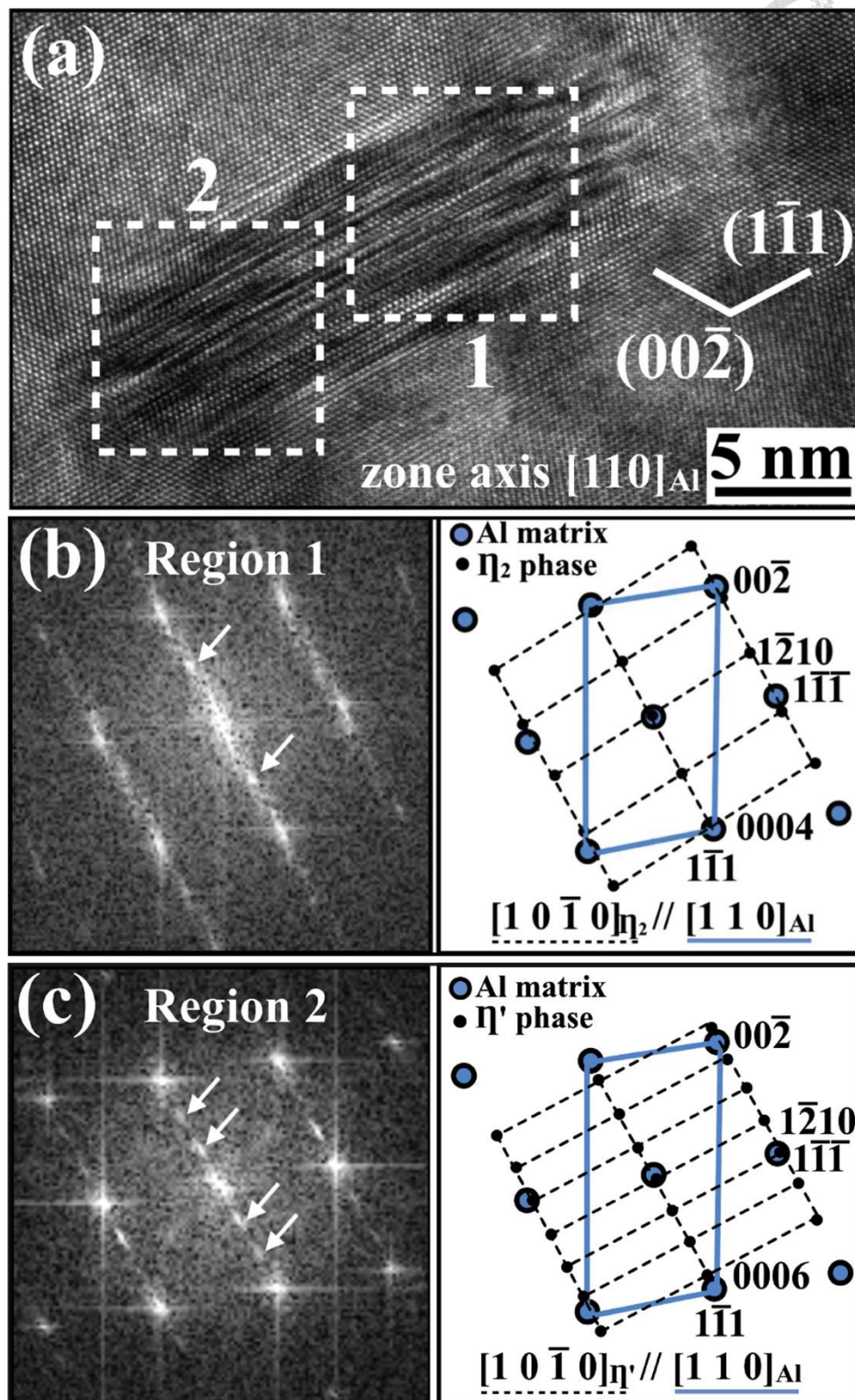


Figure 2- 19 (a) HR-TEM image of Al-6.25Zn-2.14Mg-2.23Cu-0.05Fe-0.03Si (wt.%) showing transformation of η' (region 2) to η_2 (region 1) (b)(c) FFT of region 1 and region 2 [53].

2.2.6 Overview of Precipitates

In summary, the changes in precipitates of AA7075 aluminum alloy according to heat treatment are as follows: supersaturated solid solution $\rightarrow$ VRC $\rightarrow$ GP zones $\rightarrow$ η' $\rightarrow$ η [39], shown in Figure 2-20. During solution treatment, alloy elements dispersed among the second phases and impurities in the aluminum alloy are dissolved back into the matrix, followed by quenching to form a supersaturated solid solution. With natural aging, solute atoms cluster, forming the GPI zone, and eventually, with artificial aging, the GPII zone, η' , and η are formed. The experimental process involves solution treatment followed by water quenching, then left at room temperature for 7 days for nature aging, and finally artificially aged at 120 °C for 24 hours to achieve an optimal state of precipitates.

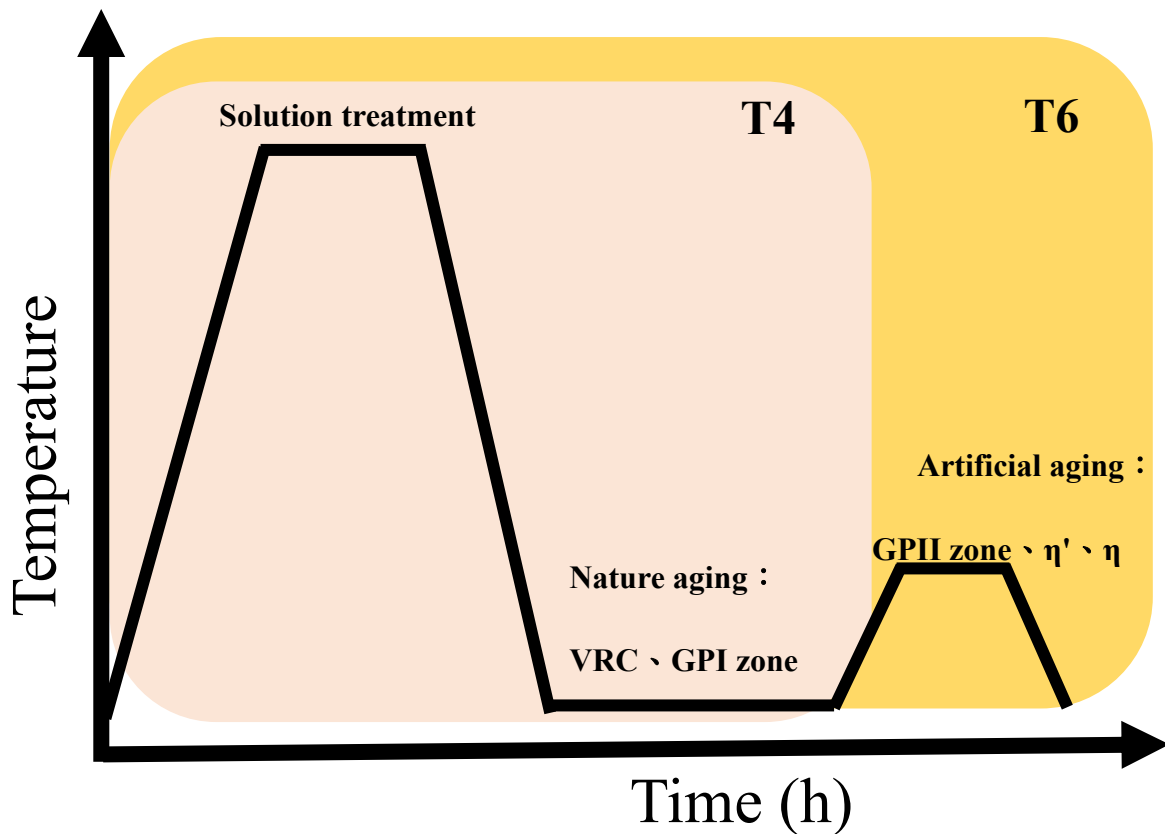


Figure 2- 20 Major precipitates in different stage of heat treatment.

2.3 Second Phases

2.3.1 Introduction to Second Phases

Aluminum alloys contain a variety of second phases [62], with size scales ranging from several micrometers to tens of micrometers [63, 64], which differ from the precipitates associated with precipitation strengthening mentioned earlier. These second phases in aluminum are treated as impurities, resulting from process defects. Second phases do not contribute to precipitation strengthening in aluminum alloys and can even decrease mechanical properties [65-68]. Firstly, since second phases are clusters of solute atoms, the formation of second phases consumes solute atoms of nanoscale precipitates, thereby reducing the ratio of precipitates [69-71]. Besides, studies have indicated that there is a certain correlation between second phases and fracture surfaces [72]. Figure 2-21-22 and Figure 2-23 shows the relationship between second phases, incipient melting and fracture surfaces, respectively. It is observed that the damage to aluminum alloys is caused by second phases. Additionally, because second phases are distributed within the aluminum alloy matrix, when the solution temperature is higher than the melting point of the second phases, it will cause direct melting of the second phases rather than diffusion into the aluminum alloy matrix, leaving voids where the second phases were, a phenomenon known as incipient melting [73]. Therefore, completely dissolving second phases while avoiding incipient melting is a key research topic.

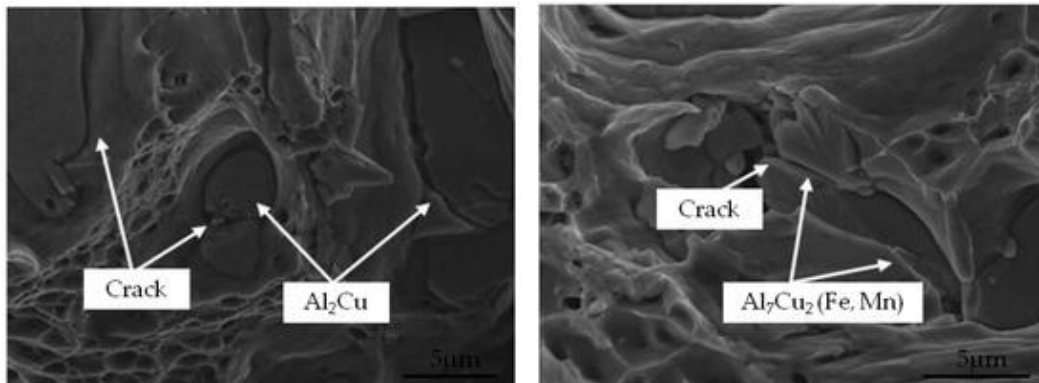


Figure 2- 21 (a) (b) The 2219 aluminum alloy fractures on the surface due to second phase [72].

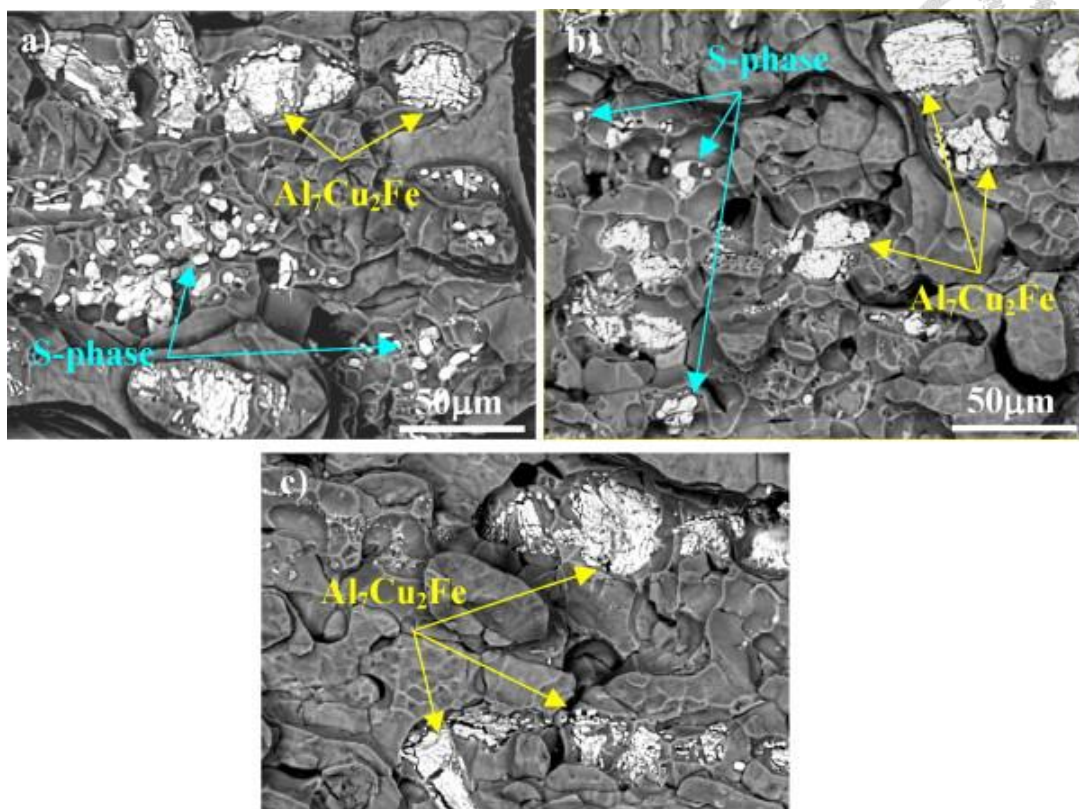


Figure 2- 22 Fracture surfaces of (a) 475 °C/8 h, (b) 475 °C/8 h + 485 °C/4 h and (c) 475 °C/8 h + 485 °C/4 h + 495 °C/2 h in Al-6.22Zn-2.11Mg-2.39Cu (wt.%) alloy [84].

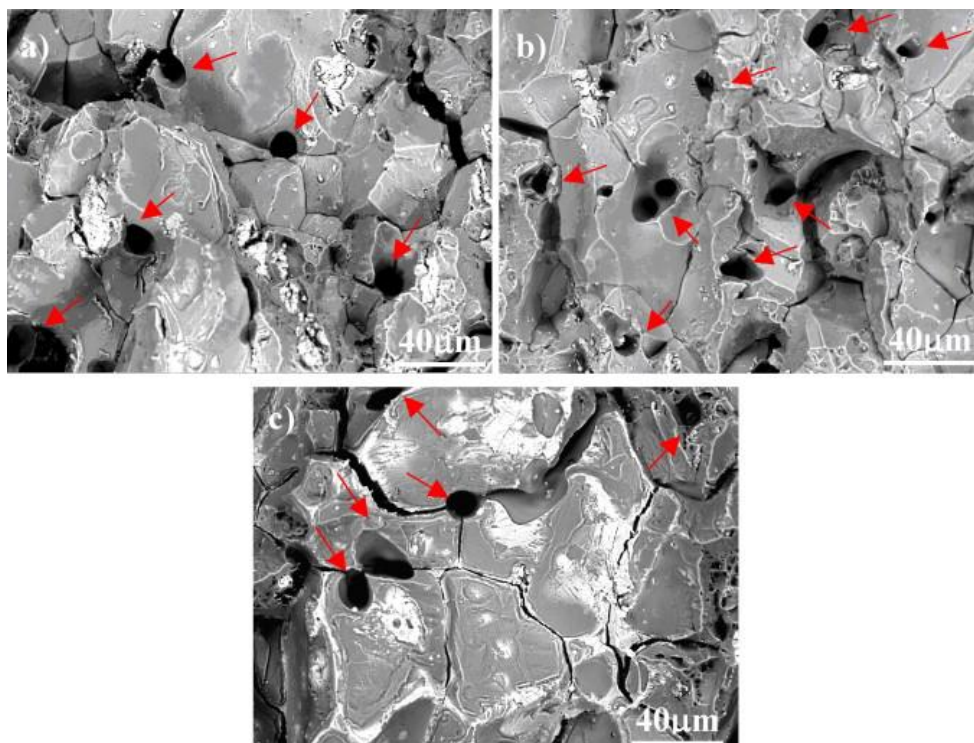


Figure 2- 23 Fracture surfaces of (a) 490 °C/2 h, (b) 505 °C/1 h and (c) 515 °C/1 h in Al-6.22Zn-2.11Mg-2.39Cu (wt.%) alloy [84].

2.3.2 Introduction to Second Phases in AA7000 Series Aluminium Alloys

Second phases in aluminum alloys vary depending on the proportions of alloying elements within the alloy [74-78]. In the AA7000 series aluminum alloys, the main second phases are η , T, S, and iron-based phases, with characteristics listed in Table 2-6, and the proportion of these phase would slightly differ in different Zn, Cu and Mg content [64, 74, 79, 80].

Table 2- 6 Main second phases in AA7000 series aluminium alloys [81-87].

	structure	composition	SEM morphology	Solution temperature
η	hexagonal	MgZn ₂	Bright, fine, elongated	475 ~ 478° C
T	cubic	Al ₂ Mg ₃ Zn ₃	Quasi-triangular	450 ~ 482° C
S	Orthorhombic	Al ₂ CuMg	Gray, round, cylindrical	490 ~ 501° C
Iron-base phase(ω)	Tetragonal	Al ₇ Fe ₂ Cu	Gray, irregular	Non-solutionable

2.3.3 The problem of second phases

The second phases are mainly metastable phases [71], and their formation is closely related to the types of elements in the aluminum alloy matrix. In this experiment, the 7075 aluminum alloy used comes in two varieties: high copper content and low copper content, with the dominant second phases illustrated in the Figure 2-24 below. Different copper content would lead to different main second phase, and the detail will be discussed in section 2.4. In the low copper material, the η phase is dominant, with the S phase being secondary, while in high copper content, the S phase is dominant, and the η phase is secondary. In both materials, the η and S phases are targeted for complete dissolution back into the aluminum alloy matrix, with avoiding incipient melting as the main goal of this experiment. °

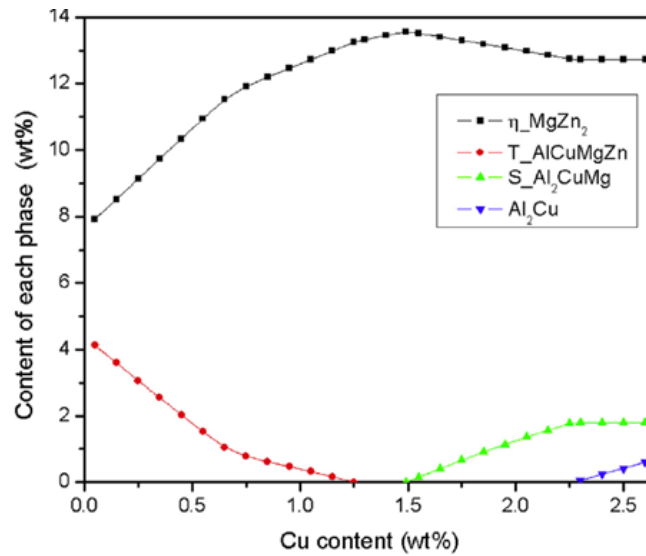


Figure 2- 24 Relation between Cu ratio and second phases in aluminium alloys [88].

2.4 The Effect of Adding Cu on 7075 Aluminum Alloy

In terms of mechanical properties, early research indicated that adding Cu to aluminum alloys with low base Cu content could increase material strength and ductility [89]. Besides leading to different second phases, changing the Cu content in the aluminum alloy significantly impacts the material properties [89]. Microscopically, studies have shown that adding Cu elements can increase the formation of precipitation strengthening precipitates such as GP zones, and slow down the transformation of GP zones into η' and η , indicating that Cu likely participates in the early stages of GP zone formation [90, 91]. Furthermore, other research suggests that Cu has a significant effect on η' , preventing the transformation of η' into η and inhibiting coarsening of the η phase [90].

However, later studies have shown that when the proportion of Cu content increases to a certain level, about 0.8wt%, although material ductility may still increase with Cu content, both YS and maximum tensile strength decrease [88], as detailed in Figure 2-25.

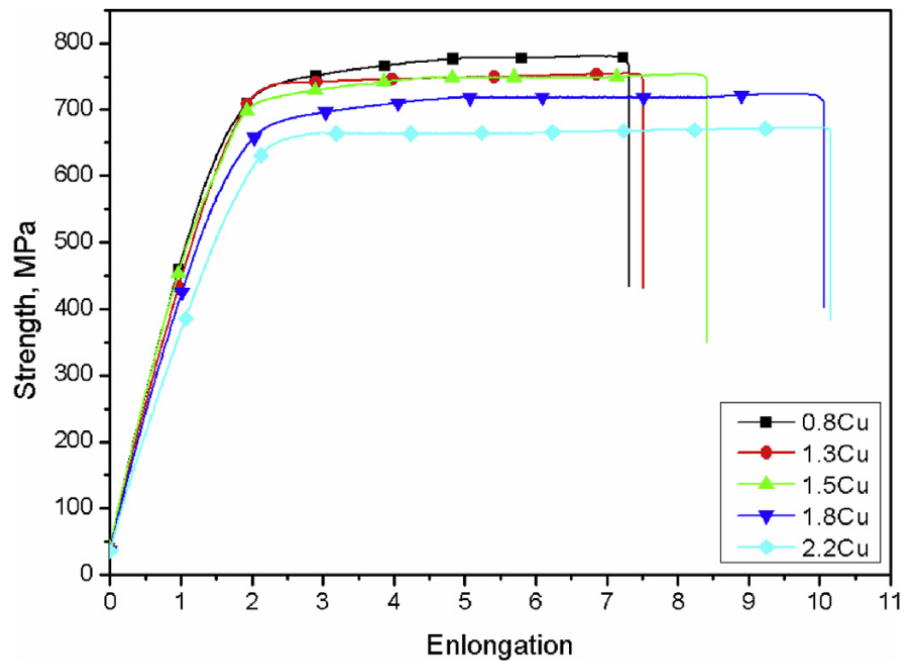


Figure 2- 25 Tensile curves of aluminum alloys with different Cu contents [88].

Recent studies indicate that when the Cu content exceeds 1wt%, the material's strength decreases with increasing Cu content due to the formation of second phases [92-94] reducing the volume fraction of precipitates. Similarly, when the Cu content exceeds 1.5wt%, the material's ductility shows the same trend, as detailed in Figure 2-26.

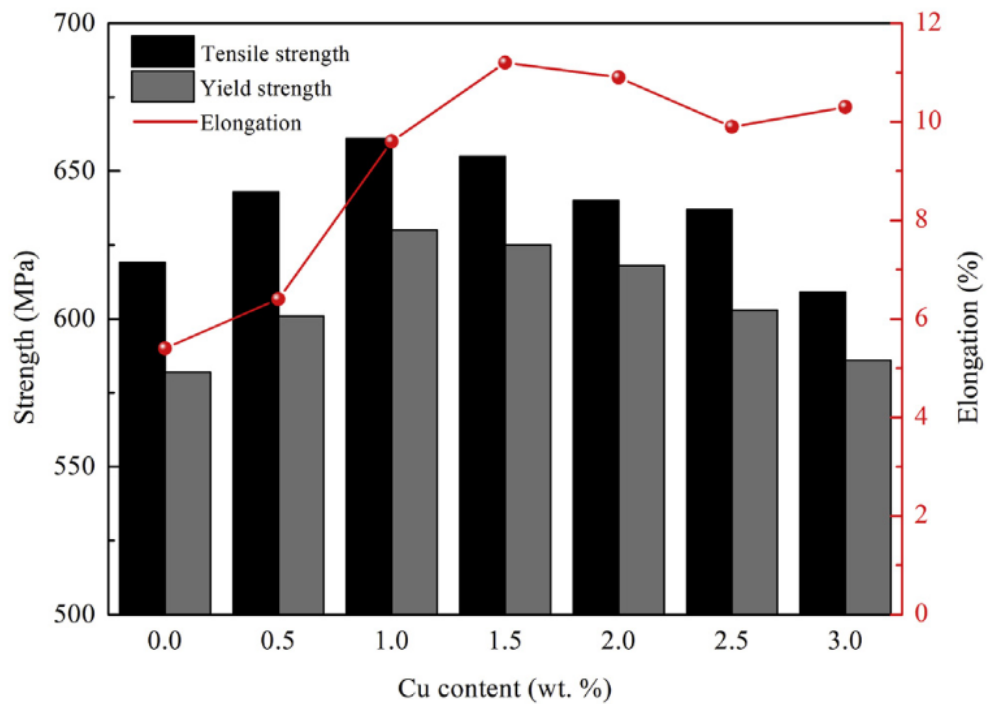


Figure 2- 26 Mechanism properties of aluminum alloys with different Cu contents [95].

Early research on whether adding copper can improve the corrosion resistance of aluminum was inconclusive. Some studies suggested that adding copper could increase the corrosion resistance of aluminum alloys [92, 96], while another school of thought believed that adding copper increases the ratio of copper-containing phases $\text{Mg}(\text{Zn}, \text{Cu}, \text{Al})_2$, which could improve aluminum's corrosion resistance [97]. However, current research [88] suggests that when copper-containing second phases precipitate at grain boundaries, it decreases the material's corrosion resistance, as shown in Figure 2-27 below.

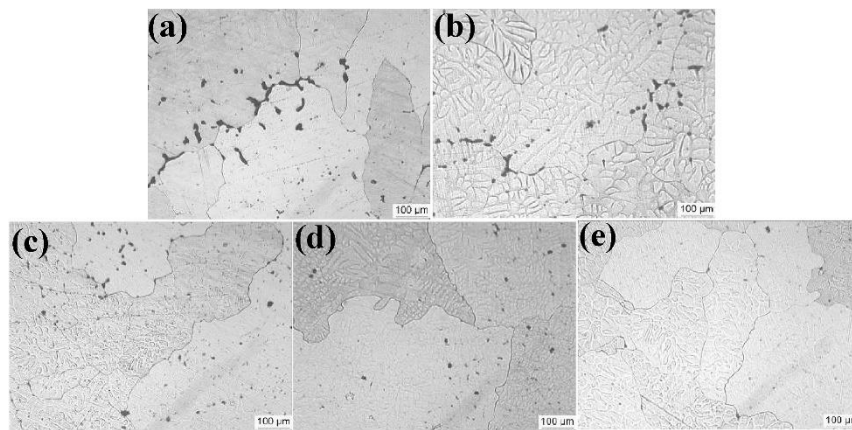


Figure 2- 27 Cu-containing second phases at the grain boundaries of different Cu content aluminum alloys (a)2.2% (b)1.8% (c)1.5% (d)1.3% (e)0.8% (Cu wt%) [88].

In summary, adding Cu content has various effects on aluminum alloys. The past researcher added copper element into aluminum matrix directly and finding that the S phase leads to several negative impacts on material properties [88]. Firstly, the S phase makes the material more prone to fracture. Research indicates that increasing the Cu content in aluminum alloys leads to the appearance of the S phase eutectic, making the fracture surface rougher, unlike the smooth surface typically seen after material fracture, a result of increased Cu content shown in Figure 2-28. Additionally, the chemical composition of the S phase is Al_2CuMg . The formation of the S phase consumes the precipitates η' and η , which are necessary for Mg elements in MgZn_2 , hence eliminating S phase and other second phases is the target of this experiment when modifying solution treatment conditions.

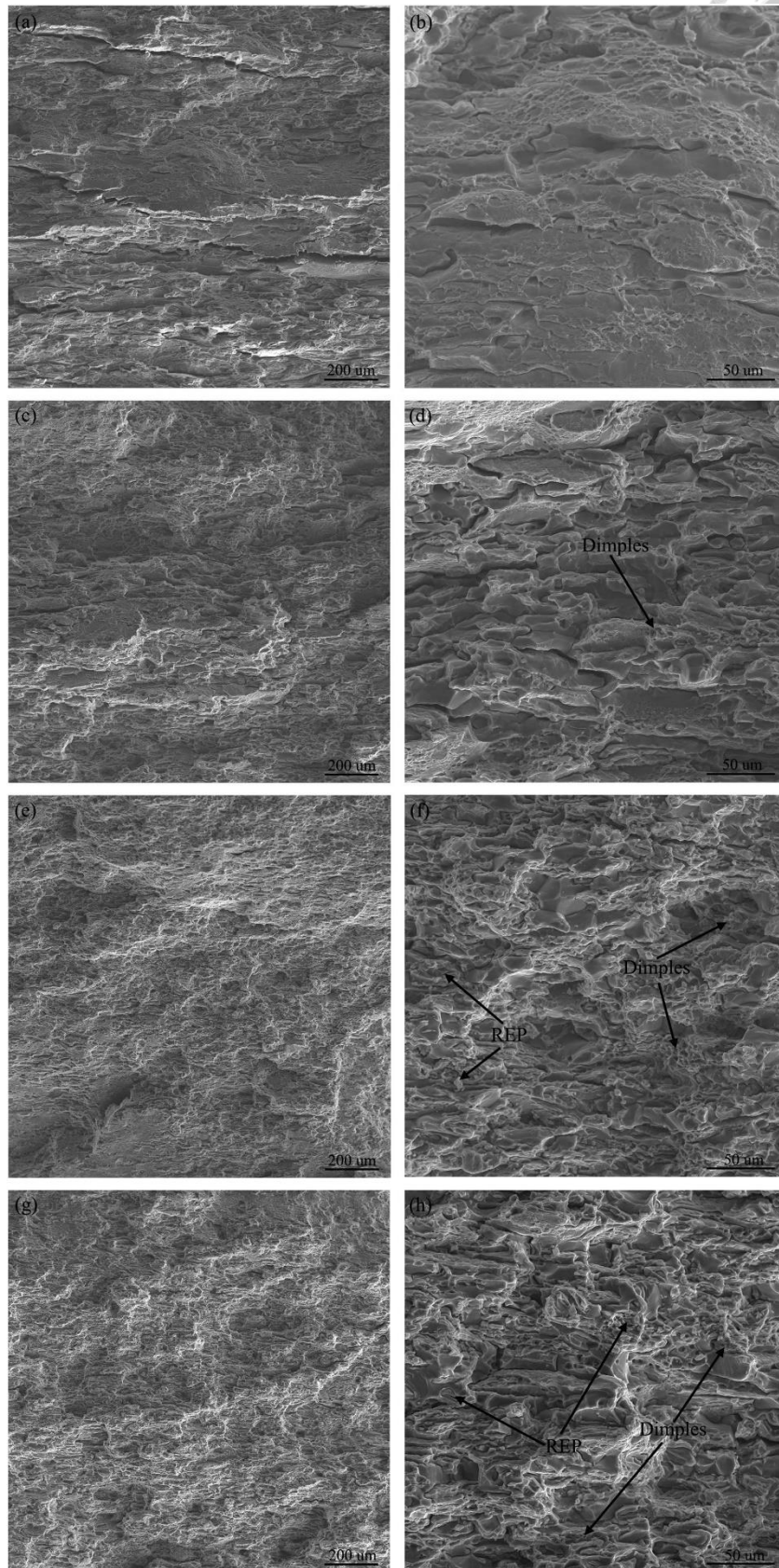
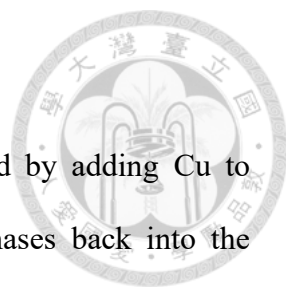


Figure 2- 28 Fracture surface morphology of aluminum alloys with different Cu contents under SEM (a, b) 0, (c, d) 1.0, (e, f) 2.0, and (g, h) 3.0 Cu wt% [88].

2.5 Mutli-Stage Solution Heat Treatment



Due to the various issues arising from second phases formed by adding Cu to AA7075 aluminum alloys, completely dissolving these second phases back into the matrix is a necessary solution. Multi-stage solution heat treatment is one method to achieve this [98-101]. Traditionally, solution heat treatment involves a single-stage fixed temperature solution, and research shows that material strength increases with solution temperature up to a certain point before decreasing [88, 102] representing in Figure 2-29. The solution temperature plays a curial role in aluminum properties [23, 103-106]. Aluminum alloys typically select the temperature that offers the best comprehensive mechanical properties as the solution temperature, which for AA7075 aluminum alloy is usually around 490 °C. However, single-temperature solution heat treatments have some problems. First, because the melting points of the various second phases dispersed within the aluminum alloy matrix differ, using a lower heat treatment temperature might lead to excessively long solution times that are not cost-effective. Moreover, if the solution temperature is much lower than the melting points of the second phases, it might prevent the second phases from dissolving and diffusing into the aluminum alloy matrix, thus losing the solution effect and failing to improve material mechanical properties. Additionally, if a solution temperature is too high, higher than the melting points of the second phases by a significant margin, it may cause direct melting of the second phases, leading to incipient melting and leaving voids, which reduces the material's mechanical properties [84]. Excessively high solution temperatures can also lead to the formation of eutectic phases between second phases and the aluminum alloy matrix, causing the material to fracture before reaching its maximum tensile strength and rendering it unusable. Therefore, if the melting points of the aluminum alloy's second phases are close,

a single-stage solution treatment can be used to dissolve all second phases at once without significant problems of incipient melting. However, if there is a considerable difference in the melting points of the internal second phases, single-stage solution treatment may lead to incomplete dissolution or incipient melting, thereby reducing material performance [107, 108].

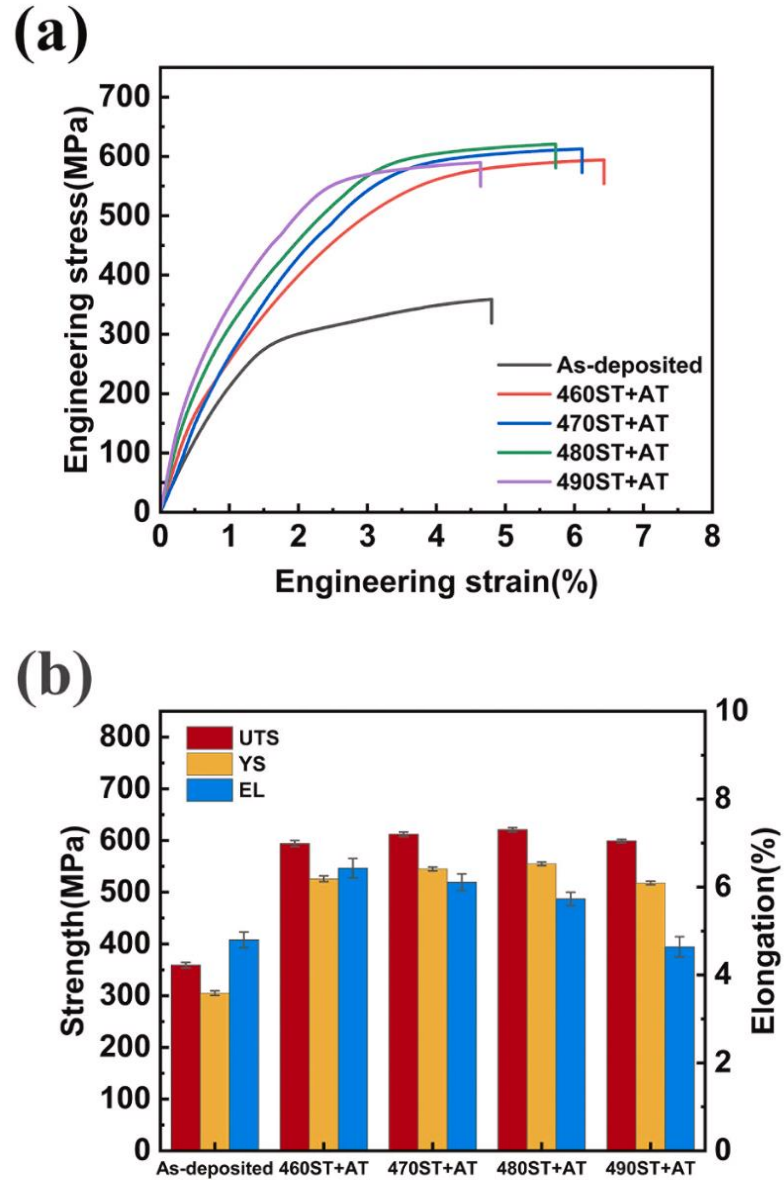
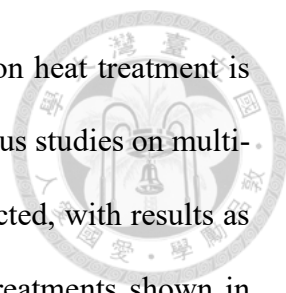


Figure 2- 29 Mechanical properties of single-stage solution treatment on (a)(b) Al-7.58Zn-2.12Mg-1.66Cu (wt.%) [102].

The materials used in this experiment have two main second phases whose melting



points differ by approximately 20 °C. Therefore, a two-stage solution heat treatment is preferable to separately address the η phase and the S phase. Numerous studies on multi-stage solution heat treatments for aluminum alloys have been conducted, with results as follows. The studies show that through multi-stage solution heat treatments shown in Figure 2-30, the yield strength and maximum tensile stress of aluminum alloys can be improved [109]. However, previous studies [110-115] had some problems. Firstly, they focused on enhancing strength rather than ductility. While there are many ways to increase strength in aluminum alloys, such as altering aging processes [116] or pre-strain [117, 118], these methods do not effectively improve ductility. Solution treatment is the only method that can effectively enhance ductility, so previous studies overlooked the importance of ductility. Moreover, if second phases could be significantly eliminated, theoretically, ductility could also increase without sacrificing strength for material strengthening. Furthermore, previous studies [111, 119] did not compare material strength for the same total solution treatment time, only comparing the addition of a new stage of solution time to previous processes. The only difference was whether a new stage of solution time was added, so the impact of extending solution time could not be ignored, making it unclear whether improvements in mechanical properties were due to multi-stage solution or just an increase in total solution time. Theoretically, extending solution time could dissolve more solute atoms into the matrix to form a supersaturated solution, potentially improving material properties with increased total solution time, a discussion absent from previous research. Finally, in previous studies [111] shown in Figure 2-31, the total solution time was extended beyond 12 hours, even up to 36 hours or more, which is highly inefficient, resembling homogenization treatment more than solution treatment, losing the essence of solution heat treatment. The excessive solution time is impractical. Therefore, the process in previous studies needs improvement.

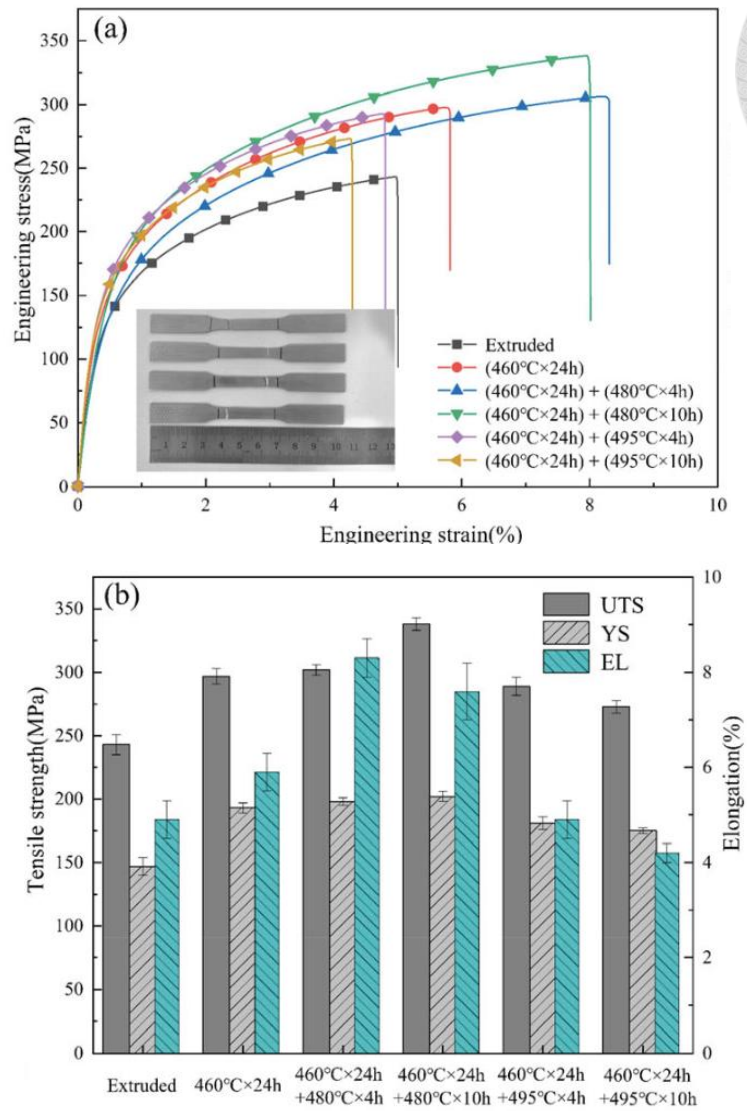


Figure 2- 30 Mechanical properties of multiple-stage solution treatment on (a) (b) Al-8.81Zn-2.23Mg-2.43Cu (wt.%) [111].

Test number	First step	Second step
As cast	—	—
H1	420 °C × 36 h	—
H2	440 °C × 36 h	—
H3	466.5 °C × 36 h	—
H4	466.5 °C × 36 h	490 °C × 2 h
H5	466.5 °C × 36 h	490 °C × 14 h
H6	466.5 °C × 36 h	490 °C × 26.4 h

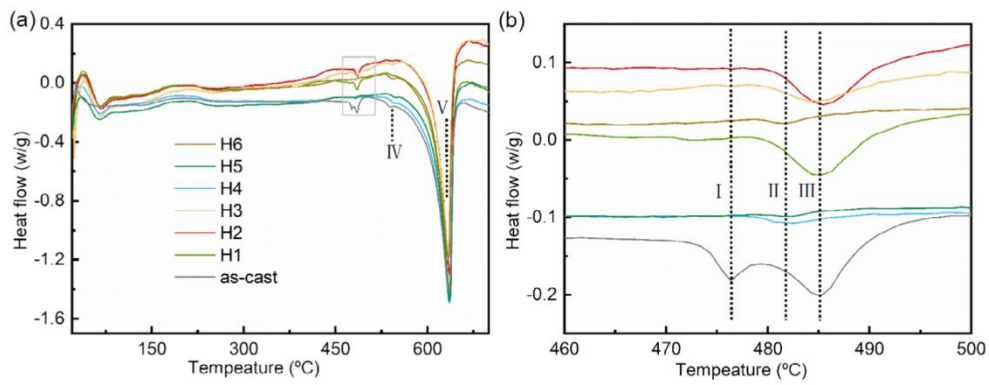


Figure 2- 31 (a) (b)DSC result of Al-6.43Zn-2.21Mg-2.15Cu (wt.%) [110].

Therefore, given the above, previous research has substantial room for improvement.

This experiment's design for multi-stage solution heat treatment aims to:

1. Enhance ductility without sacrificing strength.
2. Shorten the total solution time to optimize efficiency (less than 12 hours).

It meets both conditions to maximize the dissolution of second phases while avoiding incipient melting. At the same time, by comparing single-stage and multi-stage solution heat treatments with the same total duration, the benefits of multi-stage solution can be confirmed, and the internal microstructure of the material can be analyzed to clarify the reasons for the enhancement of material properties.

Chapter 3 Experiment Procedures and Instrument



3.1 Experiment Procedures

3.1.1 Materials

The materials used in this experiment were provided by China Steel Corporation (Kaohsiung, Taiwan) and consist of rolled materials based on 7075 wrought aluminum alloy. To these, 0.2wt% of Mn was added to form 7075Mn, and an additional 2wt% of Cu was added to create 7075MnCu. The primary difference between the two materials is the amount of Cu added. Hereafter, they are referred to as 7075LCu and 7075HCu to represent the two alloys.

Table 3- 1 Chemical composition of aluminum alloys used in the study

wt%	Al	Zn	Mg	Cu	Mn	Fe	Si	Ti	Cr
7075Mn	Bal.	5.98	2.86	1.61	0.24	0.23	0.08	0.04	0.22
7075MnCu	Bal.	5.91	2.83	1.98	0.24	0.22	0.08	0.04	0.23

3.1.2 Heat Treatment Designment

This experiment employs a heat treatment design in Figure 3-1 combining T4 and T6, involving quenching in water after solution treatment, natural aging at room temperature for 7 days, followed by artificial aging at 120 °C for 24 hours to achieve peak aging studied by previous research shown in Figure 3-2 [119]. The main variables in this experiment are the solution treatment time and temperature. Initially, a single-stage solution treatment at 490 °C for 3-24 h is performed, with increments of 3 hours, to compile data for comparison with two-stage solution treatments. The solution temperature is then divided into two stages: the first stage at 475 °C and the second stage at 490 °C, each for 3-12 h, aimed at maximizing dissolution of the η and S phases while preventing the formation of voids caused by incipient melting.

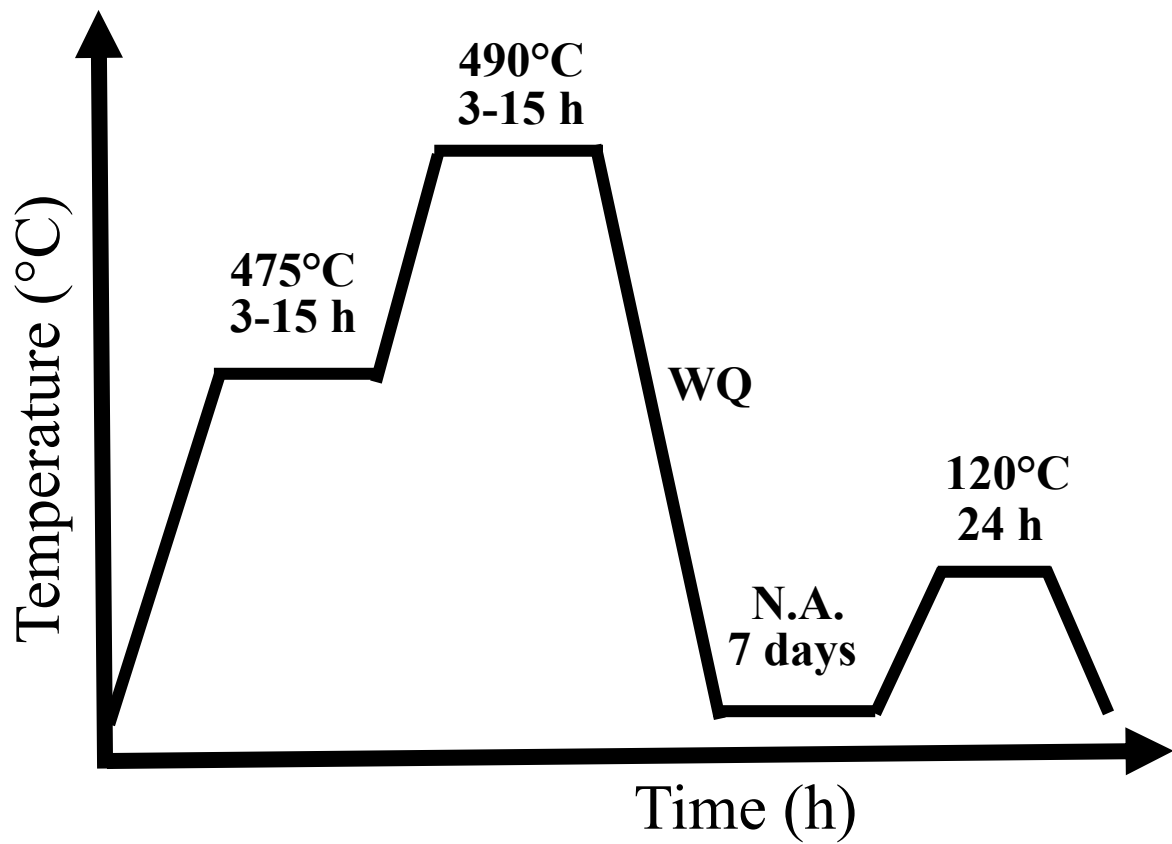
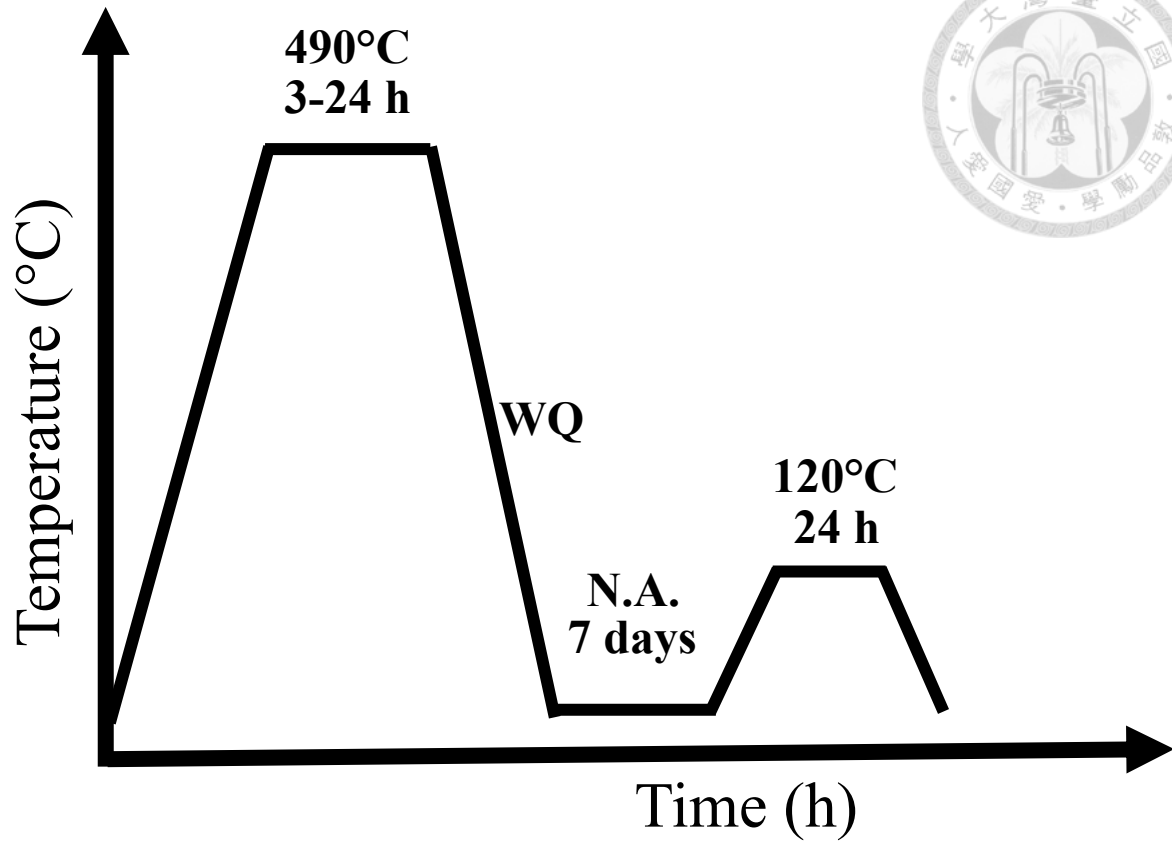


Figure 3- 1 Illustration of heat treatment used for the experiment.

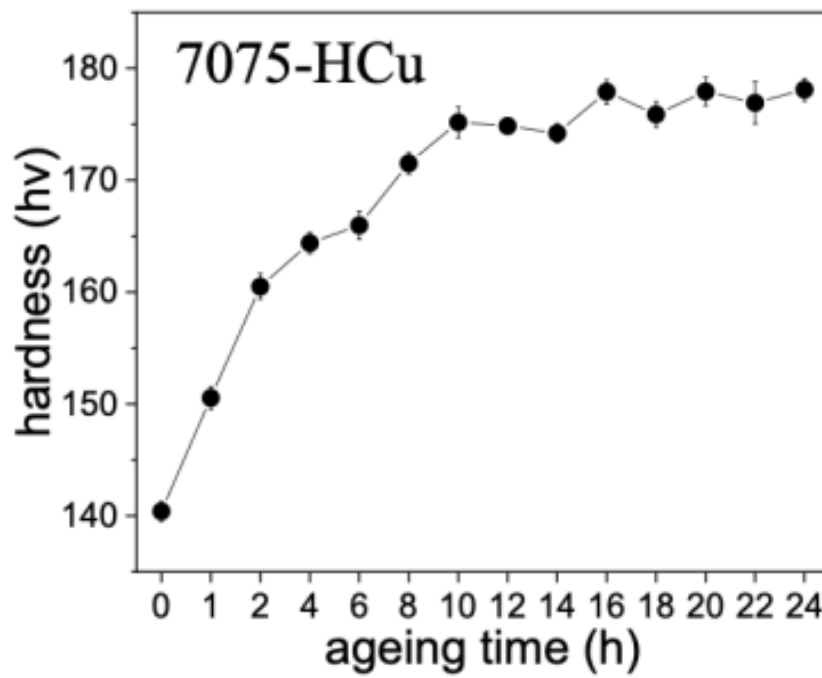
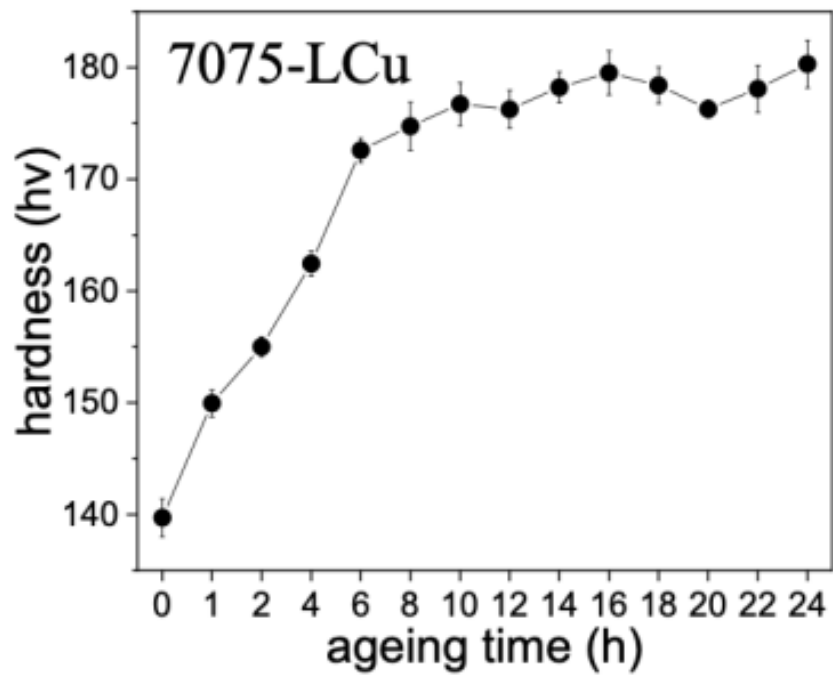


Figure 3- 2 Vickers hardness as a function in LCu and HCu specimens of the time ageing at 120 ° C [119].

3.2 Experiment Instructment and Specimens Preparation

3.2.1 Hardness Test

In this experiment, Vickers Hardness is used as the standard, employing a square-based pyramid-shaped diamond indenter to create impressions. The hardness is estimated by measuring the diagonal lengths of the impression at 500x magnification, calculating the area, and then converting it into depth. The instrument used is the FM-700 model microhardness tester from the Japanese company Future-Tech, with a set load of 500g and a duration of 8 seconds. The specimen preparation involved grounded with #1000, #1500, and #2500 mesh SiC paper sequentially, switching to a finer SiC paper once the scratches from the previous SiC paper were eliminated, commonly referred to as grounded to #2500. Finally, the specimen was polished with 3 μ m diamond paste before measuring hardness. To prevent the work hardening effect from artificially increasing material hardness, a minimum spacing of 25mm was maintained between each hardness indent, and 25 hardness tests were conducted under each condition. Extreme values were excluded, and the average was taken as the measured hardness of the material.

3.2.2 Tensile Test

The tensile specimens used in this experiment conform to the standard ASTM E8, with specifications as Figure 3-3 below. The tensile testing machine used is the MTS Landmark servo-hydraulic dynamic testing system located in the National Taiwan University Materials Mechanics Laboratory, capable of withstanding a maximum stress of 25k Newtons. The specimen preparation involved polishing the surface with #1000 mesh SiC paper until it was smooth before performing the tensile test. The tensile rate used was 10⁻³ strain per second, with a preload of 40 Newtons. Stress calculation was based on the force measured by the instrument divided by the effective cross-sectional

area of the specimen before stretching and corrected for the stress measured by the instrument after specimen fracture (corrected to 0). Strain was measured by laser distance measurement; the method involved adhering reflective tape within the effective stretching area and using laser distance measurement to determine the displacement during specimen stretching, divided by the original length of the specimen to calculate strain. This experiment did not use the instrument's strain measurements because it was found that the strain measured by the instrument was greater than that measured by laser distance, with an error of about 2-5% strain, as shown in the Figure 3-4 below. Since laser distance measurement reflects the actual movement distance of the specimen, it is considered more accurate, hence its use in this study.

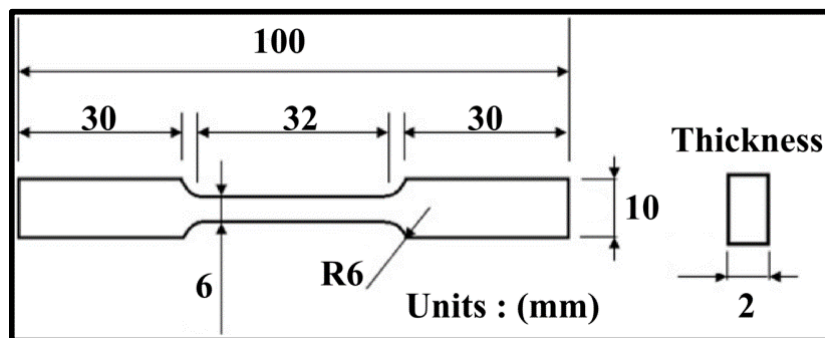


Figure 3- 3 The standard of ASTM E8 sample [120].

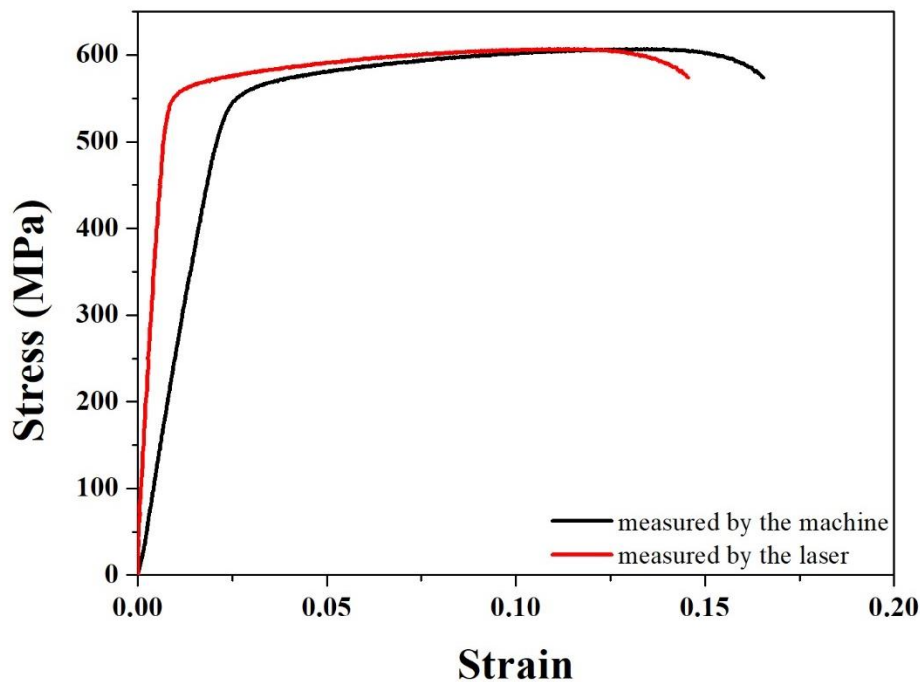


Figure 3- 4 The two tensile curve presents in the same tensile test.

3.2.3 Differential Scanning Calorimetry (DSC)

The DSC used in this experiment is the TA Q25 Differential Scanning Calorimeter from the Department of Chemical Engineering at National Taiwan University. Specimen preparation involved grounded to #2500 to remove the oxide layer, followed by cutting with a weight below 50mg. The temperature was raised to 350°C and held for 5 minutes, then increased at a rate of 2°C per minute up to 520 °C. This experiment utilizes DSC to measure the melting points of second phases to design the solution heat treatment process and to check for the presence of second phase signals after solution treatment.

3.2.4 X-Ray Diffraction (XRD)

The XRD used in this experiment is the Rigaku TTRAX III 18kW XRD, located at the Institute of Materials Science, National Taiwan University. Specimen preparation involved grounded the surface to #2500, followed by polishing with 3μm diamond paste. The scanning range was 2 theta from 15 to 65 degrees, with a scanning rate of 1 degree per minute and a step size of 0.01, without using a monochromator. This experiment utilizes XRD to measure the signals of second phases on the surface and to compare the differences in second phases before and after solution treatment, as well as between different Cu content aluminum alloys and aluminum alloy during solution treatment.

3.2.5 Scanning Electron Microscope (SEM)

In this experiment, the scanning electron microscopes (SEM) used are the FEI Nova NanoSEM 450 FEG-SEM, JEOL JXA-8530F PLUS FE-EPMA and the JeoL JSM-7800F Prime. Specimen preparation involved grounded the surface to #2500 mesh SiC paper, followed by polishing with 3μm and 1μm diamond pastes. Two imaging modes were used in SEM: secondary electron imaging (SEI) and backscattered electron imaging (BEI). Second electrons are electrons excited by the electron beam injected into the material,

with SEI images related to the surface morphology of the material. This experiment will use SEI to observe voids caused by incipient melting. BEI results from electrons backscattered by the material during interaction, with the brightness in images being positively related to the atomic number Z. Brighter areas in the images indicate regions with higher concentrations of heavier elements, which in this experiment are the locations of second phases. Different second phases have different proportions of heavier elements, resulting in varying brightness in the images.

3.2.6 Energy dispersive X-ray spectroscopy (EDS)

EDS involves the interaction of incident X-rays with the material to generate characteristic X-rays, analyzing the obtained X-ray spectrum to identify the elements within the material, providing semi-quantitative analysis. The preparation of EDS specimens is the same as that for SEM specimens, involving grounded the surface to #2500 mesh SiC paper, followed by polishing with 3 μ m and 1 μ m diamond pastes. In this experiment, EDS is used to observe surface particles and differentiate the types of second phases present. However, since EDS offers semi-quantitative analysis, more precise quantification and determination of the compositions of second phases will be conducted using EPMA.

3.2.7 Electron Back-Scattered Diffraction (EBSD)

The instrument used in this experiment is the EBSD (Electron Backscatter Diffraction) detector on the JeoL JSM-7800F Prime, with a tilt angle of about 70 degrees, operating voltage of 20kV, spot size of 5.5, and a step size resolution limit of approximately 80nm. The specimen preparation involved grounded to #2500 mesh SiC paper, followed by electrolytic polishing using a Twin-Jet Polish method at temperatures between -20°C to -25°C. The electrolyte solution was composed of 33% nitric acid and

67% methanol, under conditions of 20V voltage and approximately 40mA current for 1 minute and 30 seconds. Finally, the specimens were cleaned with anhydrous alcohol and isopropanol, and placed on the SEM stage with carbon tape. This experiment uses EBSD to observe grain size and orientation, to determine the changes in grains during solution heat treatment.

3.2.8 Electron Probe Micro Analyzer (EPMA)

The EPMA instrument used in this experiment is the JEOL JXA-8530F PLUS FE-EPMA. Specimen preparation involved grounded to #2500 mesh SiC paper, followed by polishing with 3 μ m and 1 μ m diamond pastes. EPMA allows for the observation of residual second phases not dissolved during solution treatment and the detection of voids caused by incipient melting. Element distribution visible in mapping can be used to identify the types of second phases. EPMA utilizes WDS (Wavelength Dispersive Spectroscopy) to differentiate characteristic X-rays, and its measurement depth is lower than that of EDS, reducing substrate effects. The precision in quantitative analysis is more than ten times higher than EDS, making it highly suitable for accurately determining the composition of second phases in this experiment.

3.2.9 Transmission Electron Microscope (TEM)

The TEM used in this experiment is a 200KV field emission gun transmission electron microscope (FEI Tecnai G² F20) and Thermo Fisher Spectra 300 Double-Corrected Transmission Electron Microscope, primarily for observing precipitates within aluminum alloys. The preliminary specimen preparation involved cutting the specimen into approximately 0.5mm thick slices using a cutting machine, grounded with #1000, #2000, and #2500 mesh SiC papers down to 100 μ m thickness before punching, further grounded to 60 μ m with #2500 mesh SiC paper, and then polishing using a Twin-Jet

Electropolishing method at temperatures between -20°C to -25°C . The electrolyte solution was 33% nitric acid and 67% methanol, with polishing conditions set at 15V voltage and approximately 20mA current, until the specimen perforated, detected by a photosensitive resistor, followed by cleaning with 99% anhydrous alcohol and isopropanol.

This experiment will utilize TEM to scan the thin areas of the specimen, observing along the $[110]_{\text{Al}}$ axis direction of the aluminum matrix. In Bright Field (BF) mode, it is possible to fully observe the size and morphology of nanoscale precipitates within the aluminum alloy. Combining High Resolution Transmission Electron Microscope (HRTEM) images and atomic-resolution image with Fast Fourier Transform (FFT) analysis of the precipitates' diffraction patterns will confirm the types, structures, and sizes of the precipitates.

This study used the ImageJ software to analyze the size of precipitates. Initially, the background was blurred, and only black precipitate areas were retained after background removal. Settings were adjusted to include only shapes with a circularity greater than 0.7 and an area greater than 2 nm^2 to exclude edge-on precipitates and noise interference. After calculating the area of the precipitates, corrections were made for the projection along the $[110]$ direction onto the $[100]$ plane at a cosine of 36.5 degrees. This adjustment allowed for the accurate calculation of the true area of the precipitates, which was then converted to the equivalent diameter. Each size measurement included statistics from hundreds of precipitates.

Chapter 4 Single-Stage and Two-stage solid solution treatment Experiment Result



4.1 Introduction

In Chapter 4, the experimental results of single-stage and two-stage solution treatments will be presented, comparing the differences between processes and between aluminum alloys with high and low copper content. This chapter will begin with tensile results. Given the complex mechanism of second phase dissolution and potential changes due to subsequent aging, the study directly attempts a large combination of tensile tests to identify the most suitable process. The study standardizes the peak aging conditions of the single-stage 490°C solution treatment, involving natural aging for 7 days followed by artificial aging at 120°C for 24 hours. Initially, the tensile results are based on a single-stage 490°C solution treatment, with three hours as a unit to establish a tensile database for comparison with two-stage solution treatments. Subsequently, for two-stage solution treatments, different 490°C solution times are tested with 475°C solution times ranging from 3 to 15 hours to collect mechanical properties and determine the best conditions.

Once the optimal two-stage solution treatment conditions for LCu and HCu are established, four conditions are analyzed: raw material, aging after only 475°C solution, aging after only 490°C solution, and aging after 475°C solution + 490°C solution. Firstly, DSC thermal flow and XRD peaks are used to determine the state of second phase dissolution under different solution conditions. EPMA scanning and WDS point analysis are then used to confirm the actual state of the second phases. Tensile fracture surfaces are examined to observe if second phases and voids are present. EBSD scanning of the aluminum matrix grain size is also performed to rule out grain refinement as a factor

altering mechanical properties due to different solution times. TEM imaging is used to observe the size and types of precipitates to ensure that different solution treatments do not alter precipitates under fixed peak aging conditions, thus not affecting precipitation hardening effects. By eliminating these two factors, it is confirmed that changes in mechanical properties are solely caused by second phases, further substantiating the benefits of two-stage solution treatments.

Finally, after verifying the mechanisms, a comparison is made between aluminum alloys with different copper contents under two-stage solution treatments to discuss the effects of increased copper content on aluminum alloys.

4.2 Tensile Test

4.2.1 As-received Aluminium Alloys

According to literature [83], a large number of second phases within the raw material before solution treatment can lead to poor mechanical properties of the material. Figure 4-1 shows the tensile curve of as-received LCu and HCu. Table 4-1 shows the tensile curve of as-received LCu and HCu. Actual tensile tests conducted on the raw material revealed that in LCu, the Yield Strength is 326.6 MPa, the Ultimate Tensile Strength is 329.1 MPa, and the elongation rate is 5.02%. In HCu, the YS is 352.9 MPa, the UTS is 363.2 MPa, and the elongation rate is 3.86%. It can be observed that in the raw material, HCu has higher strength than LCu, but its elongation rate is lower than that of LCu. Therefore, it is inferred that increasing the Cu element content in the aluminum alloy before solution treatment can increase the material's strength, but decrease the elongation rate.

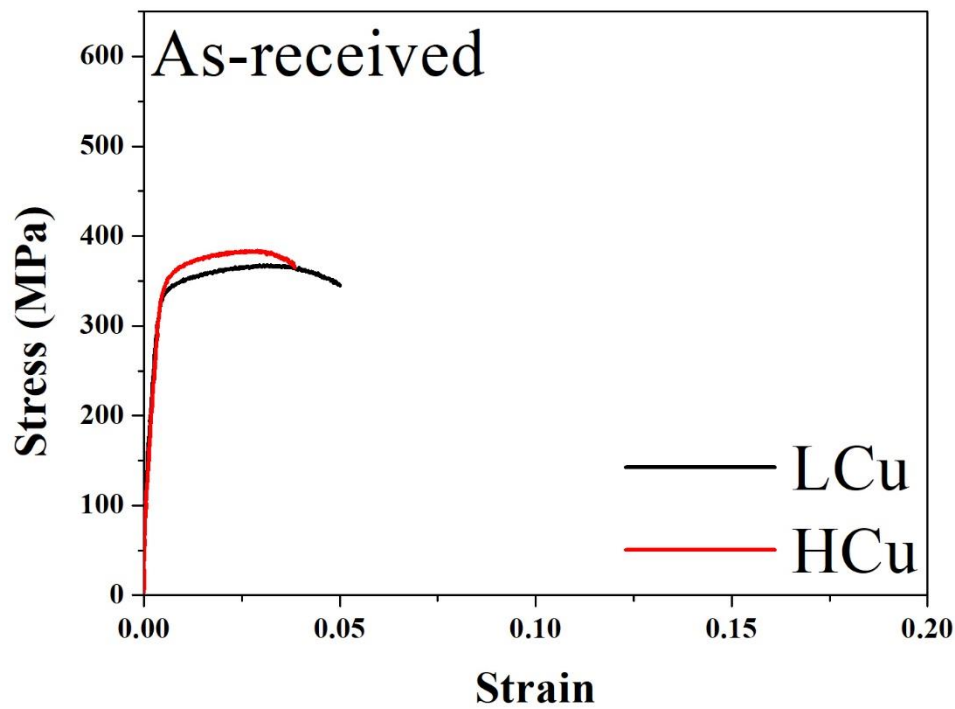


Figure 4-1 Stress-strain curve of as-received LCu and as-received HCu.

Table 4-1 Mechanical properties of as-received LCu and as-received HCu.

	YS(MPa)	UTS(MPa)	EL(%)
7075LCu	326.6	329.1	5.02
7075HCu	352.9	363.2	3.86

4.2.2 Single-Stage Solution Treatment

Table 4-2 to Table 4-4 and Figure 4-2 to Figure 4-4 shows that in the 490 °C single-stage solution treatment, LCu's overall Yield Strength is around 545 MPa and Ultimate Tensile Strength is about 600 MPa. The trend in strength increases with solution time, reaching a peak at about 15 hours of solution time with YS at 550 MPa and UTS at approximately 602 MPa, then slowly decreases to YS of 542 MPa and UTS of around

596 MPa at 24 hours of solution. The overall elongation rate is about 13.5% and tends to stabilize with time, going from an elongation rate of 11.4% at 3 hours of solution to 13.7% at 24 hours.

A similar trend for elongation rate only can be observed in HCu. The overall YS of HCu is around 540 MPa and UTS is about 595 MPa. After increasing solution time, there's no significant change in YS from 540 MPa at 3 hours of solution to 544 MPa at 15 hours, and finally, 542 MPa at 24 hours. UTS changes from 597 MPa at 3 hours to 599 MPa at 15 hours and ends up at 596 MPa at 24 hours of solution, showing little difference. The overall EL for HCu is about 13%, with the trend increasing slightly from an elongation rate of 12.6% at 3 hours of solution to 13.9% at 21 hours, similar to LCu but less pronounced.

Furthermore, both strength and ductility are lower in HCu compared to LCu, consistent with literature stating that adding an excessive amount of the same element reduces mechanical properties. The trend might be due to second phases dissolving more completely with increased solution time, but after most dissolvable second phases have dissolved, increasing solution time has no special benefit and does not solve the problem of incipient melting, hence no further improvement in ductility. This will be discussed further in EPMA analysis. Besides, increasing solution time not only dissolves second phases but also affects grain size, possibly leading to grain coarsening as the cause of strength peaking and then decreasing, which will be further discussed in EBSD analysis. After understanding the mechanical properties of single-stage solution treatment, an attempt will be made to divide the total time into two-stage solution treatments to clarify whether two-stage solution processes are superior to single-stage solution processes.

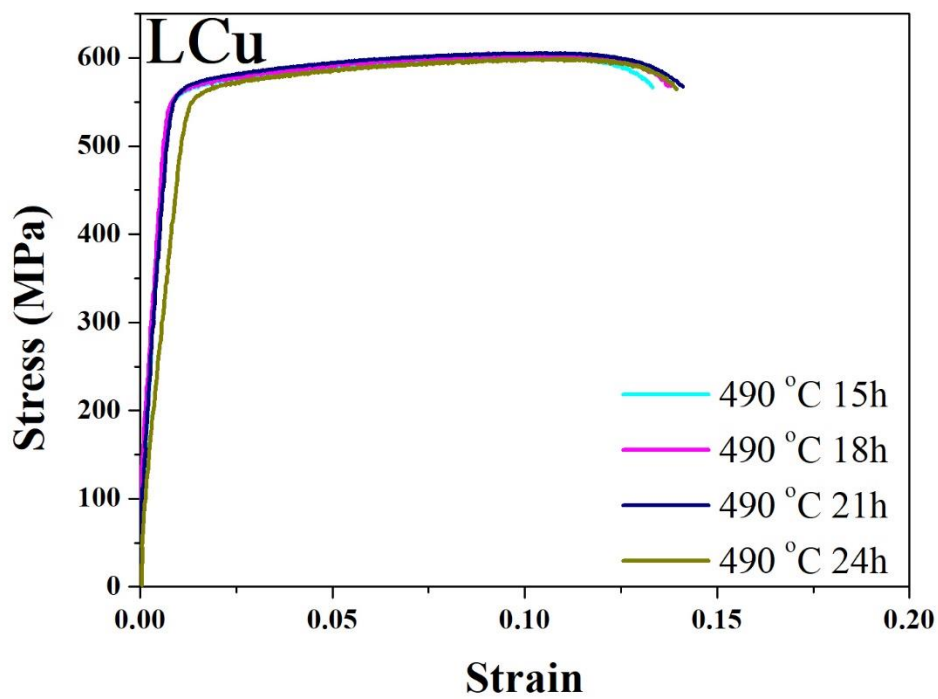
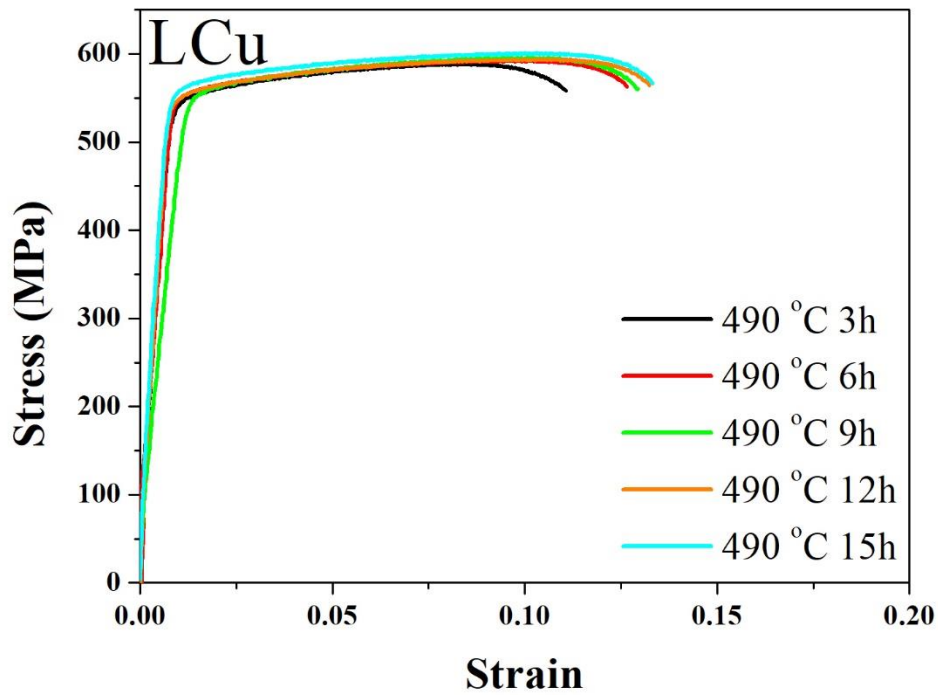


Figure 4-2 Tensile curves of single-stage solution treatment for LCu on the condition peak ageing at 120 °C.

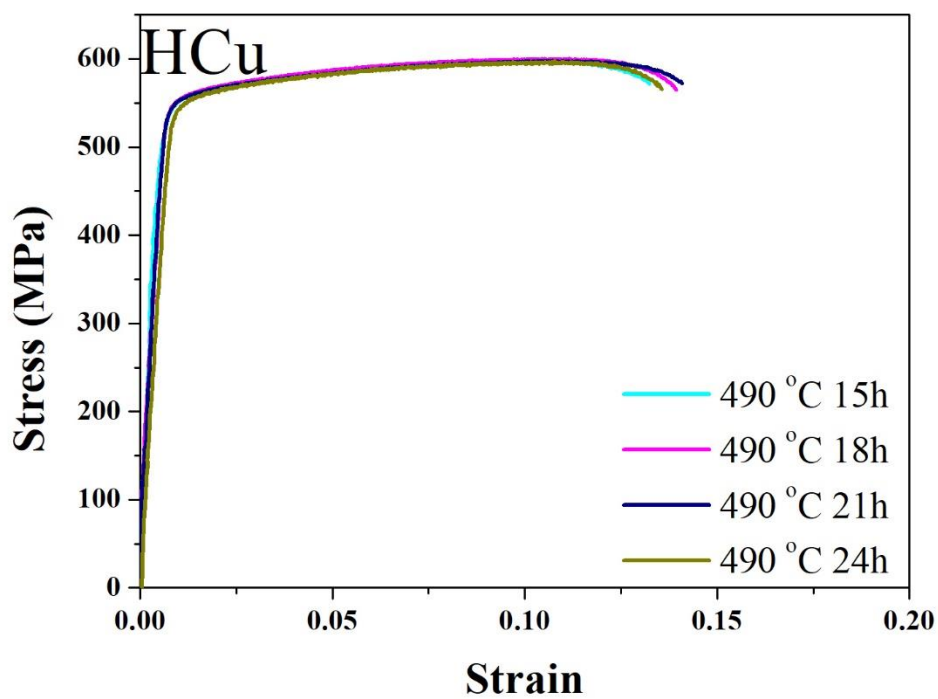
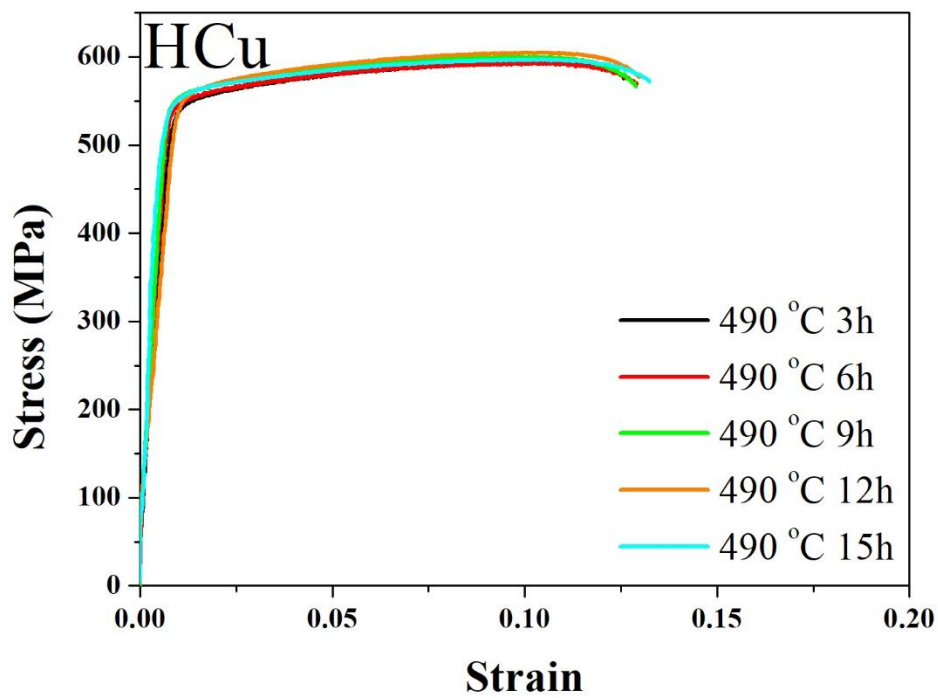


Figure 4-3 Tensile curves of single-stage solution treatment for HCu on the condition peak ageing at 120 °C.

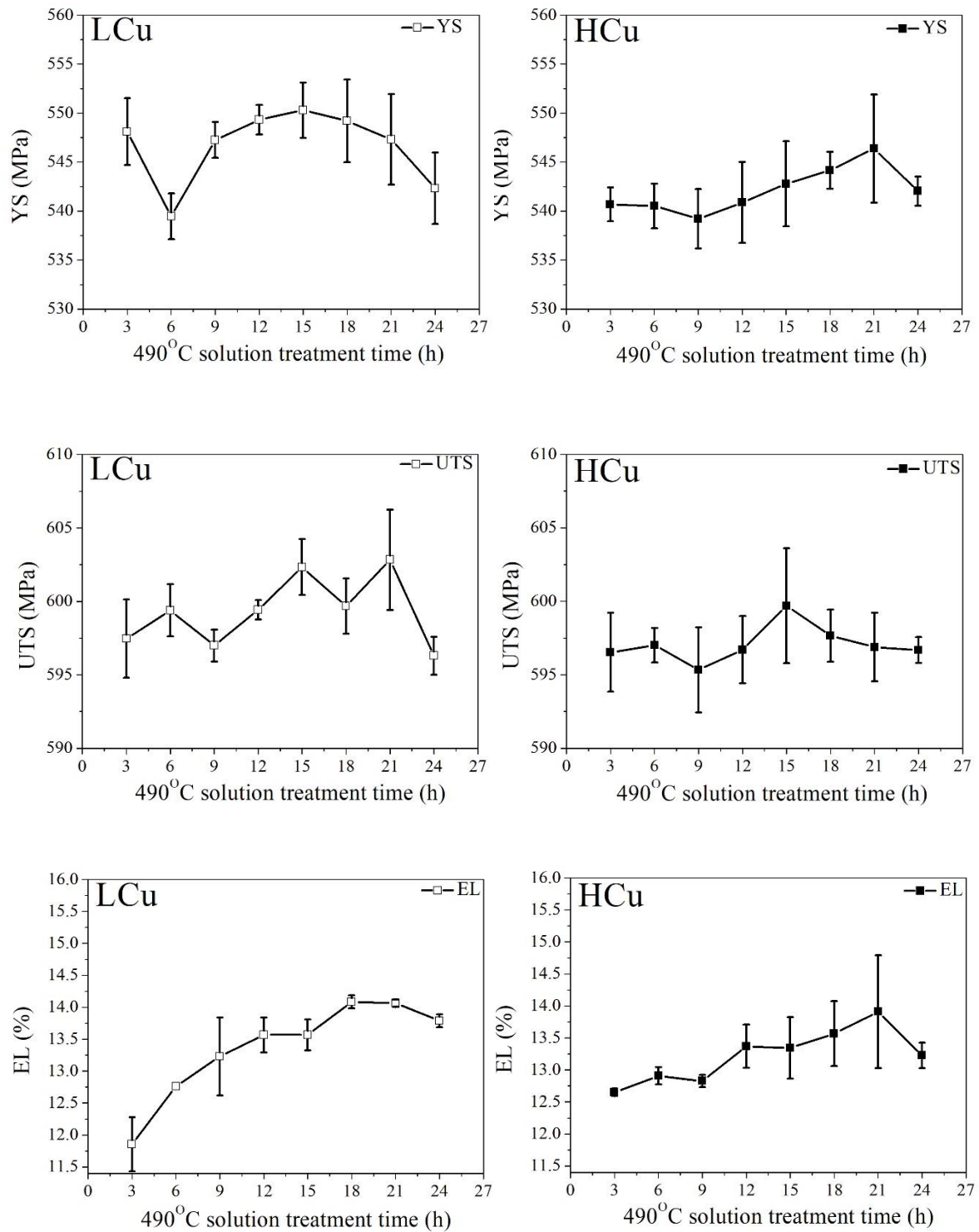


Figure 4-4 Mechanical properties of single-stage solution treatment for LCu and HCu on the condition peak ageing at 120 °C.

Table 4-2 YS of single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.

Time (h) \ alloy	7075-LCu (MPa)	7075-HCu (MPa)
St.+NA+AA		
490°C 3h	548	540
490°C 6h	539	540
490°C 9h	547	539
490°C 12h	549	540
490°C 15h	550	542
490°C 18h	549	544
490°C 21h	547	546
490°C 24h	542	542

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24 h.

Table 4-3 UTS of single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.

Time (h) \ alloy	7075-LCu (MPa)	7075-HCu (MPa)
St.+NA+AA		
490°C 3h	597	596
490°C 6h	599	597
490°C 9h	596	595
490°C 12h	599	596
490°C 15h	602	599
490°C 18h	599	597
490°C 21h	602	596
490°C 24h	596	596

Table 4-4 EL of single-stage solution treatment for LCu and HCu with peak ageing at 120 °C

Time (h) \ alloy	7075-LCu (%)	7075-HCu (%)
St.+NA+AA		
490°C 3h	11.8	12.6
490°C 6h	12.7	12.9
490°C 9h	13.2	12.8
490°C 12h	13.5	13.3
490°C 15h	13.5	13.3
490°C 18h	14.0	13.5
490°C 21h	14.0	13.9
490°C 24h	13.7	13.2

4.2.3 Two-Stage Solution Treatment

In this experiment, to avoid excessively long solution times, 12 hours at 490 °C was chosen as a baseline and then split time into a two-stage solution treatment at 475 °C for the first stage and 490 °C for the second stage, cumulatively spanning 12 hours, to observe if two-stage solution treatment yields better mechanical properties. From Figure 4-5 to Figure 4-8 and Table 4-5 to Table 4-7, the experimental results showed improvements in both strength and ductility with two-stage solution heat treatment, surpassing the properties achieved with a single-stage process. These preliminary findings confirm the benefits of two-stage solution treatment, indicating that the enhancement in mechanical properties is not merely due to extended solution times. Comparatively, LCu also outperformed HCu. After confirming the effectiveness of the two-stage process, the aim is to optimize the results. Therefore, using 3 hours as a unit, the experiment seeks to identify the heat treatment process that achieves the best mechanical properties in the shortest possible time.

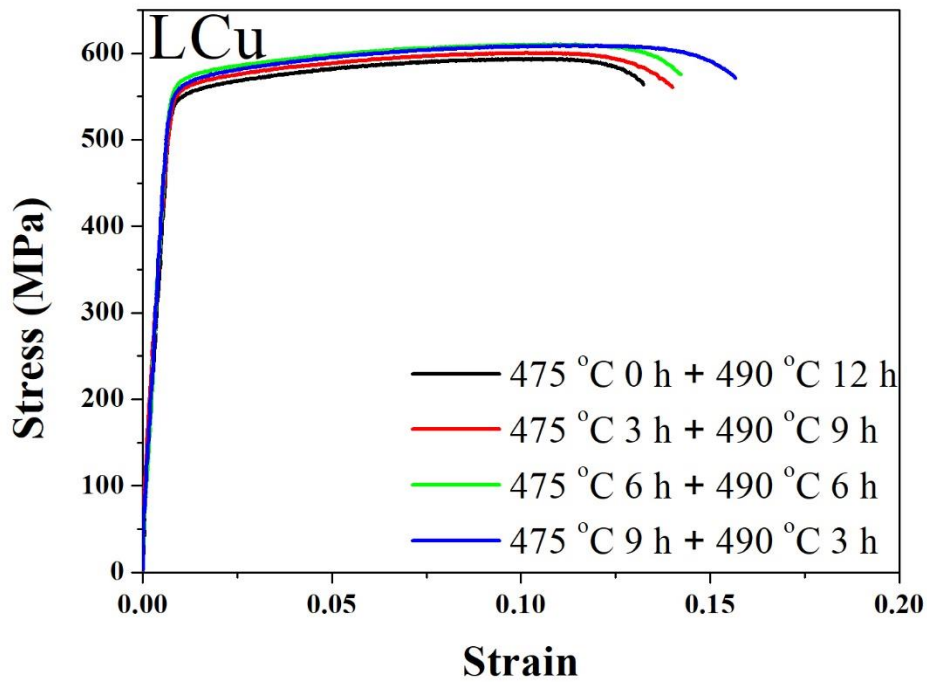


Figure 4-5 Tensile curves of two-stage and single-stage solution treatment for LCu on the condition peak ageing at 120 °C.

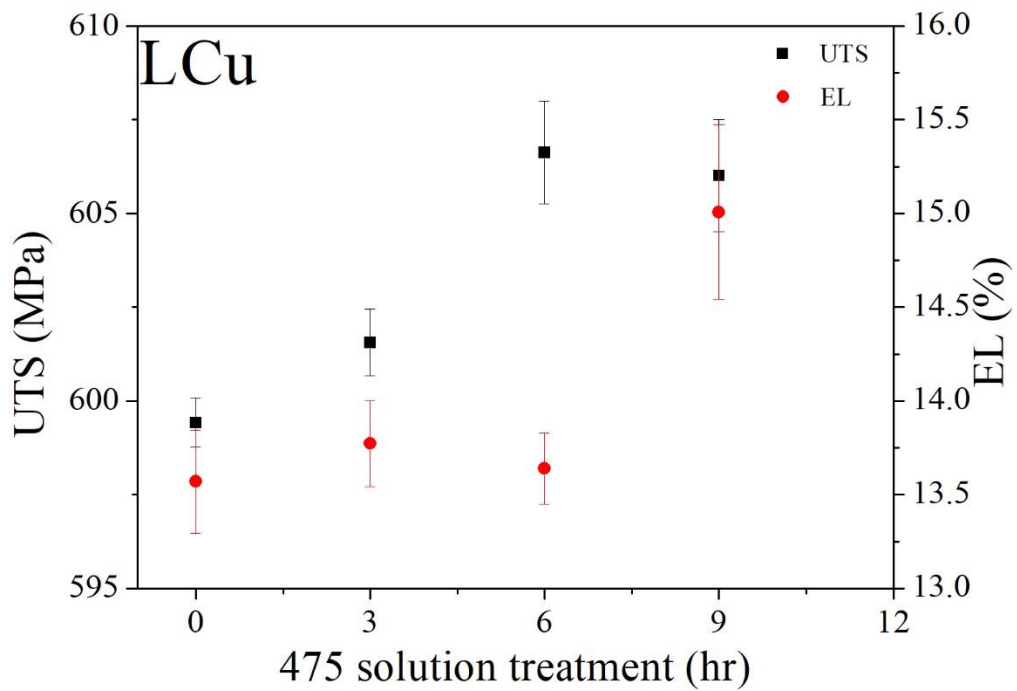


Figure 4-6 Mechanical properties of two-stage and single-stage solution treatment for LCu on the condition peak ageing at 120 °C.

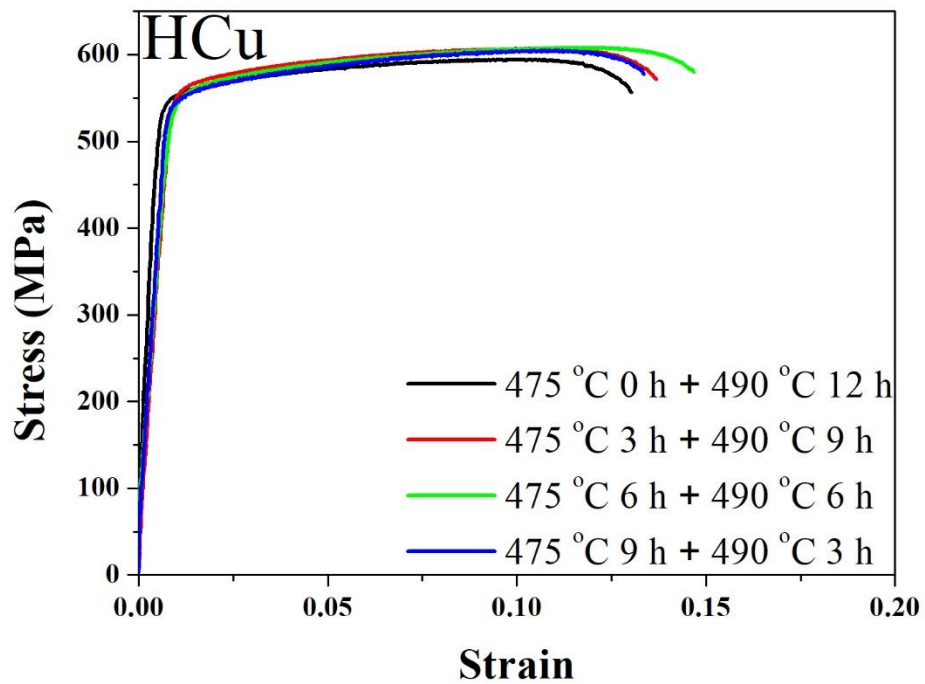


Figure 4-7 Tensile curves of two-stage and single-stage solution treatment for HCu on the condition peak ageing at 120 °C.

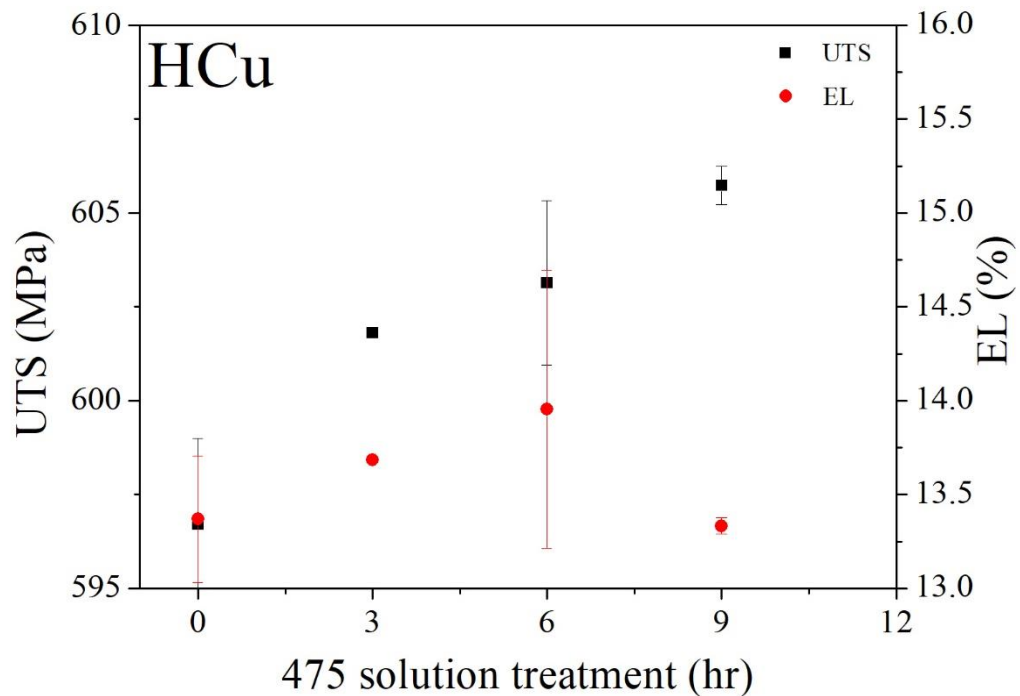


Figure 4-8 Mechanical properties of two-stage and single-stage solution treatment for HCu on the condition peak ageing at 120 °C.

Table 4-5 YS of two-stage and single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475°C 0 h + 490°C 12 h	549	540
475°C 3 h + 490°C 9 h	551	546
475°C 6 h + 490°C 6h	551	541
475°C 9 h + 490°C 3h	551	546

Table 4-6 UTS of two-stage and single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475°C 0 h + 490°C 12 h	599	596
475°C 3 h + 490°C 9 h	601	601
475°C 6 h + 490°C 6h	606	603
475°C 9 h + 490°C 3h	606	605

Table 4-7 EL of two-stage and single-stage solution treatment for LCu and HCu with peak ageing at 120 °C.

St. + NA + AA	7075-LCu (%)	7075-HCu (%)
475°C 0 h + 490°C 12 h	13.5	13.3
475°C 3 h + 490°C 9 h	13.7	13.6
475°C 6 h + 490°C 6h	13.6	13.9
475°C 9 h + 490°C 3h	15.0	13.3

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24 h.

4.2.4 Two-Stage Solution Treatment with 475 °C 0-15 h + 490 °C 3 h

To achieve the best mechanical properties of the two-stage solution treatment, the second stage is fixed at 490 °C for 3 hours, and the first stage at 475 °C for 0, 3, 6, 9, 12, and 15 hours is varied to observe changes in mechanical properties. Figure 4-9 and Figure 4-10 shows the tensile curve. Table 4-8 to Table 4-10 represents the mechanical properties. In the first condition, where the solution at 475 °C lasts for 0 hours, equivalent to single-stage solid solution treatment at 490°C, the charts reveal that both LCu and HCu show significant improvements in mechanical properties as the solution time at 475 °C increases, marking the transition from single-stage to two-stage solution treatment. Specifically, LCu shows notable increases in Ultimate Tensile Strength and Elongation, rising from 597 MPa and 11.8% to 605 MPa and 14.1%, respectively, with Yield Strength also experiencing a slight increase from 548 MPa to 551 MPa. HCu also sees improvements in UTS and EL, going from 597 MPa and 12.6% to 603 MPa and 13.6%, respectively, with YS increasing from 540 MPa to 543 MPa. These results prove the benefits of two-stage solution treatment in enhancing mechanical properties. Under the fixed condition of 3 hours of solution at 490 °C, in LCu, as the solution time at 475 °C increases, strength and ductility rise to a certain level and then plateau, with UTS and EL reaching up to 605 MPa and 15%, respectively. In HCu, with the increase in solution time at 475°C, strength and ductility initially increase and then decrease, with the maximum YS, UTS, and EL reaching 552MPa, 607MPa, and 13.7%, respectively. This suggests that there is an optimal time range at the 475°C solution stage for efficiently improving HCu's mechanical properties, and simply increasing solution time does not enhance properties.

Comparatively, LCu surpasses HCu in YS, UTS, and especially in ductility, with LCu's ductility reaching up to 15.0%, whereas HCu only achieves 13.7%. In terms of strength, LCu's YS can reach up to 557 MPa, whereas HCu's YS is only up to 552 MPa.

LCu's UTS can reach up to 606 MPa, while HCu's UTS also can reach 607 MPa. Thus, it is inferred that for aluminum alloys undergoing two-stage solution heat treatment, increasing Cu content may lead to a decline in mechanical properties.

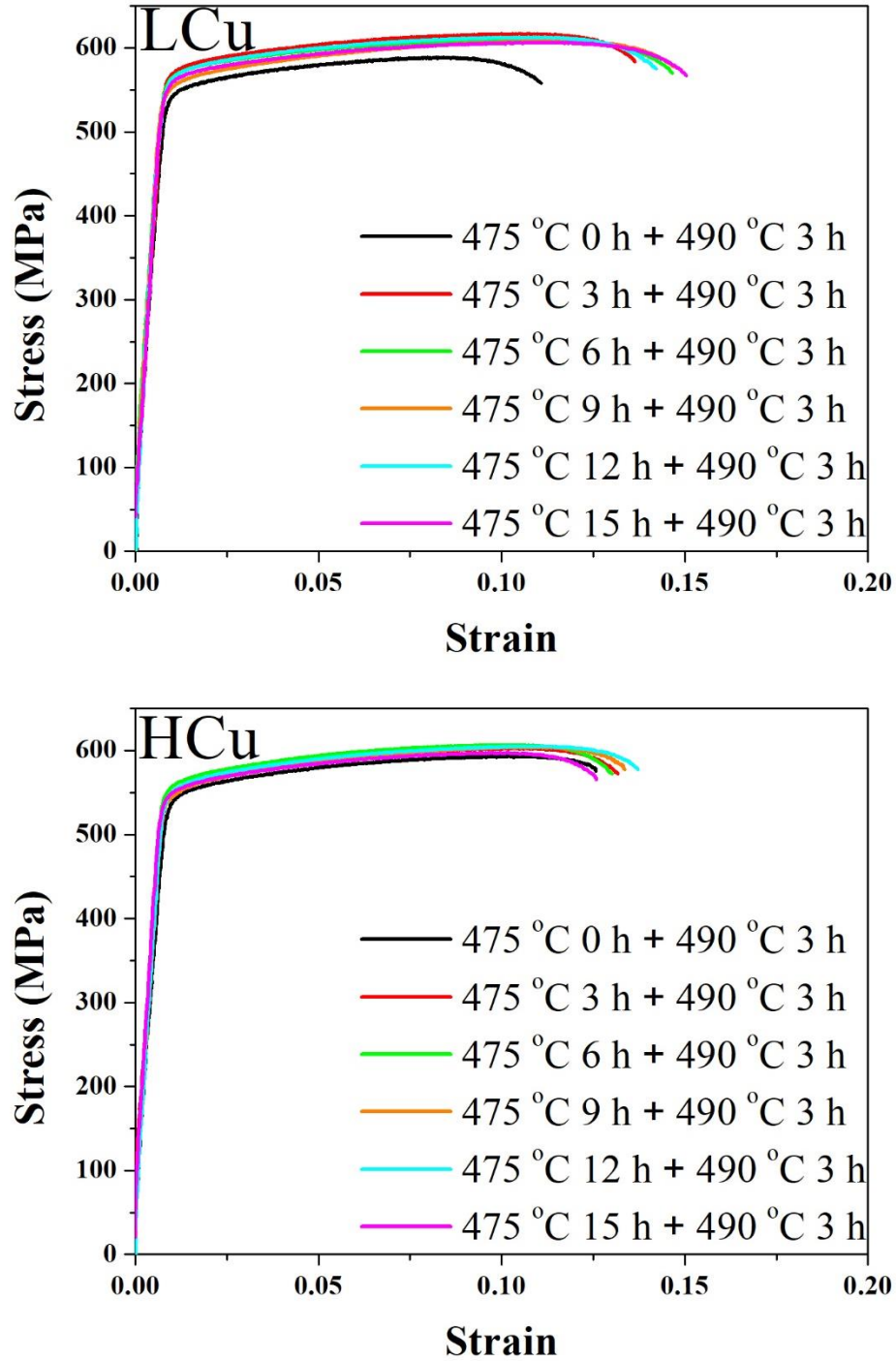
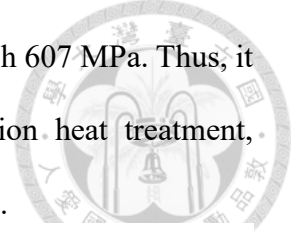


Figure 4-9 Tensile curves of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.

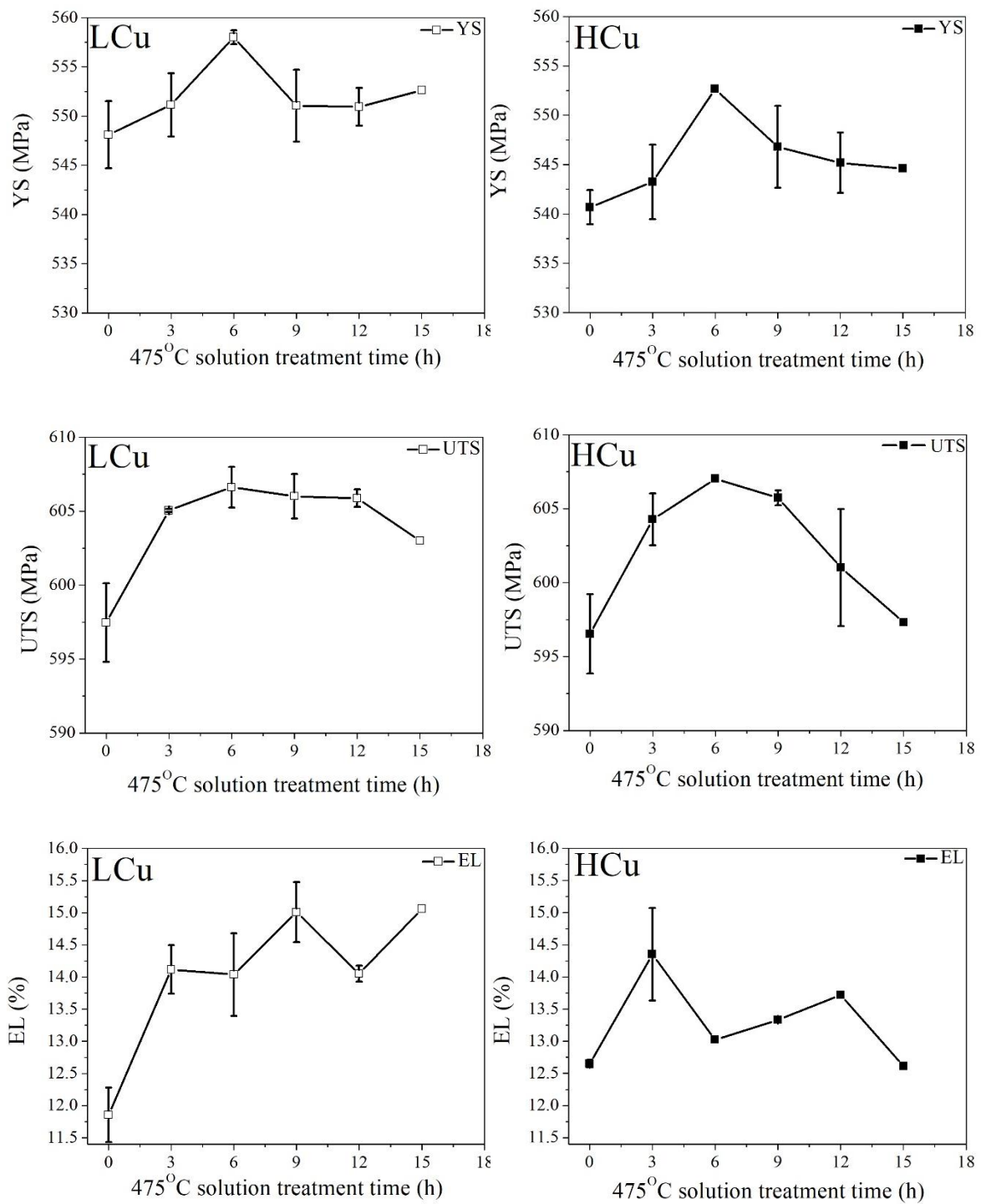


Figure 4-10 Mechanical properties of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.

Table 4-8 YS of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475°C 0 h + 490°C 3 h	548	540
475°C 3 h + 490°C 3 h	551	543
475°C 6 h + 490°C 3 h	557	552
475°C 9 h + 490°C 3 h	551	546
475°C 12 h + 490°C 3 h	550	545
475°C 15 h + 490°C 3 h	552	544

Table 4-9 UTS of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475°C 0 h + 490°C 3 h	597	596
475°C 3 h + 490°C 3 h	605	604
475°C 6 h + 490°C 3 h	606	607
475°C 9 h + 490°C 3 h	606	605
475°C 12 h + 490°C 3 h	605	601
475°C 15 h + 490°C 3 h	603	597

Table 4-10 EL of two-stage solution treatment with second stage 490 °C 3 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (%)	7075-HCu (%)
475°C 0 h + 490°C 3 h	11.8	12.6
475°C 3 h + 490°C 3 h	14.1	14.3
475°C 6 h + 490°C 3 h	14.0	13.0
475°C 9 h + 490°C 3 h	15.0	13.3
475°C 12 h + 490°C 3 h	14.0	13.7
475°C 15 h + 490°C 3 h	15.0	12.6

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24 h.

4.2.5 Two-Stage Solution Treatment with 475 °C 0-15 h + 490 °C 6 h

Next, fix the second stage at 490 °C for 6 hours, and vary the first stage at 475 °C for 0, 3, 6, 9, 12, and 15 hours to observe changes in mechanical properties. Figure 4-10 and Figure 4-11 shows the tensile curve. Table 4-11 to Table 4-13 represents the mechanical properties. Under the condition of increasing the second stage solution time to 6 hours at 490 °C, the mechanical properties of the two-stage solution process are also superior to those of the single-stage solution process. Similar to the condition of 3 hours at 490 °C, after adding the 475 °C stage, LCu's YS, UTS, and EL all see significant improvements. Especially in terms of elongation rate, YS and UTS increased from 539 MPa and 599 MPa to 551 MPa and 604 MPa, respectively, and EL from 12.7% to 14.2%. This indirectly proves that the second phases have a better solution condition. With the increase in the first stage solution time at 475°C, both strength and elongation rate show an upward trend, similar to the condition of 3 hours at 490°C, reaching up to 605 MPa and 14.8%. However, the increase is not as significant as in the condition of 3 hours at 490 °C, presumably because the total solution time under the condition of 6 hours at 490 °C is longer than that of 3 hours at 490 °C. In the single-stage solution condition, as the solution time increases, strength and elongation rate tend to plateau or even decrease. Thus, with a longer total solution time than the 3 hours at 490 °C condition, the trend is naturally less pronounced.

For HCu, although YS and UTS show an increase and then a decrease, reaching up to 547 MPa and 603 MPa respectively, the overall change in EL is not significant, only increasing from 13.2% to 13.9%. Extending the solution time further results in a decrease in elongation rate, suggesting that extending the total solution time and adding the 475°C stage in two-stage solution may not further improve the elongation rate of HCu. Comparing the two materials, LCu's strength and elongation rate are superior to those of

HCu, leading to the inference that two-stage solution may be less effective in aluminum alloys with a high Cu content.

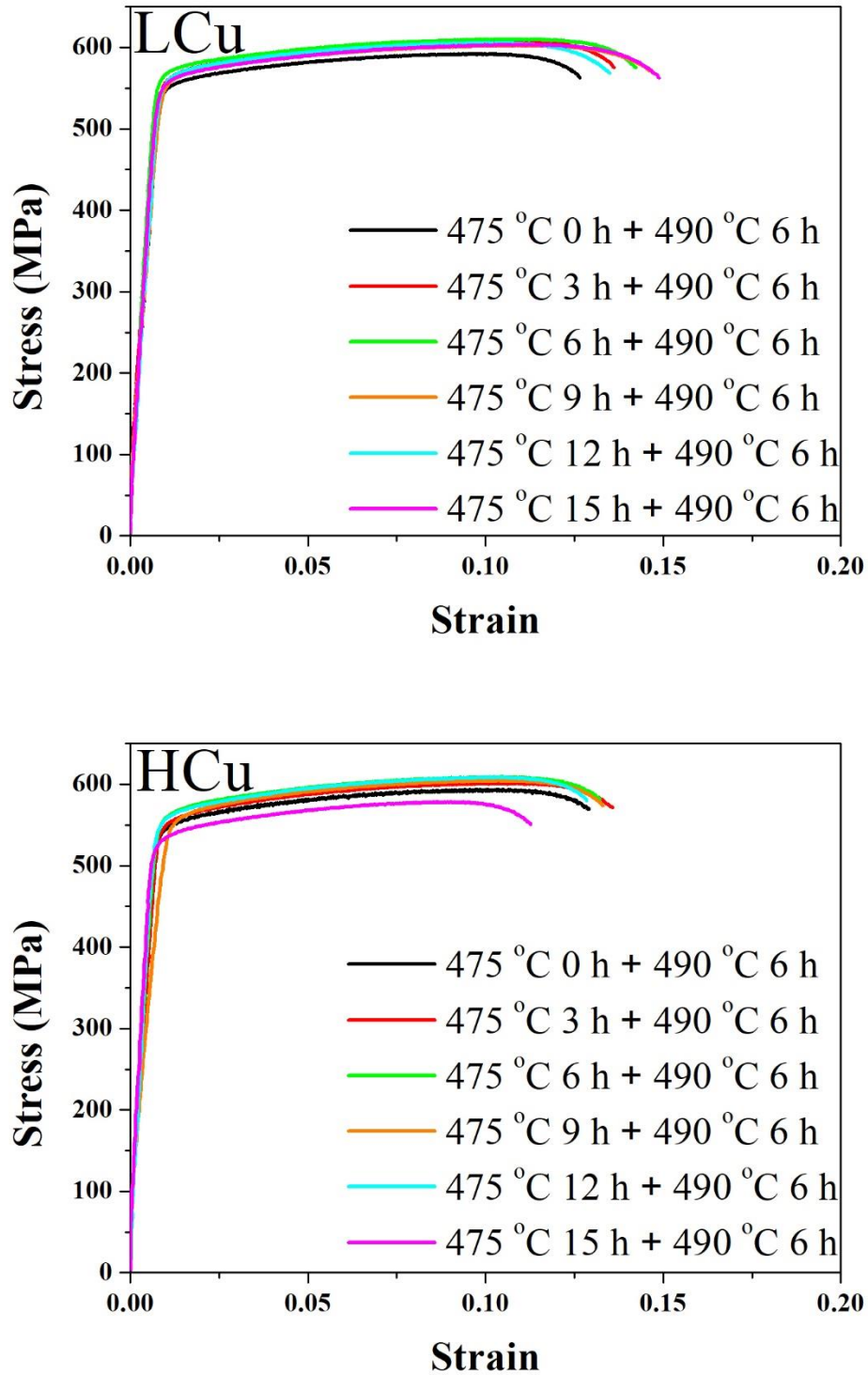


Figure 4-11 Tensile curves of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.

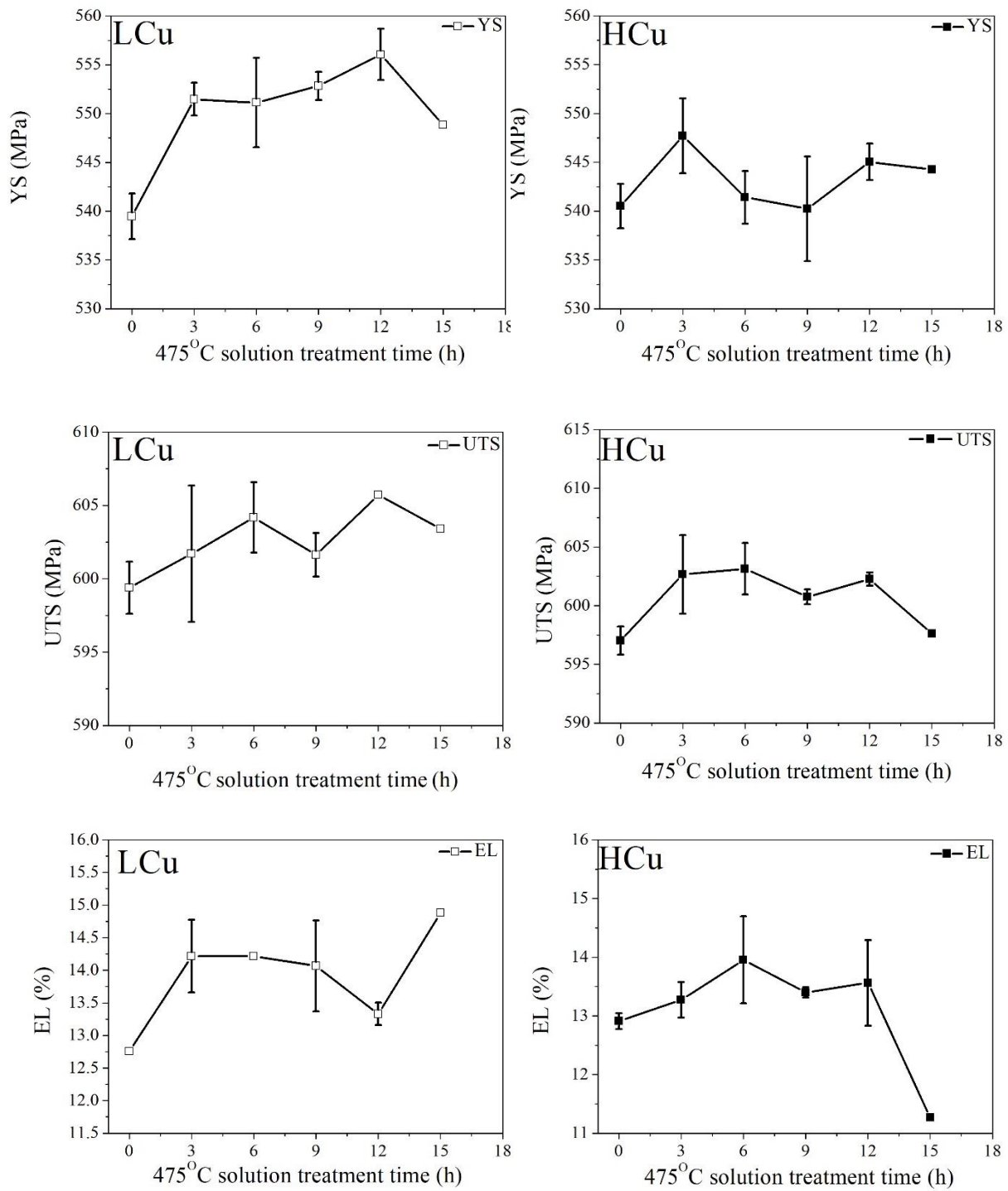


Figure 4-12 Mechanical properties of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak aging at 120 °C.

Table 4-11 YS of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475 °C 0 h + 490 °C 6 h	539	540
475 °C 3 h + 490 °C 6 h	551	547
475 °C 6 h + 490 °C 6 h	551	541
475 °C 9 h + 490 °C 6 h	552	540
475 °C 12 h + 490 °C 6 h	556	545
475 °C 15 h + 490 °C 6 h	548	544

Table 4-12 UTS of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475°C 0 h + 490°C 6 h	599	597
475°C 3 h + 490°C 6 h	601	602
475°C 6 h + 490°C 6 h	604	603
475°C 9 h + 490°C 6 h	601	600
475°C 12 h + 490°C 6 h	605	602
475°C 15 h + 490°C 6 h	603	597

Table 4-13 EL of two-stage solution treatment with second stage 490 °C 6 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (%)	7075-HCu (%)
475 °C 0 h + 490 °C 6 h	12.7	12.9
475 °C 3 h + 490 °C 6 h	14.2	13.2
475 °C 6 h + 490 °C 6 h	14.2	13.9
475 °C 9 h + 490 °C 6 h	14.0	13.4
475 °C 12 h + 490 °C 6 h	13.3	13.5
475 °C 15 h + 490 °C 6 h	14.8	11.2

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24h.

4.2.6 Two-Stage Solution Treatment with 475 °C 0-15 h + 490 °C 9 h

Besides, fix the second stage at 490 °C for 6 hours, and vary the first stage at 475 °C for 0, 3, 6, 9, 12, and 15 hours to observe changes in mechanical properties. Figure 4-13 and Figure 4-14 shows the tensile curve. Table 4-14 to Table 4-16 represents the mechanical properties. Under the condition of 9 hours of solution at 490 °C, the charts show that, consistent with earlier results, adding the first stage solution at 475 °C leads to a definite improvement in LCu's YS, UTS, and EL. YS and UTS increased from 547 MPa and 596 MPa to 551 MPa and 601 MPa, respectively, while EL increased from 13.2% to 13.7%. This underscores the benefits of two-stage solution treatment. With the extension of the first stage solution time, the mechanical properties show a similar trend as before, increasing with solution time until plateauing, with the maximum YS, UTS, and EL reaching 556 MPa, 605 MPa, and 14.8%, respectively. HCu also displays a similar trend to the condition of 6 hours at 490 °C, where two-stage solution treatment shows superiority in strength over single-stage treatment, with values first rising to a peak before declining. The peak values reached were 551 MPa for YS and 604 MPa for UTS. In terms of EL, a slight variation is observed, with EL gradually decreasing after peaking, differing only marginally from 12.8% at 9 hours of 490°C solution to the maximum elongation rate of 13.6% with 3 hours of 475 °C solution followed by 3 hours of 490 °C solution, eventually settling at 12.9% for 15 hours of 475 °C solution followed by 3 hours of 490 °C solution, suggesting that the benefit of adding 475 °C stage in two-stage solution treatment on HCu is minimal in longer periods.

It is inferred that the Cu content plays a role, with LCu superior in both strength and elongation rate to HCu. Particularly in terms of elongation rate, aluminum alloys with lower copper content perform better, with LCu reaching up to 14.8%, whereas HCu only reaches 13.6%. In terms of strength, the maximum YS for LCu can reach up to 556 MPa,

while for HCu, it is up to 551 MPa. For UTS, LCu can reach up to 605 MPa, while HCu can reach 604 MPa, with a smaller difference than in elongation rate.

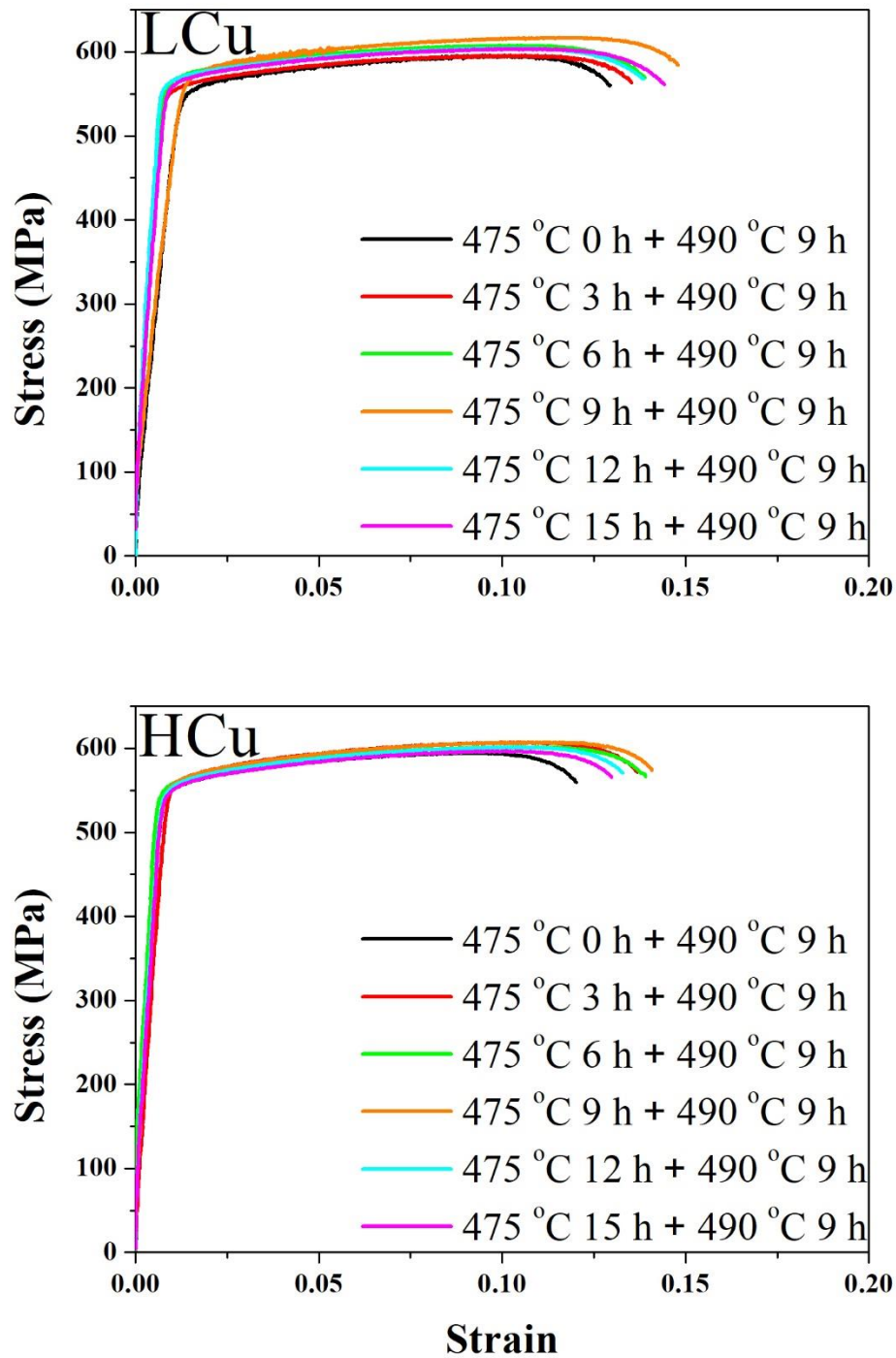
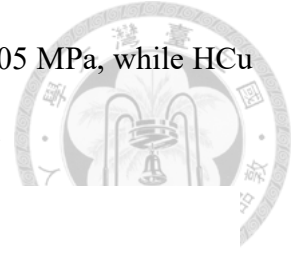


Figure 4-13 Tensile curves of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.

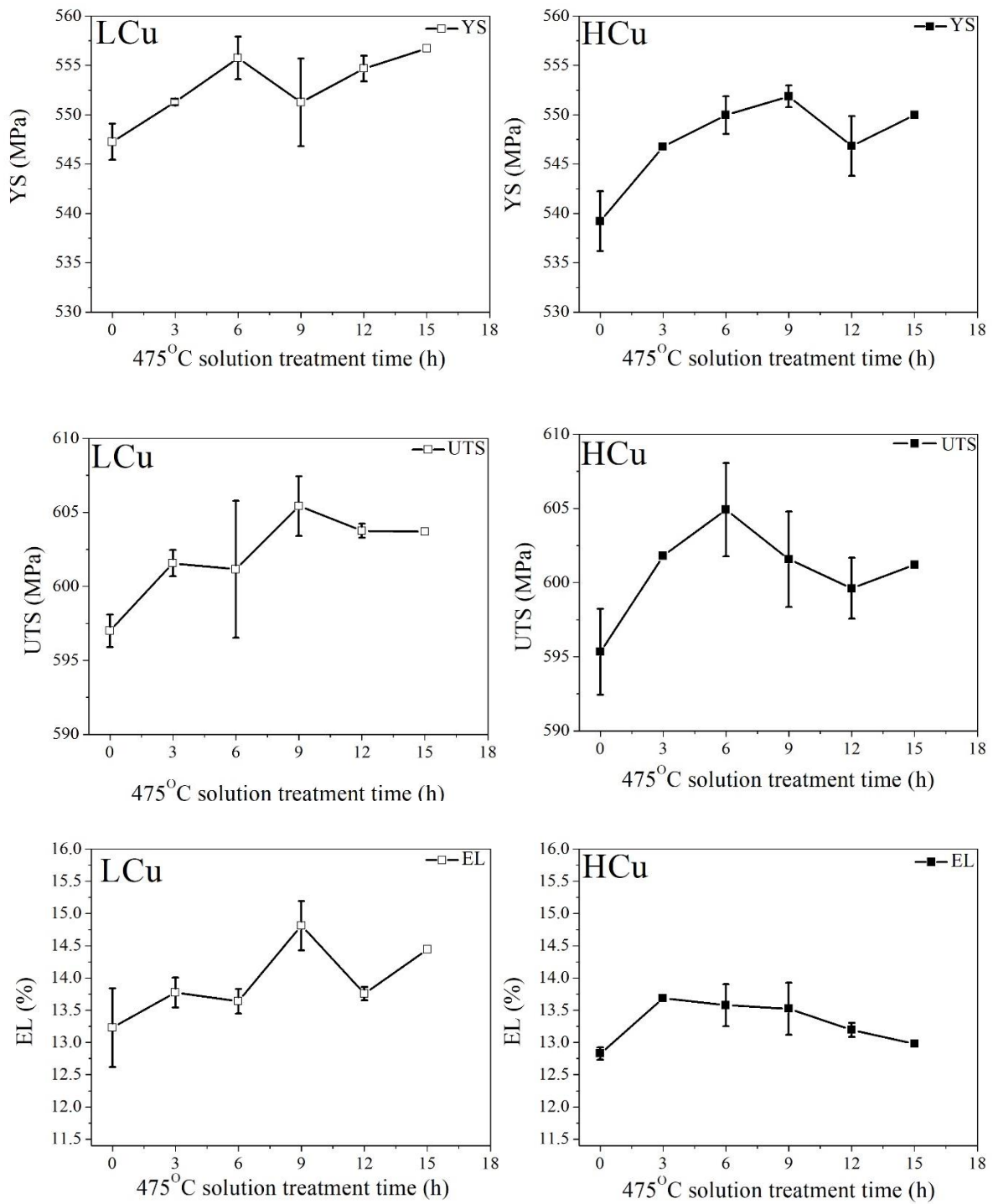


Figure 4-14 Mechanical properties of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.

Table 4-14 YS of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475 °C 0 h + 490 °C 9 h	547	539
475 °C 3 h + 490 °C 9 h	551	546
475 °C 6 h + 490 °C 9 h	555	549
475 °C 9 h + 490 °C 9 h	551	551
475 °C 12 h + 490 °C 9 h	554	546
475 °C 15 h + 490 °C 9 h	556	549

Table 4-15 UTS of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475 °C 0 h + 490 °C 9 h	596	595
475 °C 3 h + 490 °C 9 h	601	601
475 °C 6 h + 490 °C 9 h	601	604
475 °C 9 h + 490 °C 9 h	605	601
475 °C 12 h + 490 °C 9 h	603	599
475 °C 15 h + 490 °C 9 h	603	601

Table 4-16 EL of two-stage solution treatment with second stage 490 °C 9 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (%)	7075-HCu (%)
475 °C 0 h + 490 °C 9 h	13.2	12.8
475 °C 3 h + 490 °C 9 h	13.7	13.6
475 °C 6 h + 490 °C 9 h	13.6	13.5
475 °C 9 h + 490 °C 9 h	14.8	13.5
475 °C 12 h + 490 °C 9 h	13.7	13.1
475 °C 15 h + 490 °C 9 h	14.4	12.9

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24h.

4.2.7 Two-Stage Solution Treatment with 475 °C 0-15 h + 490 °C 12 h

Finally, to avoid an overly long total solution treatment time, with a goal of keeping the total duration below 24 hours, the second stage is fixed at 490 °C for 12 hours. The first stage at 475 °C for 0, 3, 6, 9, and 12 hours is varied to observe changes in mechanical properties. Figure 4-15 and Figure 4-16 shows the tensile curve. Table 4-17 to Table 4-19 represents the mechanical properties. Under the condition of 12 hours of solution at 490 °C, the charts show a consistent trend with increased second stage solution time, but with diminished magnitude of change. In LCu, two-stage solution treatment exhibits superior mechanical properties in Yield Strength, Ultimate Tensile Strength, and Elongation compared to single-stage solution treatment, with YS and UTS increasing from 549 MPa and 599 MPa to 551 MPa and 606 MPa, respectively, and EL from 13.5% to 14.3%. The increase in mechanical properties with solution time is less pronounced than in earlier stages, showing a plateauing trend.

For HCu, the benefit of two-stage solution treatment is not as apparent as short-duration solution at 490 °C. In terms of strength, YS and UTS first increase then show a declining trend, from 540 MPa and 596 MPa to 556 MPa and 606 MPa, respectively. However, EL remains almost flat but slightly decreases, from 13.3% to 13.2%. Comparing the two materials, LCu is clearly superior to HCu in YS, UTS, and EL. Synthesizing previous data, it can be concluded that, overall, LCu benefits more from two-stage solution treatment than HCu. While short-duration solution treatment does offer some minor improvements in elongation rate for HCu, this effect flattens out with longer solution times.

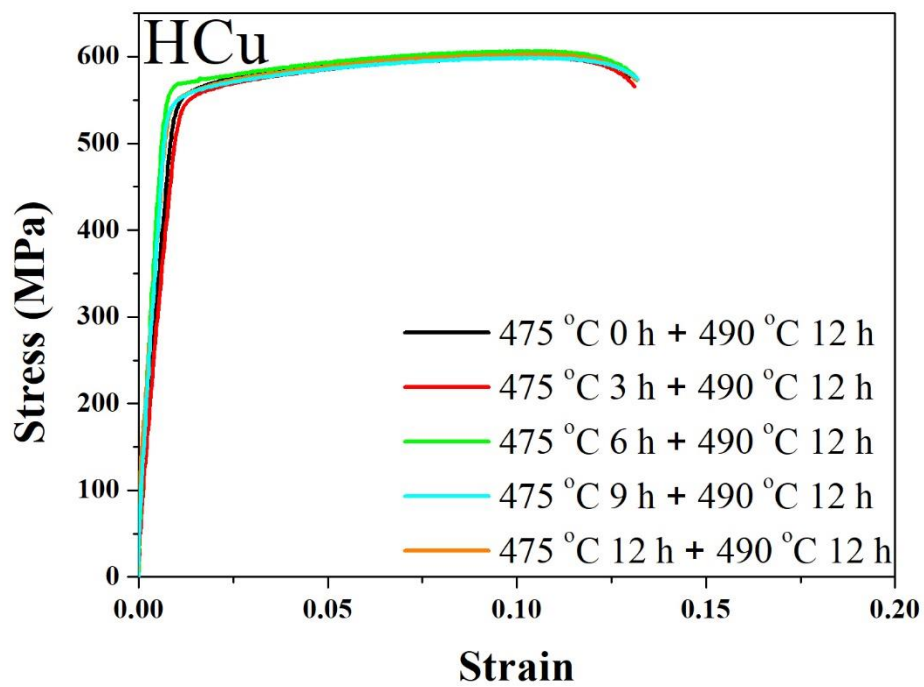
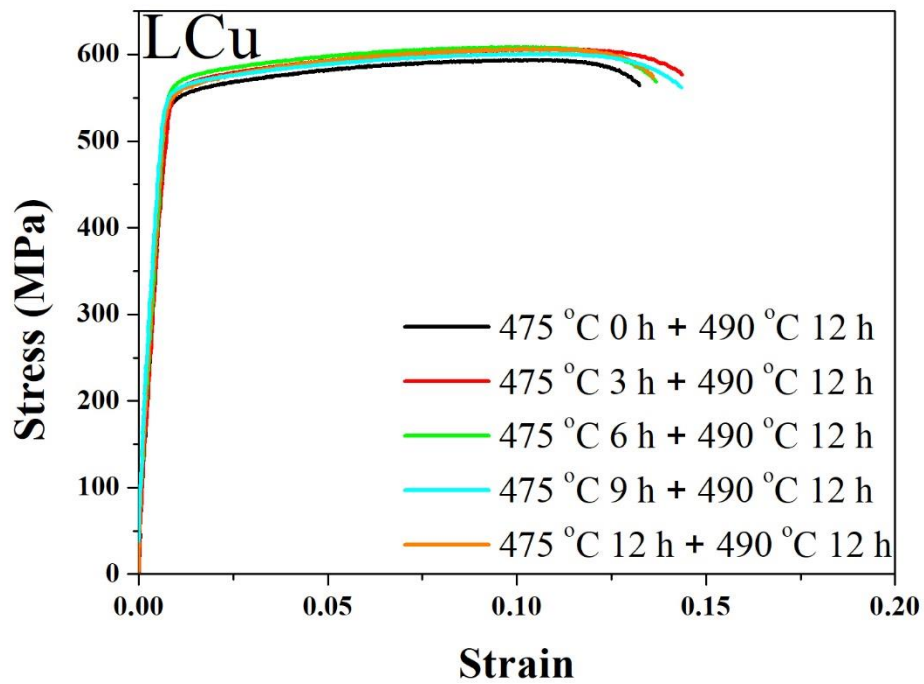


Figure 4-15 Tensile curves of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.

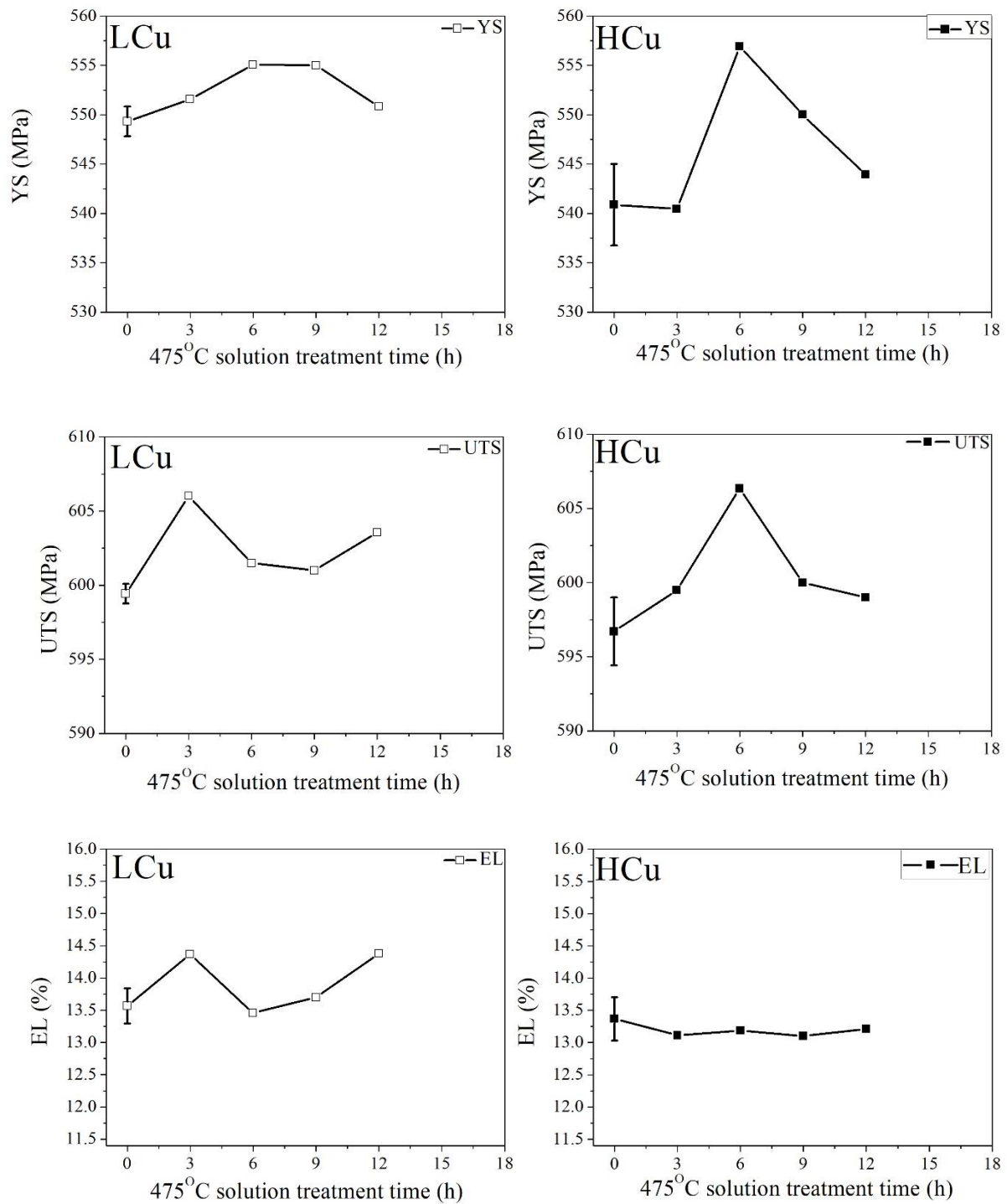


Figure 4-16 Mechanical properties of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.

Table 4-17 YS of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475 °C 0 h + 490 °C 12 h	549	540
475 °C 3 h + 490 °C 12 h	551	540
475 °C 6 h + 490 °C 12 h	555	556
475 °C 9 h + 490 °C 12 h	552	550
475 °C 12 h + 490 °C 12 h	550	543

Table 4-18 UTS of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475 °C 0 h + 490 °C 12 h	599	596
475 °C 3 h + 490 °C 12 h	606	599
475 °C 6 h + 490 °C 12 h	601	606
475 °C 9 h + 490 °C 12 h	600	601
475 °C 12 h + 490 °C 12 h	603	599

Table 4-19 EL of two-stage solution treatment with second stage 490 °C 12 h for LCu and HCu on the condition peak ageing at 120 °C.

St. + NA + AA	7075-LCu (%)	7075-HCu (%)
475 °C 0 h + 490 °C 12 h	13.5	13.3
475 °C 3 h + 490 °C 12 h	14.3	13.1
475 °C 6 h + 490 °C 12 h	13.4	13.1
475 °C 9 h + 490 °C 12 h	13.7	13.1
475 °C 12 h + 490 °C 12 h	14.3	13.2

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24h.

4.2.8 Two-Stage Solution Treatment Organizing the Variation in 475 °C Solution Time with a Fixed 490 °C from 3 to 12 Hours

Summarizing the results, to examine the impact of different solution treatment temperatures on mechanical properties, the procedure first involves fixing the solution treatment at 490°C for 3, 6, 9, and 12 hours, while varying the duration of solution treatment at 475 °C. In Figure 4-17 shown all mechanical properties of LCu and HCu, using the duration at 475 °C as the horizontal axis, the changes in strength and elongation are observed to identify the solution treatment conditions that yield the best mechanical properties. Observing the change in material strength with 475 °C as the variable, the charts indicate that for LCu, the mechanical properties of two-stage solution treatment are superior to those of single-stage solution treatment. Both Yield Strength and Ultimate Tensile Strength increase with the duration of solution treatment at 475°C and then plateau, with YS reaching approximately 555 MPa and UTS around 605 MPa. Elongation increases with solution time at 475°C, rising to over 14%. However, the benefits of two-stage solution treatment during short-duration solution treatment at 490°C exceed those of long-duration solution at 490 °C, with a more significant change in mechanical properties in short-duration solution treatments. Thus, short-duration two-stage solution treatment is more effective.

In HCu, the strength (YS and UTS) first increases and then shows a declining trend, with YS peaking at approximately 555 MPa and UTS at about 605 MPa, before decreasing to 545 MPa and 597 MPa, respectively. For elongation, short-duration solution treatment at 490°C shows an increase with solution time at 475°C, followed by a decline. Under long-duration solution treatment at 490°C, EL does not show an increasing trend but rather remains flat before decreasing, settling around 13.5%. This indicates that the

improvement in mechanical properties due to two-stage solution treatment at 475°C is more significant for LCu than for HCu, suggesting that the benefits of two-stage solution treatment may decrease with an increase in the Cu content of the aluminum alloy.

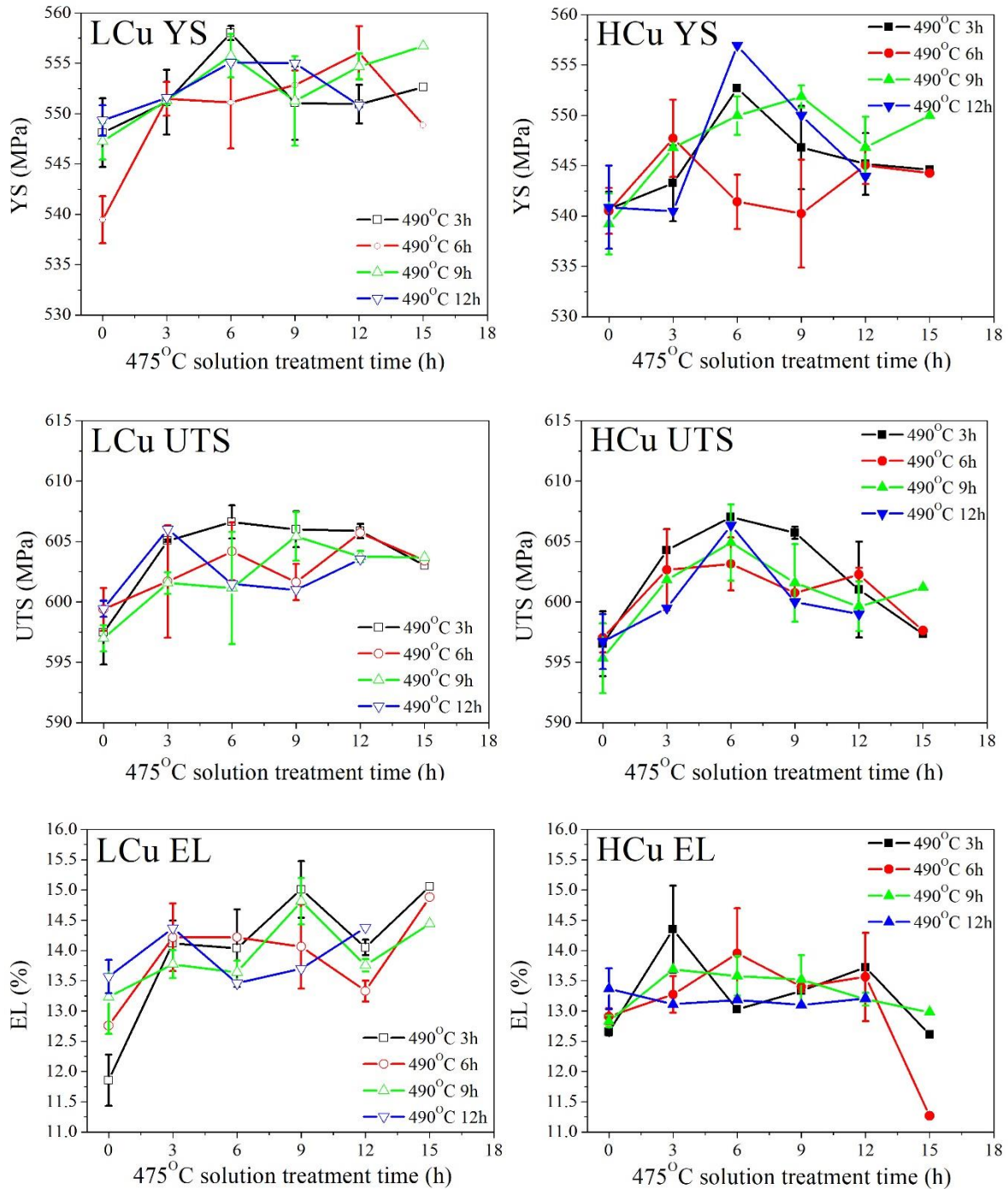


Figure 4-17 Mechanical properties of two-stage solution treatment with 475 °C as the variable for LCu and HCu on the condition peak ageing at 120 °C.

4.2.9 Two-Stage Solution Treatment Organizing the Variation in 490 °C Solution Time with a Fixed 475 °C from 3 to 15 Hours

Altering the fixed variables to understand the impact of the duration of solution treatment at 490 °C on mechanical properties, this approach involves setting the solution treatment at 475°C for 3, 6, 9, 12, and 15 hours. In Figure 4-18 shown all mechanical properties of LCu and HCu, by using the duration at 490 °C as the horizontal axis, the changes in strength and elongation as a function of the 490 °C treatment time are observed. This method aims to identify the optimal solution treatment conditions that enhance mechanical properties. By reorganizing the mechanical property trends with 490 °C as the variable and fixing the 475 °C solution time, different trends emerge when observing the impact of 490 °C solution times on LCu and HCu. The charts show that as the 490 °C solution time increases, YS of LCu in some cases first rises then falls, while in others, it remains steady with an increase in 490 °C solution time, suggesting that extending the two-stage 490 °C solution time has a limited impact on YS for LCu. In contrast, HCu shows varying directions: in some conditions, YS increases with longer 490 °C solution times, but in others, it plateaus or decreases, indicating no uniform trend for HCu with extended two-stage 490 °C solution times, which should be determined based on specific conditions.

For LCu, increasing the 490°C solution time generally leads to a plateau or decline in UTS, suggesting that 490 °C solution times should not be excessively long to enhance strength. For HCu, some conditions show an increase in UTS with longer 490 °C solution times, so, like YS, the optimal 490 °C solution time needs adjustment based on different conditions to achieve the best strength. Regarding elongation, most conditions for LCu show an increase or remain within the standard deviation as the 490 °C solution time

increases. However, under the condition of 15 hours of solution at 475 °C, increasing the 490 °C solution time results in a decreased elongation rate over time. For HCu, elongation mostly stays within the standard deviation, indicating no substantial benefit to elongation with increased 490 °C solution time in aluminum alloys with higher Cu content.

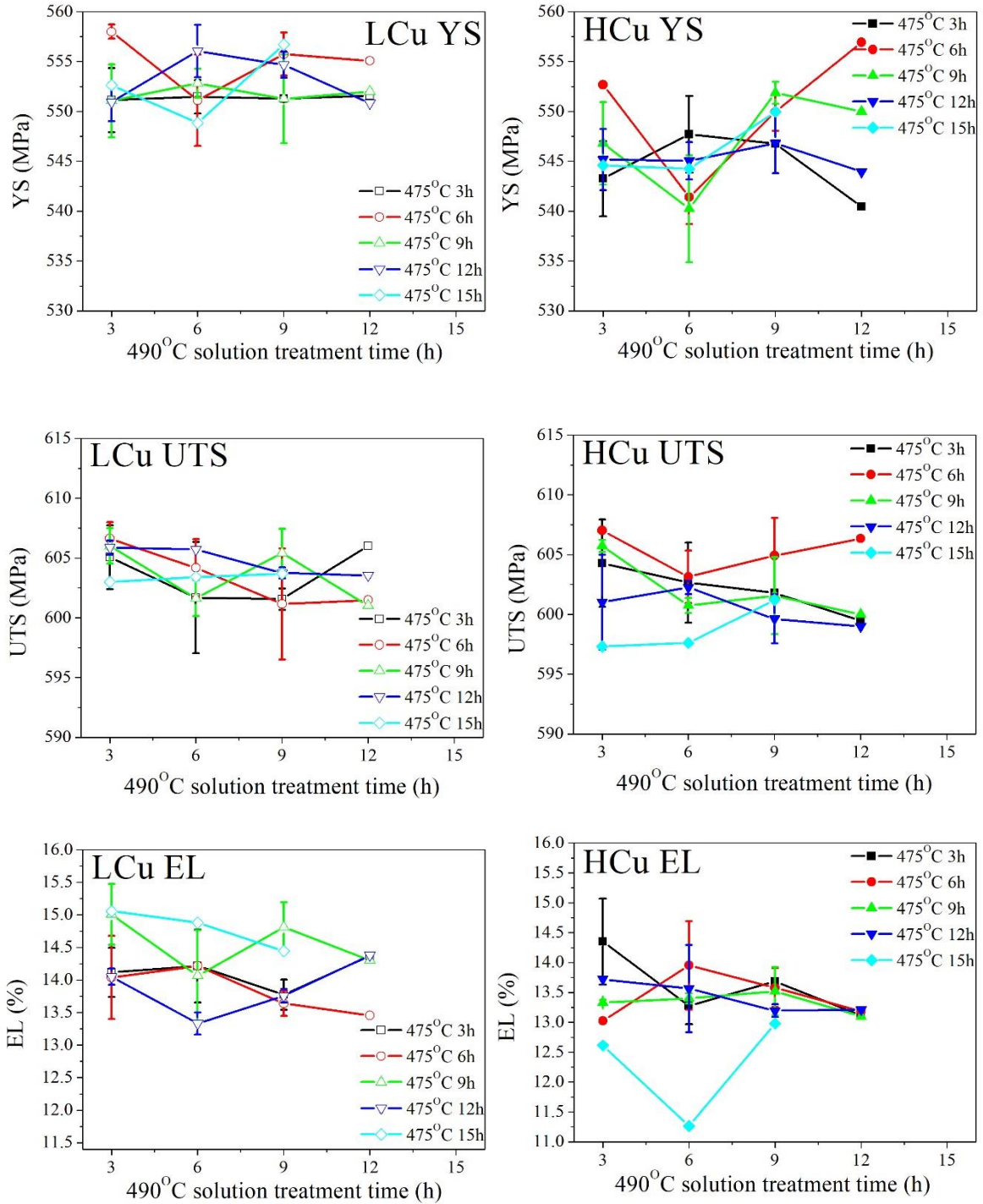


Figure 4-18 Mechanical properties of two-stage solution treatment with 490 °C as the variable for LCu and HCu on the condition peak ageing at 120 °C.

4.2.10 Single-stage Solution Treatment for 475 °C

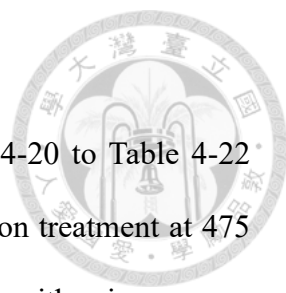


Figure 4-19 shows the tensile curve of LCu and HCu. Table 4-20 to Table 4-22 represents the mechanical properties. If only the first stage of solution treatment at 475 °C is considered, the charts show that overall mechanical properties either increase or plateau with increasing solution time, including both strength and ductility. For LCu, after 3 hours of solution, the YS is 539 MPa and the UTS is approximately 595 MPa. After 15 hours of solution, YS drops slightly to 535 MPa and UTS to approximately 594 MPa, a minor decrease. However, the elongation rate increases from 11.8% at 3 hours of solution to 12.9% at 15 hours. In HCu, after 3 hours of solution, YS is 528 MPa and UTS is approximately 579 MPa, showing an upward trend to 534 MPa for YS and approximately 581 MPa for UTS after 15 hours, while the elongation rate slightly increases from 10.3% at 3 hours of solution to 10.5% at 15 hours, remaining relatively steady.

Upon closer examination, the mechanical properties exhibited by solution treatment at 475 °C overall do not match those achieved by solution treatment at 490 °C. Specifically, for LCu, extending the solution time at 475 °C yields slightly lower mechanical properties than after 3 hours of solution at 490 °C, with YS, UTS, and EL at 548 MPa, 597 MPa, and 11.8%, respectively. For HCu, the mechanical properties obtained from solution treatment at 475°C are inferior to those obtained by LCu under the same conditions, with 3 hours of solution at 490 °C resulting in YS, UTS, and EL of 540 MPa, 597 MPa, and 12.6%, respectively. Therefore, it is suggested that a solution temperature of 490 °C is preferable to 475 °C. When considering material differences, aluminum alloys with higher Cu content are less suited for single-stage solution treatment at 475 °C, as their mechanical properties significantly lag behind those achieved at 490 °C.

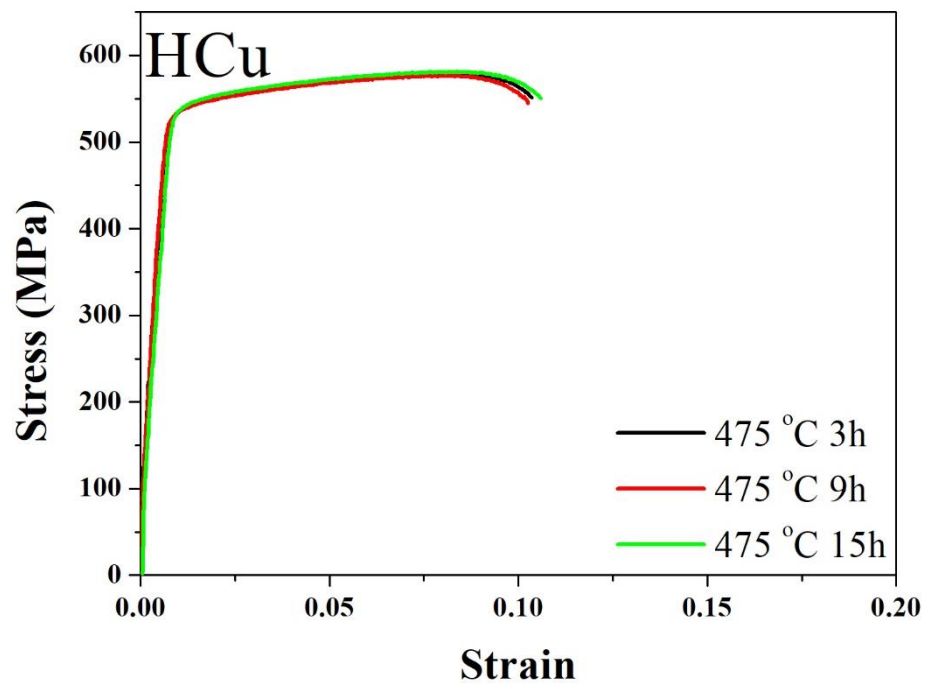
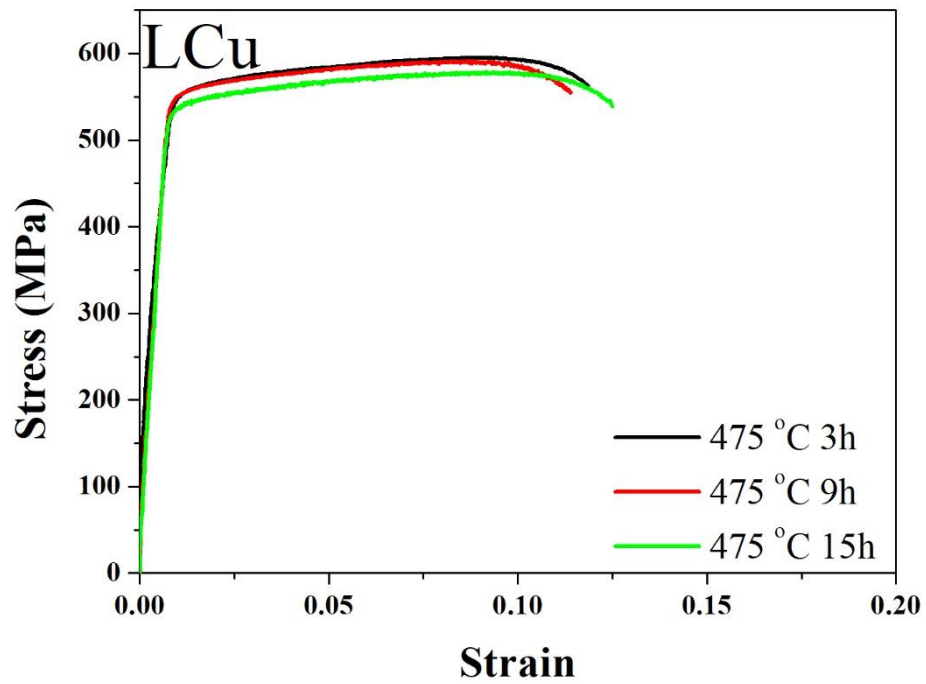


Figure 4-19 Tensile curves of single-stage solution treatment with 475 °C for LCu and HCu on the condition peak ageing at 120 °C.

Table 4-20 YS of single-stage solution treatment with 475 °C for LCu and HCu on the condition peak ageing at 120 °C.

St. +NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475 °C 3h	539	528
475 °C 9h	548	528
475 °C 15h	535	534

Table 4-21 UTS of single-stage solution treatment with 475 °C for LCu and HCu on the condition peak ageing at 120 °C.

St. +NA + AA	7075-LCu (MPa)	7075-HCu (MPa)
475 °C 3h	595	579
475 °C 9h	594	576
475 °C 15h	594	581

Table 4-22 EL of single-stage solution treatment with 475 °C for LCu and HCu on the condition peak ageing at 120 °C.

St. +NA + AA	7075-LCu (%)	7075-HCu (%)
475 °C 3h	11.8	10.3
475 °C 9h	10.8	10.1
475 °C 15h	12.9	10.5

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24h.

4.2.11 Optimized Solution Treatment Process and Comparison

Since the dissolution of the second phases back into the base matrix increases the elements available for precipitates in the aluminum alloy, which is related to subsequent aging processes, and considering the complexity added by incipient melting factors, the mechanism by which the material properties are altered is intricate. Therefore, the optimal conditions are determined directly by observing the changes in mechanical properties across different temperatures. Figure 4-20 shows the tensile curve. Table 4-23 to Table 4-24 represents the mechanical properties.

From the summarized data, the optimized two-stage solution treatment process is identified as 9 hours at 475 °C followed by 3 hours at 490 °C for LCu, and 3 hours at 475 °C followed by 3 hours at 490 °C for HCu. The comparisons show that LCu experiences a significant improvement in overall mechanical properties with the addition of 9 hours at 475 °C, with strength increasing by about 10 MPa and elongation by 3.2%. This improvement is not simply due to extending the solution time, as mechanical properties from a single-stage 12-hour solution treatment do not match those of the two-stage solution treatment. Compared to 3 hours at 490 °C solution treatment, strength only increases by about 2 MPa and elongation by 1.7%. Moreover, the improvement is not solely due to adding 9 hours at 475 °C, as mechanical properties from 9 hours of solution at 475°C alone are not favorable, indicating that two-stage solution treatment is beneficial for enhancing mechanical properties.

For HCu, the overall mechanical properties also improve after adding 3 hours at 475 °C, with strength increasing by about 6 MPa and elongation by 1.7%. Compared to 3 hours at 490 °C solution treatment, the 6-hour solution treatment at 490 °C results in a strength increase of about 1 MPa and elongation by 0.7%, further demonstrating the benefits of two-stage solution treatment. However, when comparing between materials,

LCu clearly benefits more from two-stage solution treatment than HCu, suggesting that adding Cu elements to aluminum alloy is not advantageous for two-stage solution treatment at 475 °C.

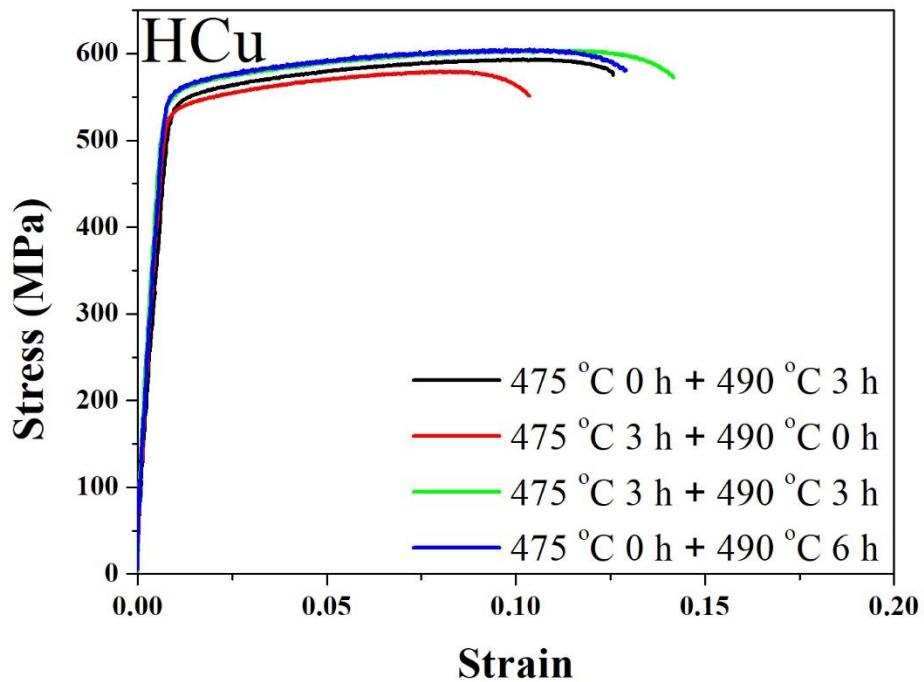
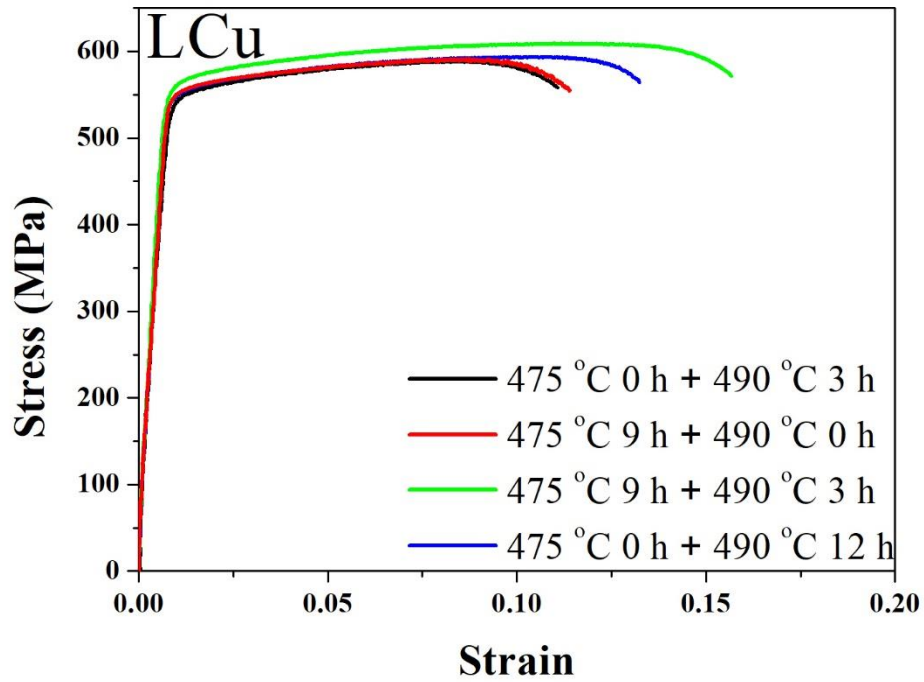


Figure 4-20 Tensile curves of two-stage and single-stage solution treatment for LCu and HCu on the condition peak ageing at 120 °C.

Table 4-23 Mechanical properties of two-stage and single-stage solution treatment for LCu on the condition peak ageing at 120 °C.

LCu	YS(MPa)	UTS(MPa)	EL(%)
St. + NA + AA			
490°C 3h	548 ± 3.4	597.4 ± 2.6	11.8 ± 0.42
475°C 9h	548	594.0 ± 2.5	10.8 ± 0.37
475°C 9h + 490°C 3h	551 ± 3.6	606.0 ± 1.4	15.0 ± 0.46
490°C 12h	549 ± 1.5	599.4 ± 0.66	13.5 ± 0.27

Table 4-24 Mechanical properties of two-stage and single-stage solution treatment for HCu on the condition peak ageing at 120 °C.

HCu	YS(MPa)	UTS(MPa)	EL(%)
St. + NA + AA			
490°C 3h	540.6 ± 1.7	596.5 ± 2.7	12.6 ± 0.056
475°C 3h	528	579	10.3
475°C 3h + 490°C 3h	543.2 ± 3.7	604.2 ± 3.6	14.3 ± 0.71
490°C 6h	540.5 ± 2.2	597.0 ± 1.1	12.9 ± 0.13

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24h.

4.3 DSC, XRD and Hardness Test Analysis

4.3.1 DSC Result

Differential Scanning Calorimetry (DSC) thermograms indicate the formation and dissolution of phases. Figure 4-21 and Figure 4-22 show the DSC curve of LCu and HCu. Table 4-25 identifies the peaks in as-received LCu. The charts reveal two distinct endothermic peaks at 481 °C and 493 °C in the raw material, presumed to represent the dissolution of η and S phases, respectively. The signal intensity differences between LCu and HCu for these phases—one strong and one weak—align with these assumptions.

For LCu, after a 9-hour solution treatment at 475 °C, the exothermic peak near 481 °C disappears, suggesting the η phase is completely dissolved by this treatment. However, the peak at 493 °C remains, indicating that the S phase does not fully dissolve at 475 °C, hence its presence in the matrix contributes to the inferior mechanical properties observed. In contrast, after 3 hours at 490 °C and the combination of 9 hours at 475 °C followed by 3 hours at 490 °C solution treatments, no significant exothermic peaks are evident, implying the complete dissolution of the S phase under these conditions. Therefore, the 490°C solution treatment is effective for the S phase dissolution, and the absence of η and S phase signals in both treatments suggests that differences in mechanical properties may be attributed to incipient melting. This microstructural difference will be explored further in subsequent electron microscopy sections.

Similarly, HCu exhibits comparable results in DSC analysis. In the raw material, the signal for the η phase is weaker in HCu than in LCu, whereas the signal for the S phase is stronger, likely due to different Cu content. In a single-stage solution treatment of 3 hours at 475 °C, a strong signal persists at 491 °C, even more pronounced than in LCu's single-stage solution at 475 °C, indicating a significant amount of the S phase remains



undissolved. After the 490 °C solution treatment, no significant signals are observed, suggesting that most second phases are completely dissolved in both cases.

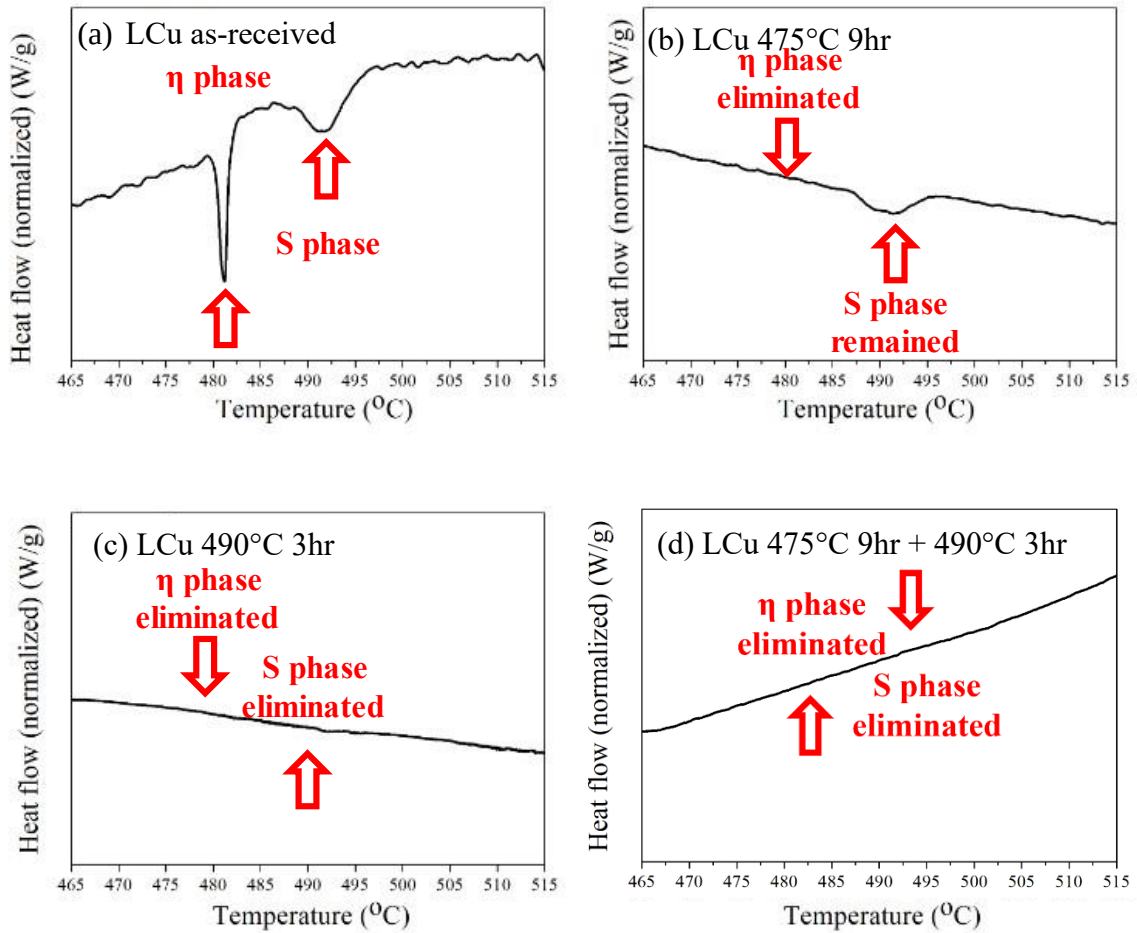
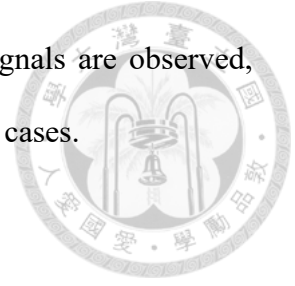


Figure 4-21 DSC curves of (a) as-received (b) first stage solution treatment (c) second stage solution treatment (d) two-stage solution treatment on LCu.

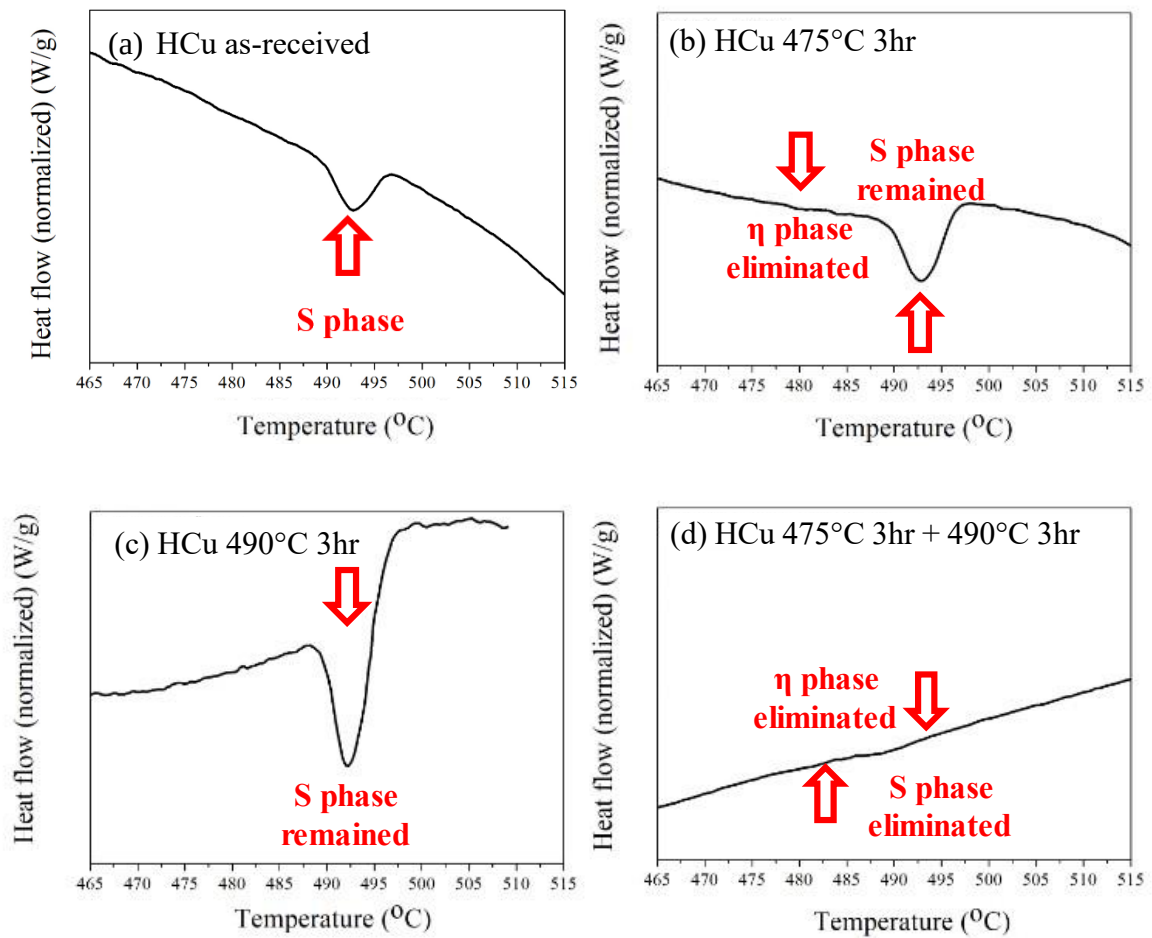


Figure 4-22 DSC curves of (a) as-received (b) first stage solution treatment (c) second stage solution treatment (d) two-stage solution treatment on HCu.

Table 4-25 Incipient melting peaks on Figure 4-21(a)

Peak value	Eutectic phase	Melting point
Peak 1	$\alpha + \text{MgZn}_2$	481° C
Peak 2	$\alpha + \text{Al}_2\text{CuMg}$	493° C

4.3.2 XRD Result

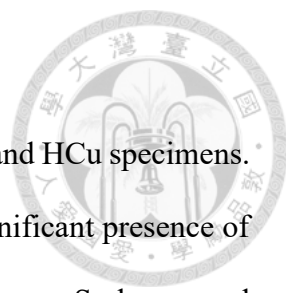


Figure 4-23 and Figure 4-24 represents the XRD curves in LCu and HCu specimens. In the XRD results, both LCu and HCu raw materials exhibited a significant presence of second phase signals, aligning with DSC outcomes, identifying η phases, S phases, and the aluminum alloy matrix. This confirms that both types of second phases are extensively distributed within LCu and HCu aluminum alloy raw materials. Calculations based on literature data for the second phases' lattice constants and X-ray wavelengths show η phase signals at scattering angles of 19.8, 21.2, 22.5, 29.2, 34.7, 38.0, 41.0, 41.8, 52.4, and 55.4 degrees, with clear signals observed in both copper variants. Additionally, S phase signals appear at scattering angles of 12.3, 19.1, 22.8, 24.1, 24.9, 27.2, 34.9, 45.1, 47.0, 52.2, 56.1, and 71.2 degrees. After solution heat treatment, regardless of being a single-stage or two-stage process, the signals for η and S phases significantly decrease, blending indistinguishably into the background. The S phase signal is detectable in the DSC for aluminum alloys treated at 475 °C in a single-stage process, but the XRD outcomes reveal no significant difference from those treated at 490 °C, showing no S phase signals. This suggests the total volume percentage of second phases post-solution treatment is too low for XRD resolution, rendering it unable to differentiate between different solution treatment processes, only leaving the aluminum alloy base signal. A comparison between the raw materials and post-solution treatment processes shows a significant reduction of η and S phases.

In HCu, second phase signals in the raw material almost mirror those in LCu, with peak signal angles closely aligning, indicating η and S phases. Following a 475°C solution treatment for 3 hours, peaks were observed at 19.1, 22.8, 24.1, 24.9, 27.2, 34.9, and 52.2 degrees, corresponding to S phase signals, but η phase areas exhibited noise without distinct peaks. Similar patterns are seen after the 490 °C solution treatment for 3 hours,

albeit with lower peak heights, yet S phase signals persisted. This contrast with LCu, where post-solution treatment eradicated S phase signals, suggests that copper content differences might cause disparate outcomes. The hypothesis is that after the second stage of solution treatment, S phase still remains within the base, aligning with DSC findings. After undergoing a solution treatment of 475 °C for 3 hours followed by 490 °C for 3 hours, no prominent second phase signals were detected, only the highest peak signals corresponding to the aluminum base, similar to LCu. This suggests that the S phase might have dissolved back into the matrix.

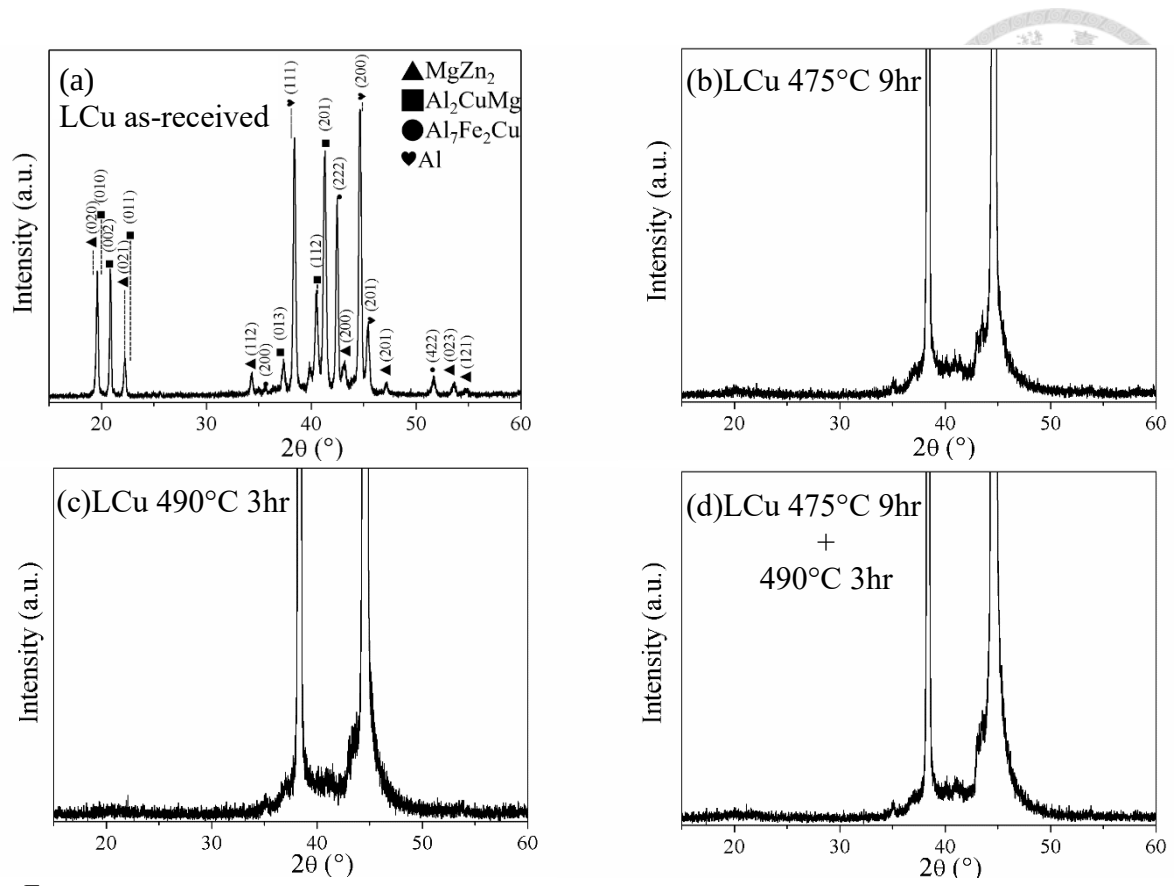


Figure 4-23 XRD curves of (a) as-received (b) first stage solution treatment (c) second stage solution treatment (d) two-stage solution treatment on LCu.

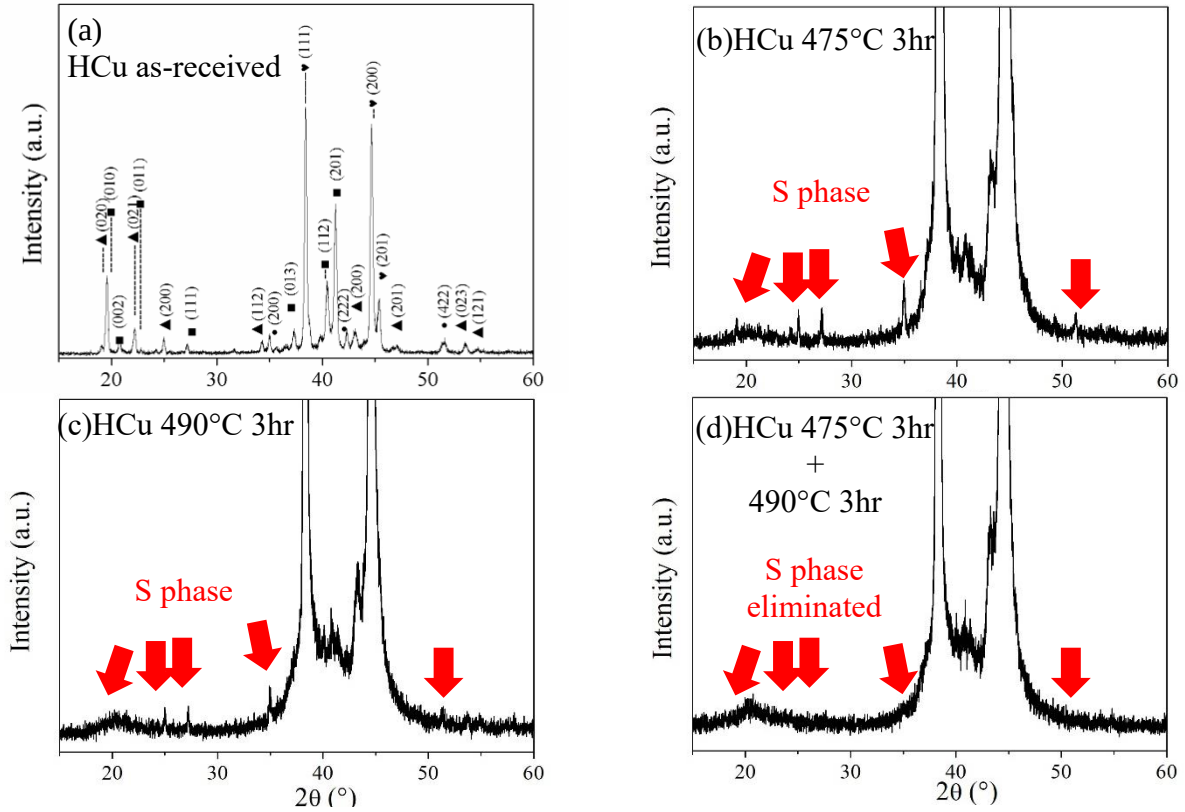
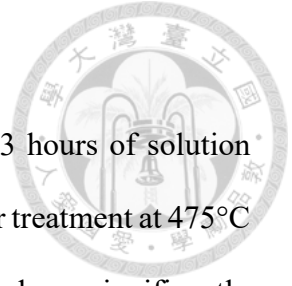


Figure 4-24 XRD curves of (a) as-received (b) first stage solution treatment (c) second stage solution treatment (d) two-stage solution treatment on HCu.



4.3.3 Hardness Test

The hardness tests reveal that for LCu, the process involving 3 hours of solution treatment at 490°C results in a hardness of 193 HV. By adding a 9-hour treatment at 475°C ahead of the 490°C solution treatment in a two-stage process, the hardness significantly increases to 199 HV, showing a similar trend to the tensile results. Even increasing the total solution time at 490°C to 12 hours only raises the hardness to 194 HV, which is still less than the two-stage solution treatment, and the difference falls outside the standard deviation. Thus, it is confirmed that two-stage solution heat treatment can enhance the hardness of aluminum alloys. In HCu, the hardness after 3 hours of solution treatment at 490°C is 183 HV. After the two-stage solution treatment, the hardness increases to 193 HV, which is also higher than the 185 HV observed with total 6 hours of solution at 490°C. This indicates that the benefits of two-stage solution treatment are evident in both LCu and HCu. When comparing between the materials under various conditions, LCu consistently shows higher hardness than HCu, suggesting that aluminum with lower Cu content have higher strength, aligning with the tensile results and previous literature.

Table 4-26 Hardness comparison of different solution treatment for LCu on peak ageing.

LCu	Hardness(HV)
490°C 3h	187.4 ± 3.1
475°C 9h	193.5 ± 2.8
475°C 9h + 490°C 3h	199.8 ± 2.5
490°C 12h	194.3 ± 2.1

Table 4-27 Hardness comparison of different solution treatment for HCu on peak ageing.

HCu	Hardness(HV)
490°C 3h	177.2 ± 3.6
475°C 3h	183.6 ± 3.1
475°C 3h + 490°C 3h	193.6 ± 3.2
490°C 6h	185.9 ± 1.4

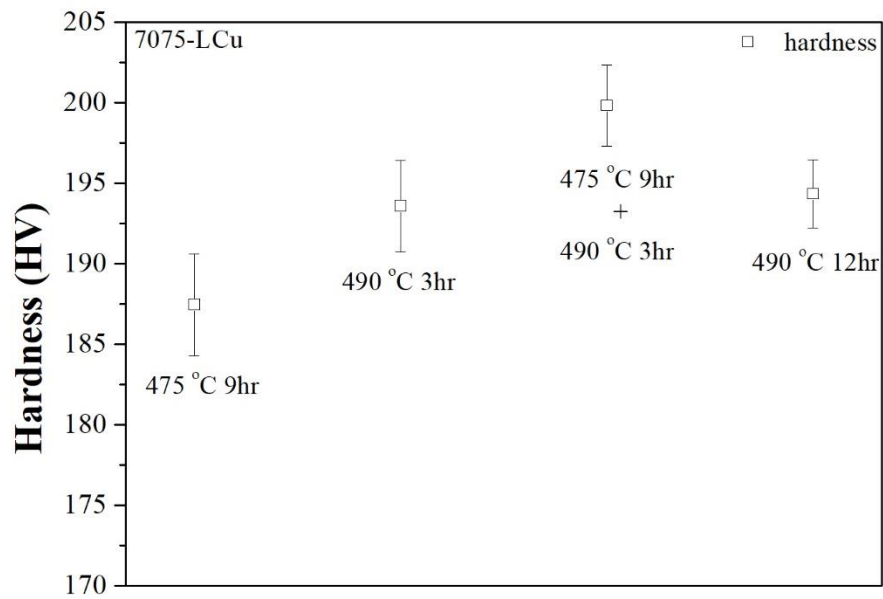


Figure 4-25 Hardness distribution of as-received/ first stage solution treatment/ second stage solution treatment/ two-stage solution treatment on LCu with peak aging.

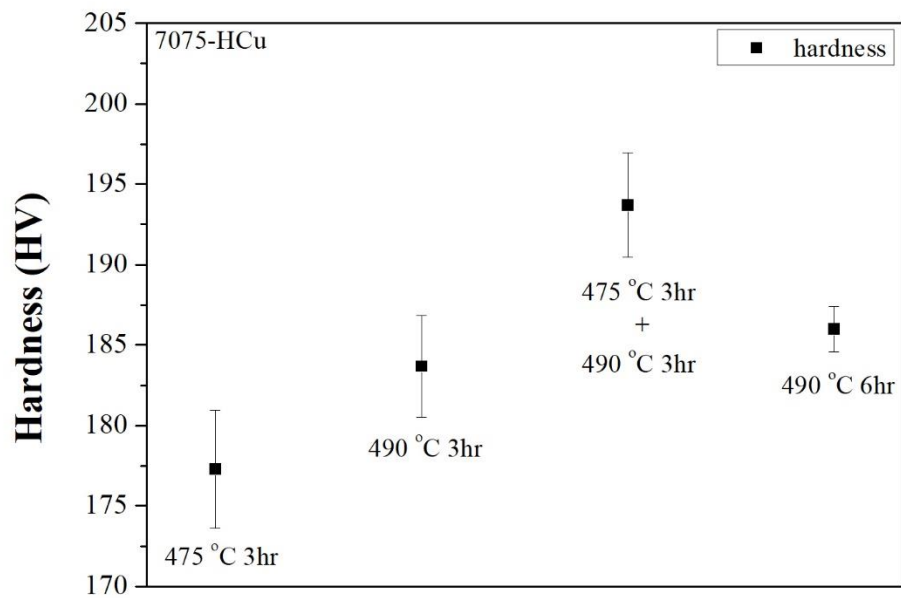
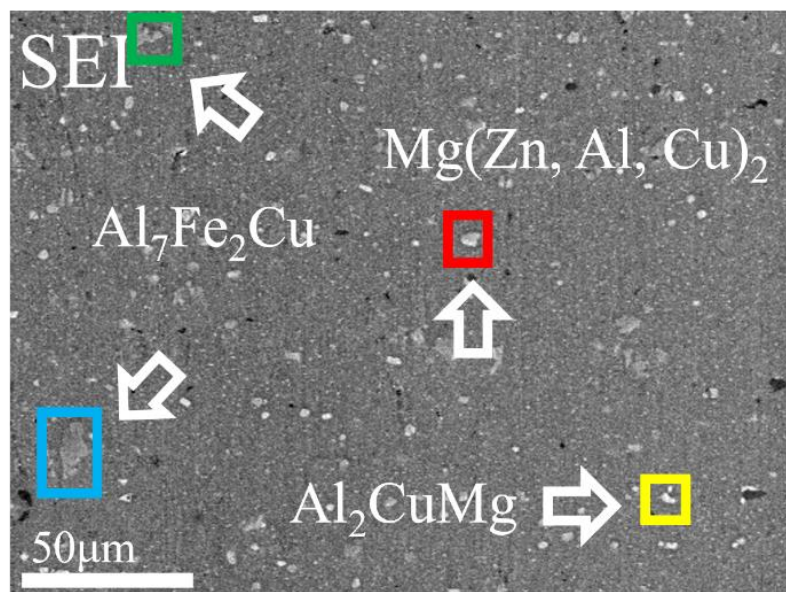


Figure 4-26 Hardness distribution of as-received/ first stage solution treatment/ second stage solution treatment/ two-stage solution treatment on HCu with peak aging.

4.4 SEI, BEI, EDS, and EPMA Analysis

4.4.1 As-received LCu

Scanning Electron Microscope (SEM) observations in Figure 4-27 reveal that the surface of the raw material, before solution heat treatment, is uneven. Secondary Electron Imaging (SEI) shows many particles, and Backscattered Electron Imaging (BEI) displays bright particles, suggesting these are agglomerations of alloying elements, indicative of second phases yet to be dissolved. For LCu, the presence of numerous undissolved impurities within the raw material is noted through electron microscopy. To further identify the types of second phases, Energy Dispersive Spectroscopy (EDS) spot analysis is conducted to assess the elemental ratios, revealing approximately three different types of second phases based on the alloying element ratios. Phases containing zinc, magnesium, aluminum, and copper are presumed to be η phase, $\text{Mg}(\text{Zn}, \text{Al}, \text{Cu})_2$. Phases with only aluminum, magnesium, and copper, based on their elemental ratios, are presumed to be S phase, Al_2CuMg . Finally, phases containing iron are speculated to be ω phase. However, as EDS is semi-quantitative, Electron Probe Micro Analyzer (EPMA) scanning and spot analysis are needed for more accurate determination of elemental ratios and distributions.



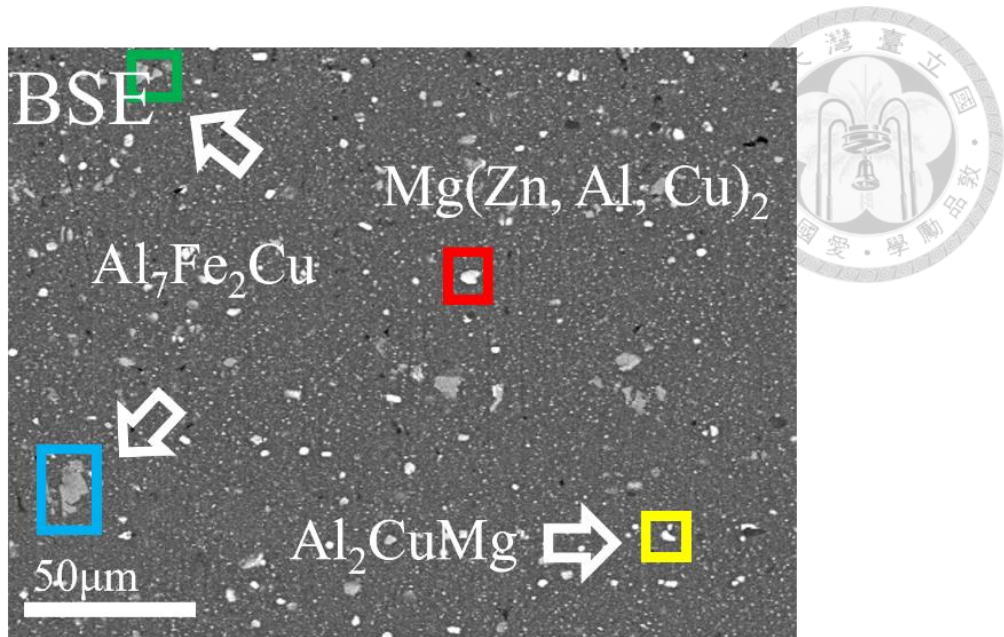


Figure 4- 27 SEI and BEI of LCu as-received with EDS(red : η ; yellow : S; green, blue : ω).

In Figure 4-28 second phase components are marked with colors: brown areas represent η phase, orange areas indicate S phase, yellow areas are for ω phase, red areas denote incipient melting sites, and green areas are holes left by Mg_2Si during polishing. EPMA scans show that particle agglomerations primarily consist of magnesium, copper, and zinc, with iron present in a few areas. Aluminum levels significantly decrease at the particle sites. Manganese, chromium, and silicon elements are almost not agglomerated, being mostly dispersed within the alloy matrix. Analysis of the coinciding elements in the second phases suggests the presence of η , S, and ω phases. In Figure 4-29, WDS quantification, color indicate locations. In the table, the same color borders present the quantification of elements at marked locations. Precise spot analysis confirms the η phase, excluding the aluminum signal from the alloy matrix, closely matching the η phase's elemental ratio, i.e., $Mg(Zn, Al, Cu)_2$. Another identified phase is the S phase, where copper and magnesium each roughly account for a quarter, with almost no other elements present, aligning with the S phase's elemental ratio, i.e., Al_2CuMg . It is concluded that the main dissolvable second phases are η and S phases, both dispersed within LCu raw material, along with the ω phase, which literature suggests cannot be dissolved.

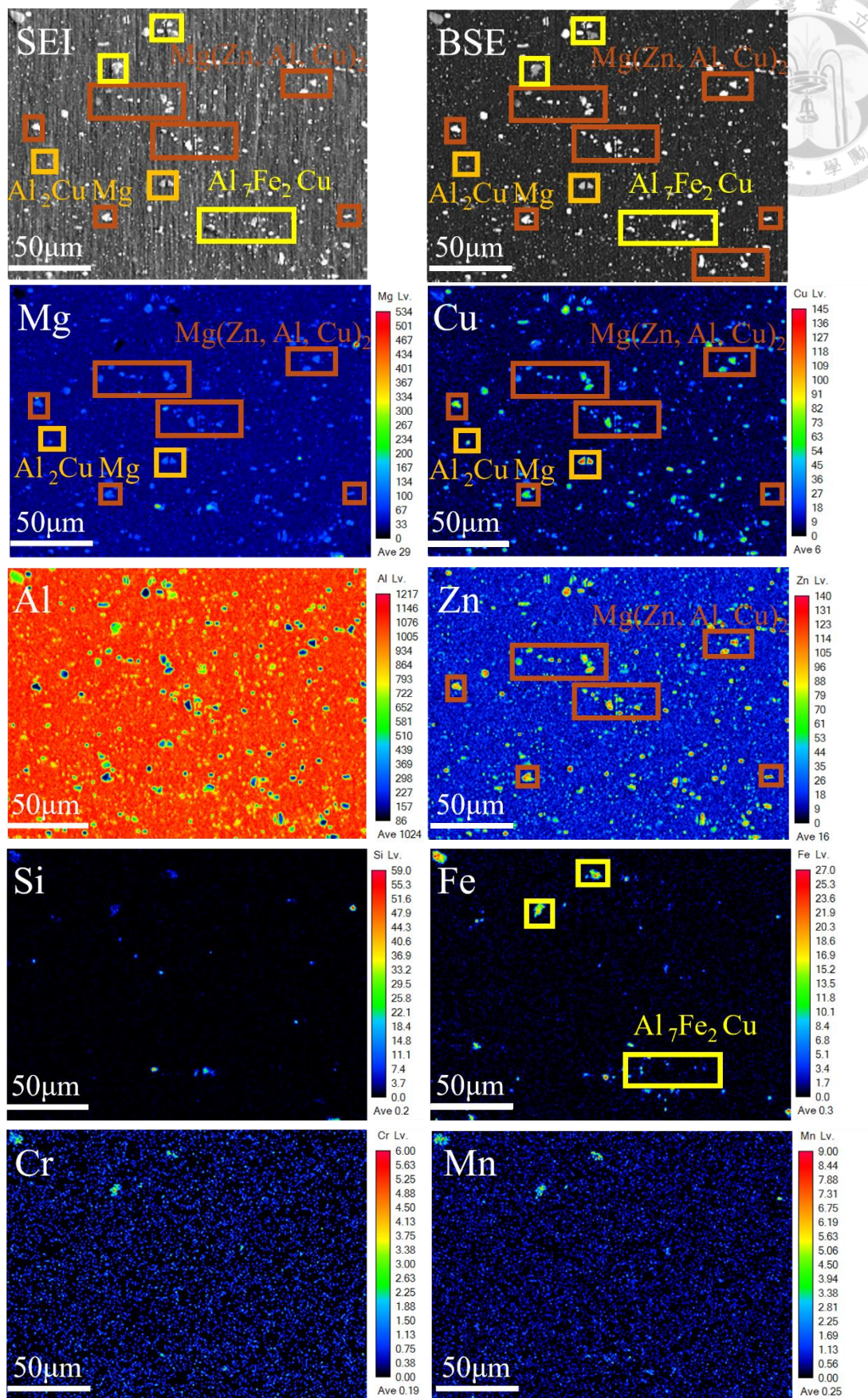
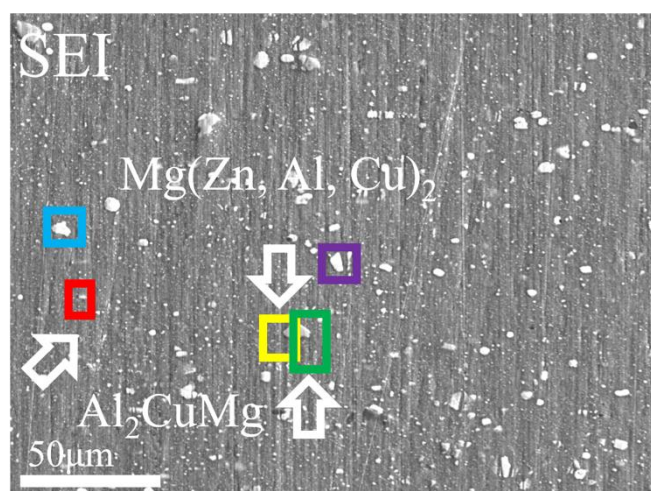
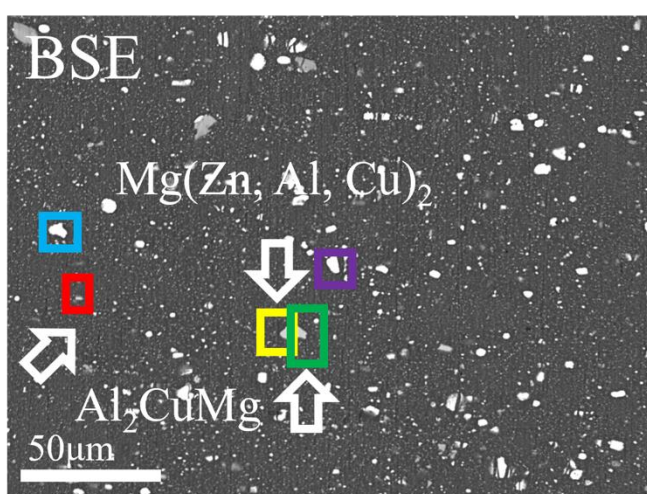


Figure 4-28 EPMA mapping of LCu as-received (brown : η; orange : S; yellow : ω).



S phase

WDS	at (%)
Al	62.4
Mg	17.8
Cu	18
Zn	1.5
Fe	0
Si	0
Mn	0



S phase

WDS	at (%)
Al	56.8
Mg	20.8
Cu	20.6
Zn	1.4
Fe	0
Si	0
Mn	0

S phase

WDS	at (%)
Al	60.2
Mg	18.1
Cu	20
Zn	1.4
Fe	0
Si	0
Mn	0

η phase

WDS	at (%)
Al	42.3
Mg	21.9
Cu	13.1
Zn	22.4
Fe	0
Si	0
Mn	0

η phase

WDS	at (%)
Al	32.6
Mg	28.3
Cu	13.6
Zn	25.1
Fe	0
Si	0
Mn	0

Figure 4-29 WDS elemental quantification of LCU as-received.

4.4.2 LCu after 475 °C 9 h Solution Treatment

After the first stage of solution treatment at 475°C, Secondary Electron Imaging in Figure 4-30 shows a significant reduction in surface particles compared to the raw material, with only a few particles remaining. Backscattered Electron Imaging in Figure 4-30 also reveals that the remaining particles are not as bright as most particles in the raw material, suggesting that the η phase, which contains Zn, might have been dissolved back into the aluminum alloy matrix, leaving behind the S phase and iron-containing second phases. To determine the composition and proportion of the surface secondary phases, EDS signals were measured, revealing high aluminum and copper ratios with some magnesium elements, while iron and Zn signals were very low, suggesting the residue is likely the S phase. Additionally, some particles showed iron element signals, suggesting these are iron-containing second phases, likely the ω phase.

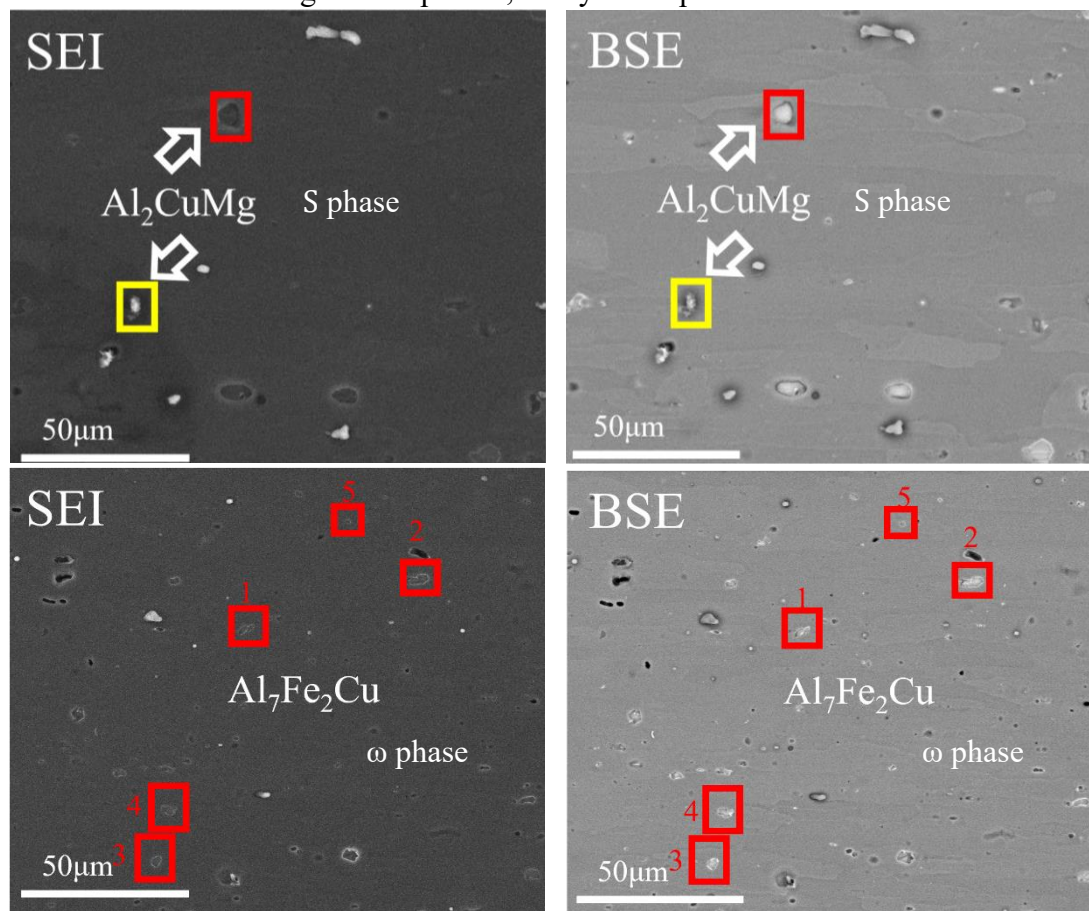


Figure 4-30 SEI and BEI of LCu after 475°C 9h solution treatment with peak aging with EDS.

Further confirmation of the surface second phases' composition and proportions was sought by scanning the LCu treated for 9 hours at 475°C with Electron Probe Micro Analyzer in Figure 4-31. The results confirmed copper element agglomerations at particle sites, with a lower proportion of Zn compared to the matrix, suggesting the remaining second phases are not η phase, $\text{Mg}(\text{Zn}, \text{Al}, \text{Cu})_2$, due to its partial Zn content, but are likely the S phase and iron-containing second phases like the ω phase, with most particles containing iron elements. Thus, it is inferred that after 9 hours of solution treatment at 475°C, the majority of the remaining phases are iron-containing second phases, with a minor presence of the S phase, and nearly no η phase.

Furthermore, holes observed in SEI and BEI, where significant Mg and Si signals were detected, might be due to Mg_2Si formed during polishing. Although some Mg_2Si has been dissolved, the remaining element signals at these sites were detectable by EPMA, suggesting these holes were not caused by high-temperature incipient melting. Spot analysis in Figure 4-32 of non-iron-containing second phases showed approximately half aluminum, with Cu and Mg each accounting for about a quarter, confirming the presence of residual S phase, Al_2CuMg .

In summary, the first stage of solution treatment at 475 °C appears to dissolve the η phase but leaves behind the S phase in the matrix.

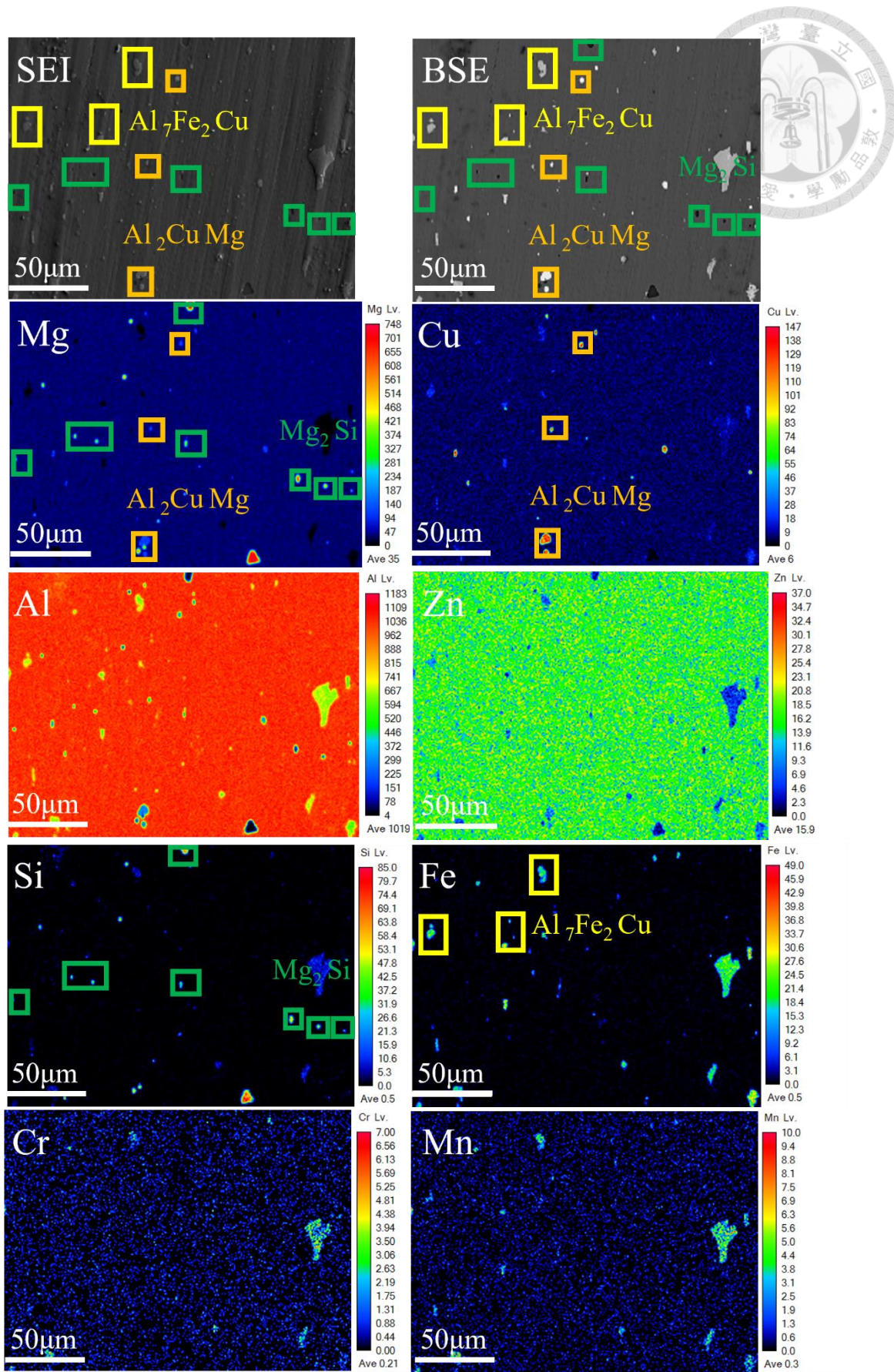
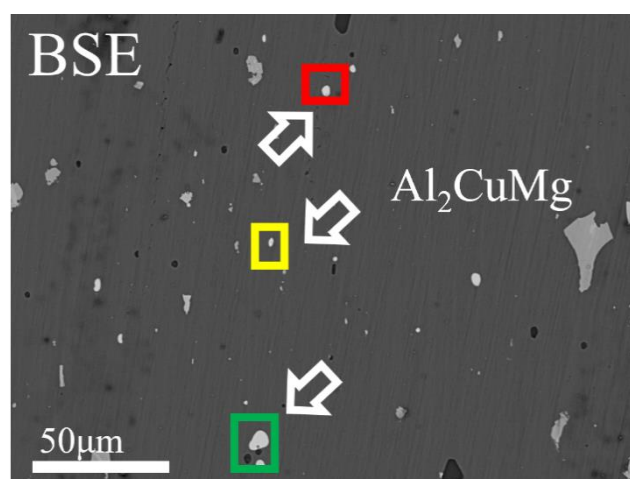
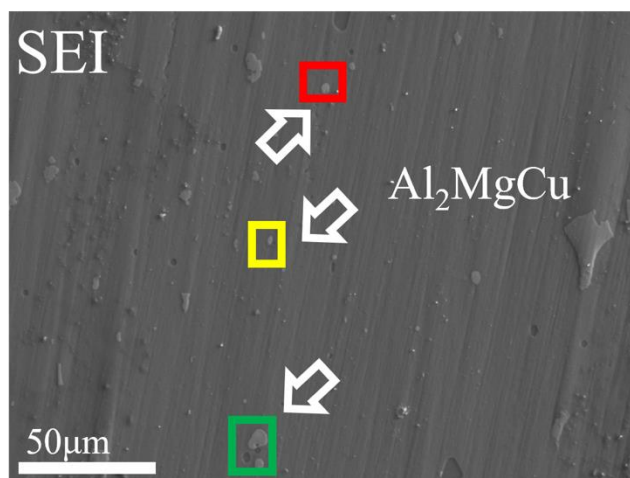


Figure 4-31 EPMA mapping of LCu after 475 °C 9h solution treatment with peak aging (orange : S; yellow : ω ; green : Mg_2Si holes).



S phase

S phase

S phase

WDS	at (%)
Al	53.5
Mg	22.2
Cu	22.5
Zn	1.6
Fe	0
Si	0
Mn	0

WDS	at (%)
Al	57.5
Mg	20.3
Cu	19.9
Zn	2.1
Fe	0
Si	0
Mn	0

WDS	at (%)
Al	50.8
Mg	23.5
Cu	23.8
Zn	1.6
Fe	0
Si	0
Mn	0

Figure 4-32 WDS elemental quantification of LCu after 475 °C 9h solution treatment with peak aging.

4.4.3 LCu after 490 °C 3h Solution Treatment

After the solution treatment at 490 °C for 3 hours, shown in Figure 4-33, there is a significant reduction in surface particles, leaving only a few behind, along with the formation of holes, which are presumed to be caused by incipient melting. To distinguish whether these holes are a result of polishing or the solution treatment, further examination with EPMA is necessary.

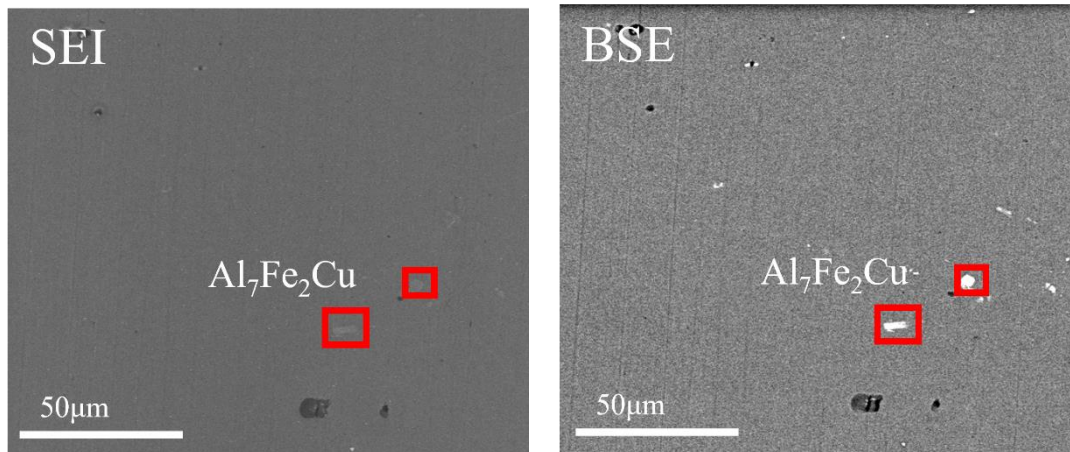


Figure 4-33 SEI and BEI of LCu after 490 °C 3h solution treatment with peak aging with EDS.

EPMA scans after the 490 °C for 3 hours treatment in Figure 4-34 show fewer surface particles than after the 475 °C for 9 hours treatment, but more holes are present. Detailed analysis indicates that the particles aggregate copper and iron elements, suggesting that these are not η or S phases but likely iron-containing ω phases, $\text{Al}_7\text{Fe}_2\text{Cu}$. Some holes (marked in green) show a high concentration of magnesium and silicon, likely caused by polishing with Mg_2Si , not related to the solution process. Other holes (marked in red) do not show magnesium or silicon, suggesting these were formed by incipient melting at high temperatures during solution treatment. Figure 4-35 shows the WDS quantification of remained second phases, which are recognized as ω phases.

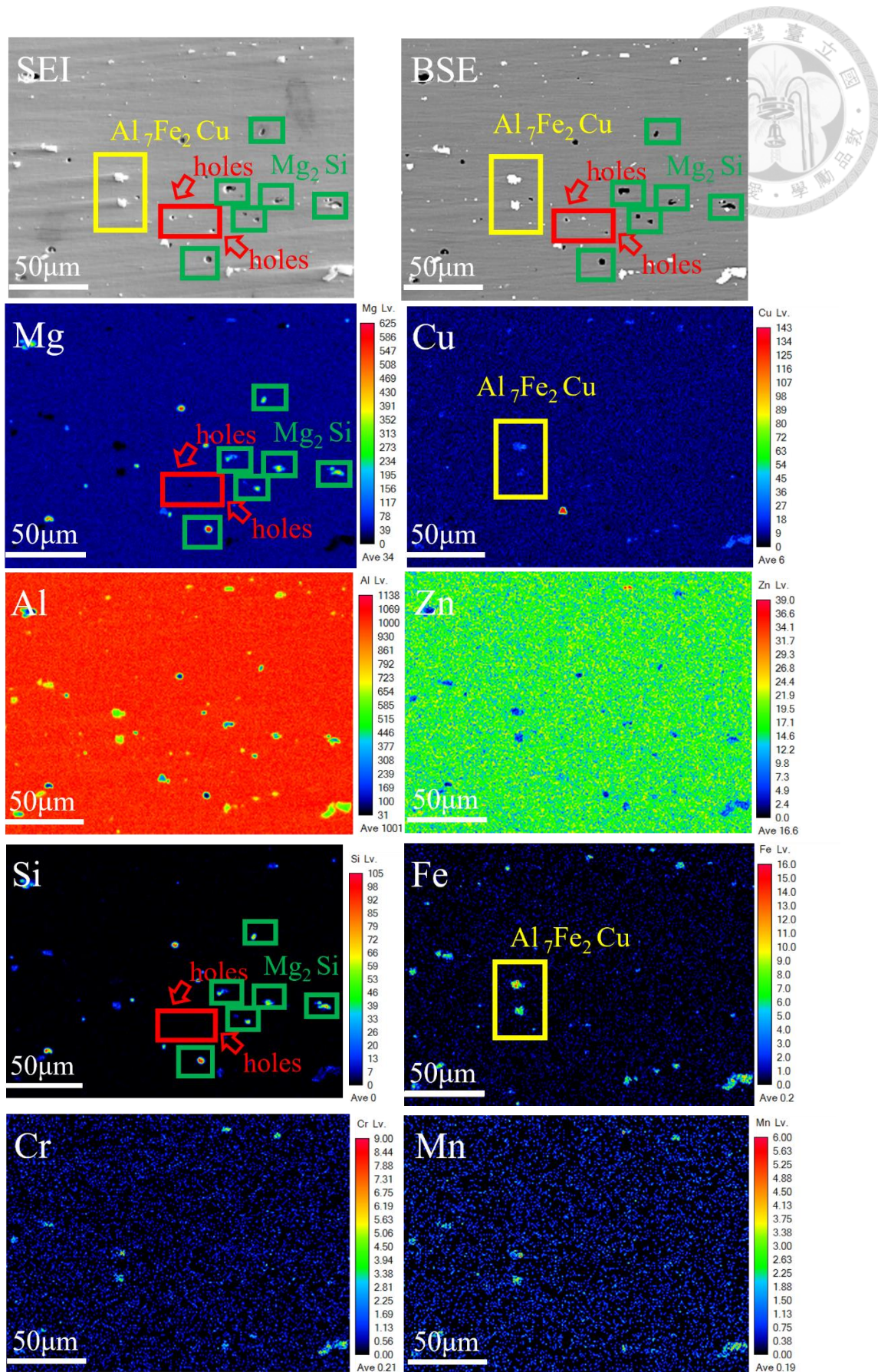
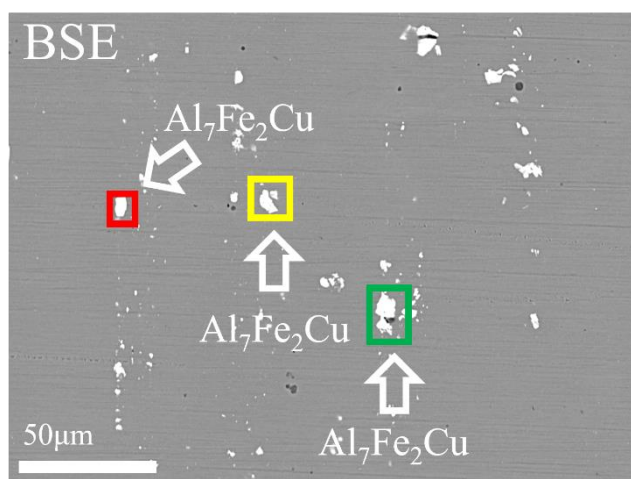
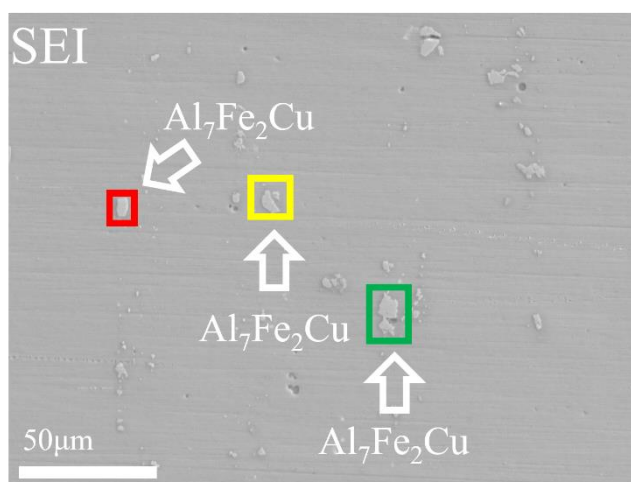


Figure 4-34 EPMA mapping of LCu after 490 °C 3h solution treatment with peak aging (yellow : ω ; green : Mg_2Si ; red : incipient melting).



ω phase

ω phase

ω phase

WDS	at (%)
Al	78.5
Mg	0.6
Cu	1.4
Zn	5.1
Fe	9.7
Si	1.3
Mn	2.9

WDS	at (%)
Al	81.7
Mg	0.23
Cu	1.6
Zn	3.0
Fe	11.7
Si	0.07
Mn	1.4

WDS	at (%)
Al	81.3
Mg	0.12
Cu	1.5
Zn	3.0
Fe	12.2
Si	0.02
Mn	1.5

Figure 4-35 WDS elemental quantification of LCu after 490 °C 3h solution treatment with peak aging.

4.4.4 LCu after 475 °C 9 h + 490 °C 3 h Solution Treatment

Shown in Figure 4-36 to Figure 4-38 preliminary comparisons indicate that the number of surface holes significantly increases after the 490 °C for 3 hours solution treatment compared to the process involving a first stage at 475 °C for 9 hours followed by 490 °C for 3 hours. The latter results in a smoother surface with fewer holes, indicating that two-stage solution treatment may effectively dissolve more second phases than a single-stage treatment.

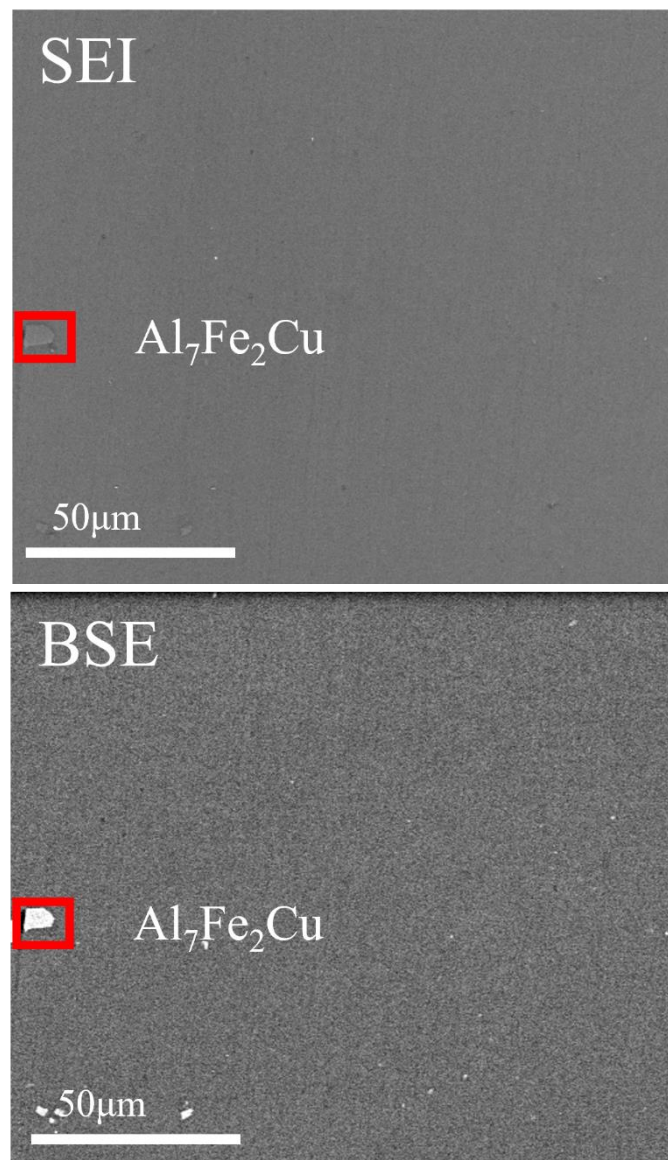


Figure 4-36 SEI and BEI of LCu after 475 °C 9 h+490 °C 3 h solution treatment with peak aging with EDS

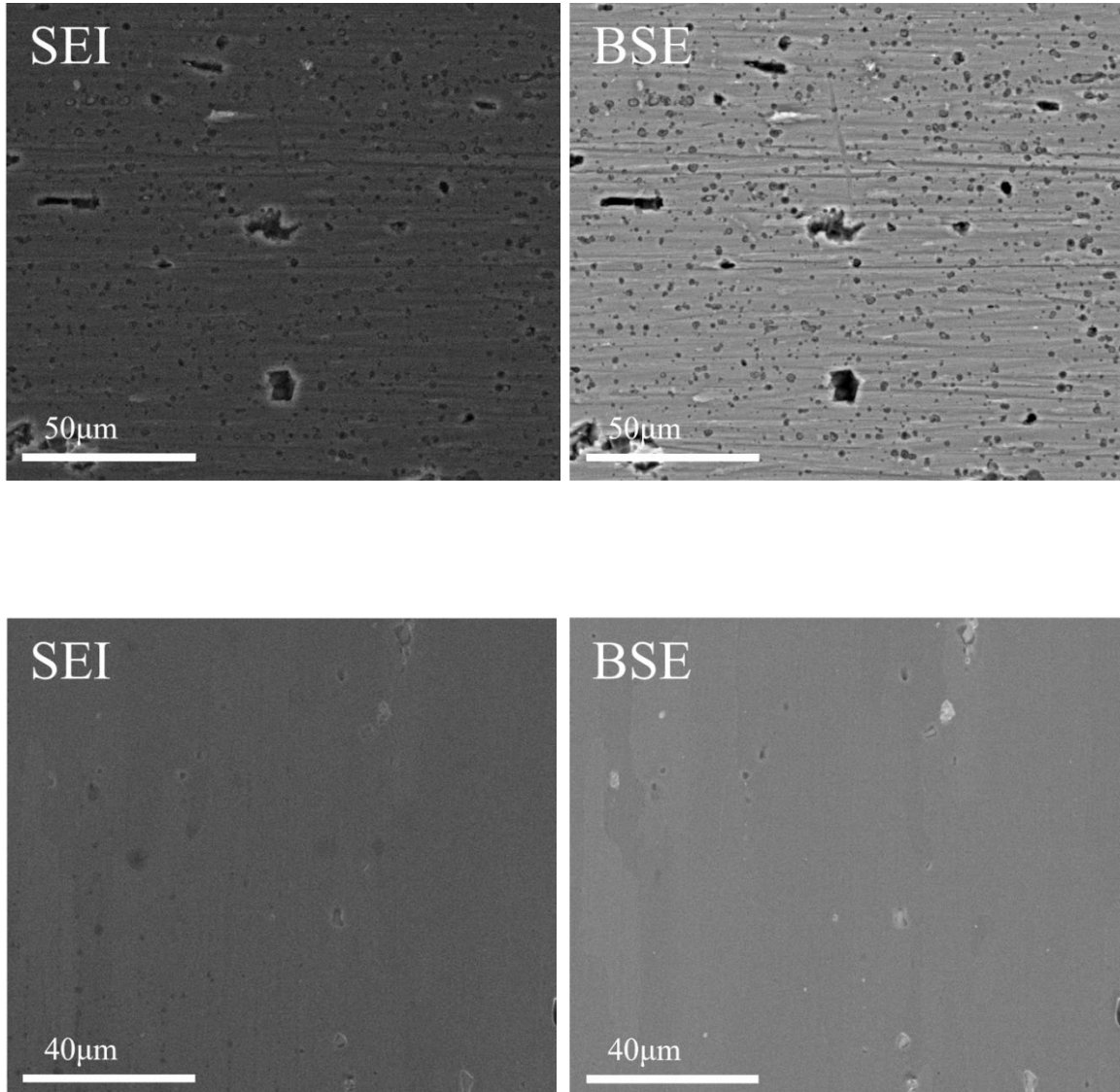


Figure 4-37 SEI and BEI of LCU after 490 °C 3 h and 475 °C 9 h+490 °C 3 h solution treatment with peak aginon condition of mechanical polishing.

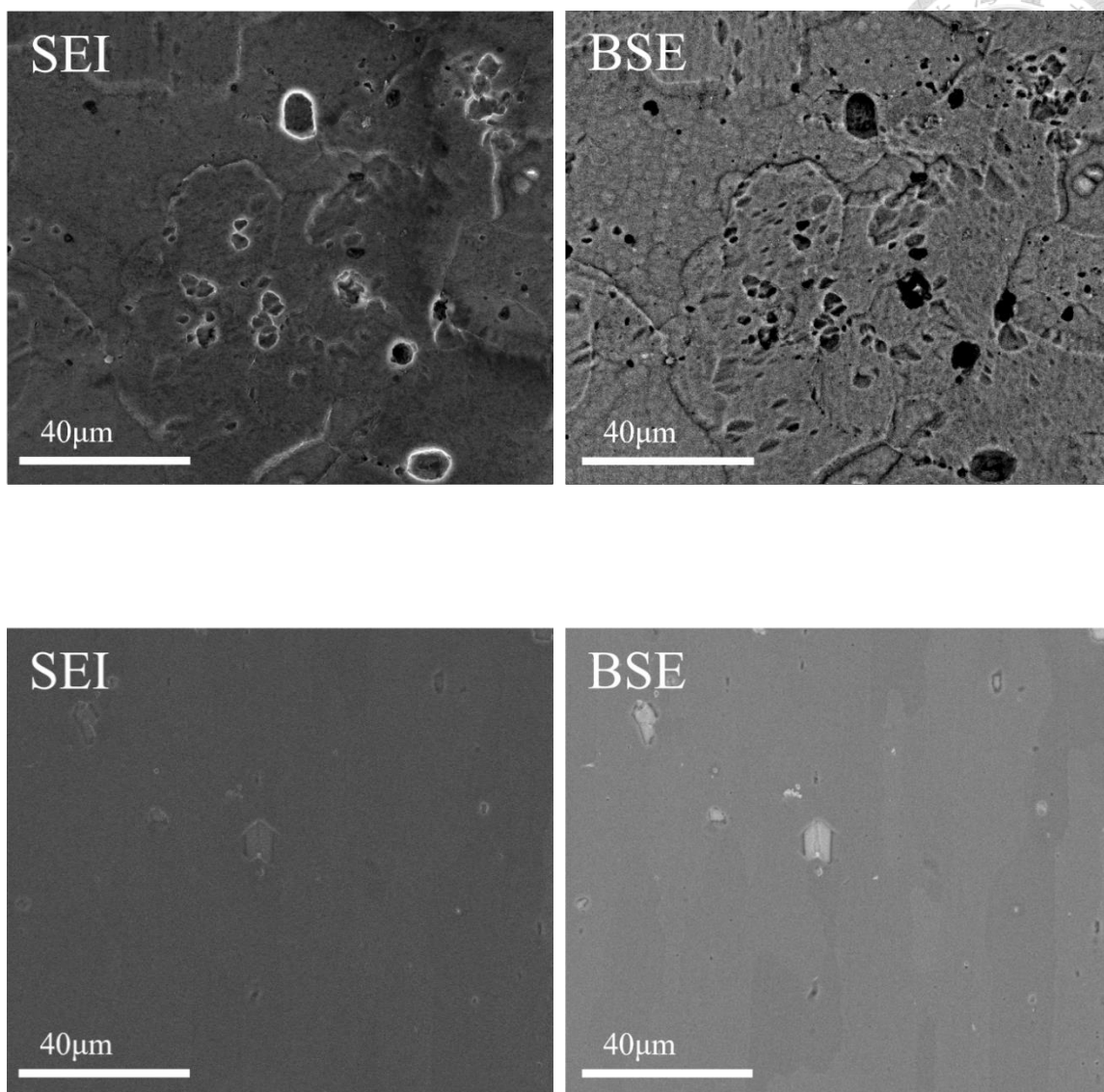


Figure 4-38 SEI and BEI of LCu after 490 °C 3 h and 475 °C 9 h+490 °C 3 h solution treatment with peak aging on condition of electrolytic polishing.

After the two-stage solution treatment (475 °C for 9 hours followed by 490 °C for 3 hours), shown in Figure 4-39, EPMA scans show very few particles on the aluminum alloy base and a smooth surface. The elemental distribution suggests these particles are primarily copper and iron aggregates, similar to the ω phase observed after 490 °C for 3 hours treatment. Although there are some holes on the surface, they contain a high concentration of magnesium and silicon, indicating they were likely caused by Mg_2Si during polishing, not by incipient melting. Furthermore, in WDS quantification shown in Figure 4-40, the second phases are indeed ω phases.

The key difference between the single-stage and two-stage solution treatments is that the former has holes indicative of incipient melting on the surface, suggesting that the two-stage solution treatment, particularly the addition of a first stage at 475 °C, effectively resolves the incipient melting issues associated with a single-stage solution treatment at 490 °C.

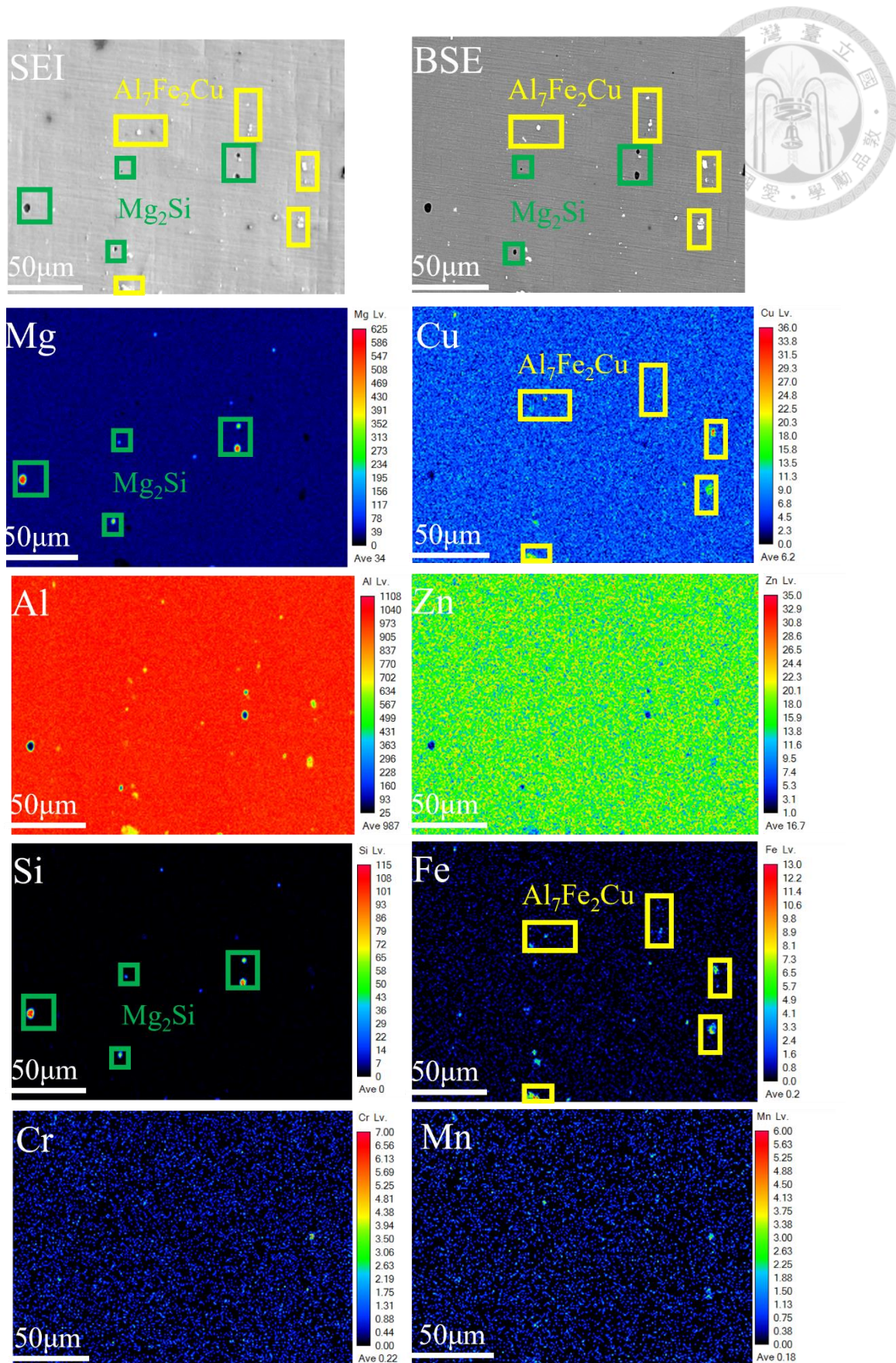
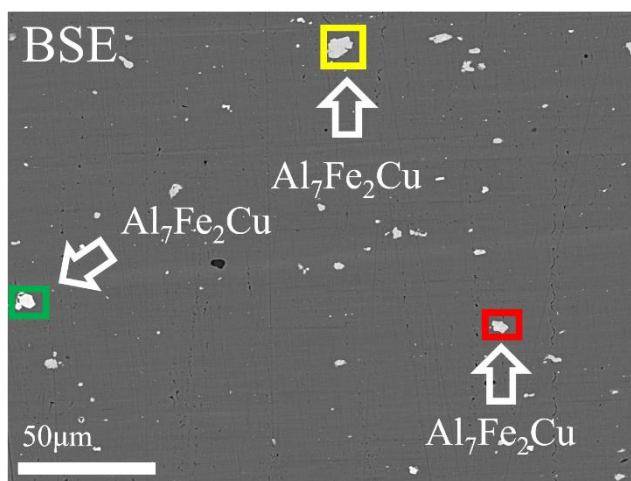
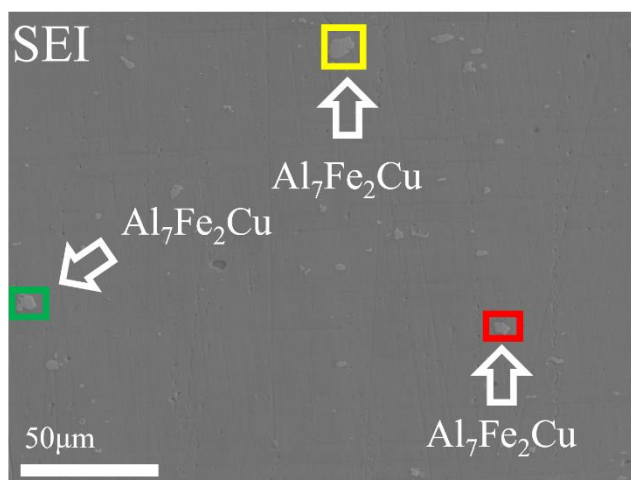


Figure 4-39 EPMA mapping of LCU after 475 °C 9h + 490 °C 3h solution treatment with peak aging (yellow : ω ; green : Mg_2Si).



ω phase

ω phase

ω phase

WDS	at (%)
Al	78.0
Mg	0.2
Cu	1.0
Zn	2.6
Fe	11.0
Si	3.8
Mn	3.0

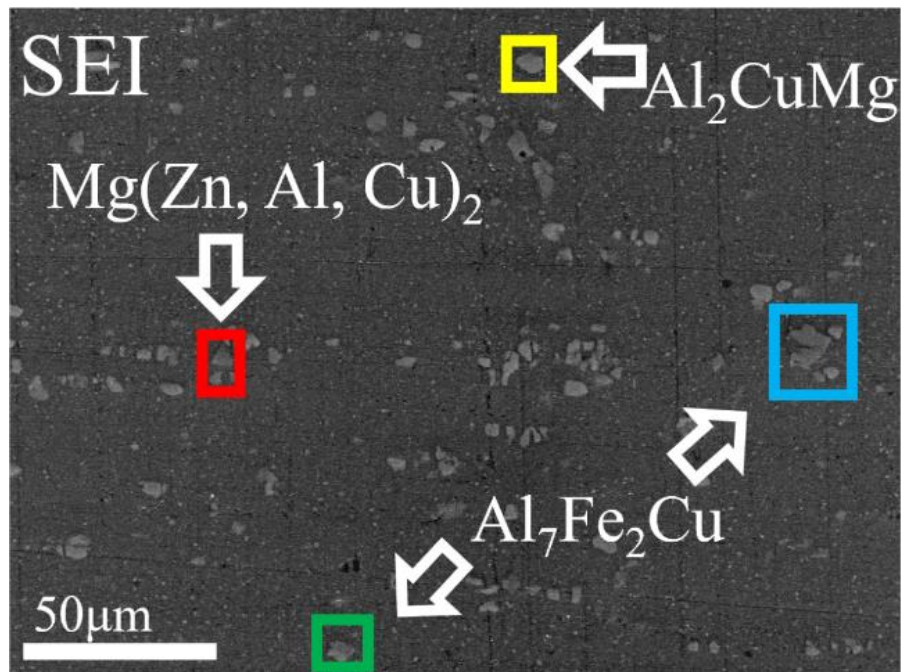
WDS	at (%)
Al	82
Mg	0.08
Cu	1.4
Zn	2.7
Fe	11.7
Si	0.04
Mn	1.83

WDS	at (%)
Al	78.9
Mg	0.4
Cu	1.3
Zn	4.7
Fe	9.8
Si	1.3
Mn	3.2

Figure 4-40 WDS elemental quantification of LCu after 475 °C 9h + 490 °C 3h solution treatment with peak aging.

4.4.5 As-received HCu

In Figure 4-41, observing HCu raw materials under SEM reveals an uneven surface with many particles visible in the SEI mode, which appear bright in the BSE mode, indicating the aggregation of heavy elements. This suggests that these particles are undissolved second phases. Further EDX analysis of the surface particles reveals that the main second phases are the same as those in LCu: the η phase $\text{Mg}(\text{Zn}, \text{Al}, \text{Cu})_2$, the S phase Al_2CuMg , and the iron-containing ω phase, $\text{Al}_7\text{Cu}_2\text{Fe}$. The band-like distribution on the surface is likely the η phase, primarily composed of magnesium, zinc, and aluminum, while the small spherical shapes are the S phase, with main elemental signals of aluminum, magnesium, and copper. The irregular shapes are identified as the iron-containing ω phase. Preliminary observations indicate that, compared to LCu, HCu has a higher proportion of the S phase on the surface, consistent with literature findings that suggest this is due to the higher Cu content in HCu.



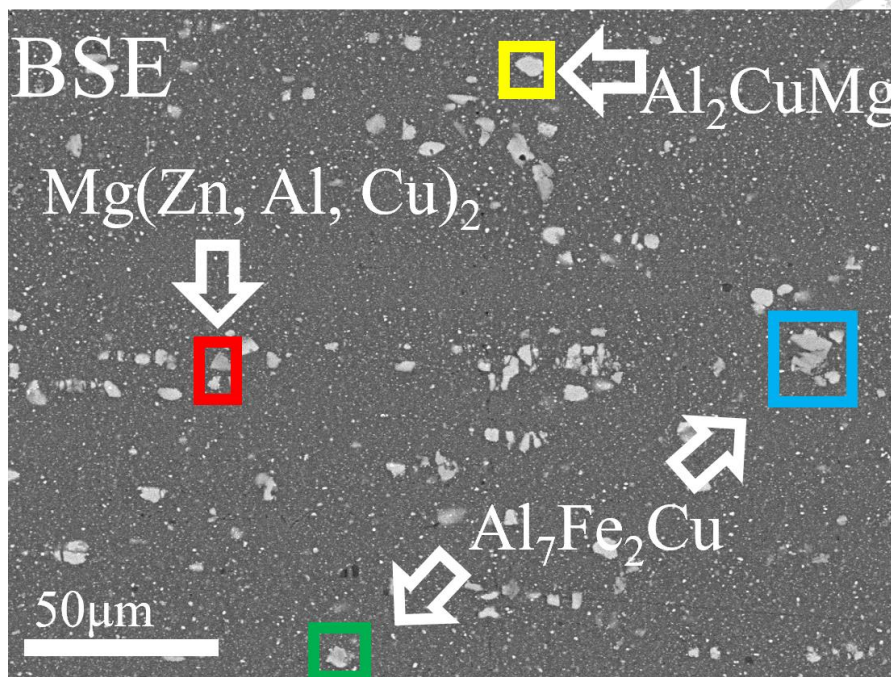


Figure 4-41 SEI and BEI of HCu as-received with EDS(red : η ; yellow : S; green, blue : ω).

Shown in Figure 4-42, after EPMA scanning, it is observed that most surface particles have an accumulation of Cu elements, with some particles showing iron element signals, suggesting they are predominantly the S phase and the ω phase, respectively, with only a few areas having an accumulation of zinc elements, indicating a lower proportion of the η phase. Compared to LCu, there is a significant reduction in the quantity of the η phase on the surface, and an evident increase in the S phase, which may be attributed to the higher copper content in HCu, as indicated by literature. Figure 4-43 indicates actual WDS point analysis of the surface element distribution, suggesting that particles with high zinc content could be the η phase. However, due to the thinness of the second phases, the electron beam penetrates and receives signals from the aluminum alloy base, resulting in an overrepresentation of aluminum signals. Analysis of other particles aggregating copper and magnesium elements, where aluminum constitutes about 50% and copper and magnesium each about 20%, with an elemental ratio approximately 2:1:1, confirms them as the S phase, Al_2CuMg .

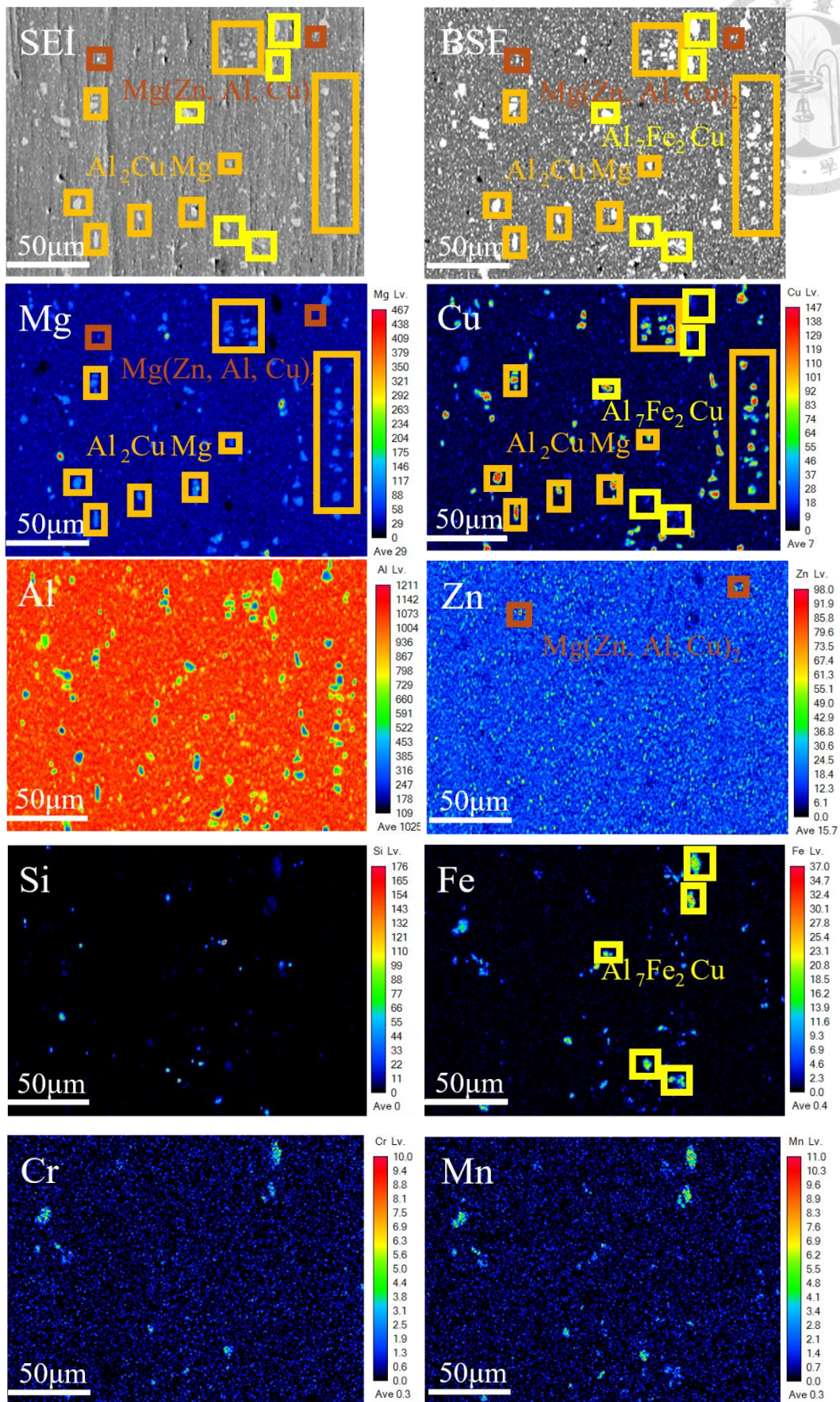
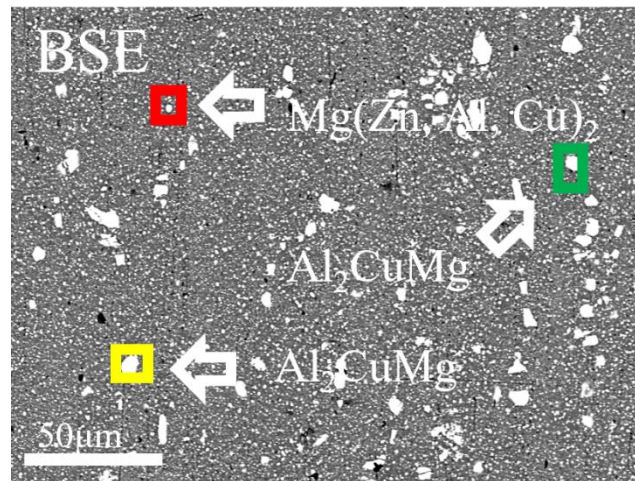
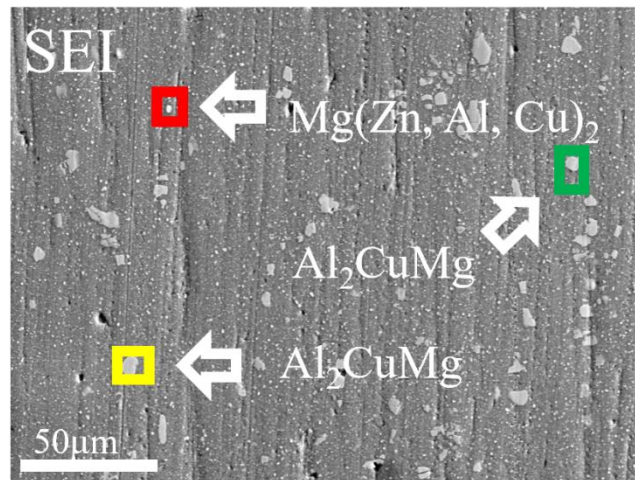


Figure 4-42 EPMA mapping of HCu as-received (brown : η ; orange : S; yellow : ω).



η phase		S phase		S phase	
WDS	at (%)	WDS	at (%)	WDS	at (%)
Al	53.0	Al	52.2	Al	58.4
Mg	17.5	Mg	22.8	Mg	17.8
Cu	7.0	Cu	23.2	Cu	19.1
Zn	21.9	Zn	1.6	Zn	1.5
Fe	0	Fe	0.0	Fe	2.4
Si	0.0	Si	0.0	Si	0.0
Mn	0.1	Mn	0.0	Mn	0.3

Figure 4-43 WDS elemental quantification of as-received HCu.

4.4.6 HCu after 475 °C 3 h Solution Treatment

After undergoing the 475 °C solution treatment for 3 hours, in Figure 4-44 HCu materials showed a significant reduction in surface particles compared to the untreated raw material, indicating that most second phases have dissolved back into the base, with only a few particles remaining. The surface appeared smooth and almost hole-free. EDS spot qualitative analysis revealed that the elemental signals of the remaining particles were primarily aluminum, copper, and iron, with lower zinc signals, suggesting that these particles are likely the S phase, Al_2CuMg , and the iron-containing ω phase, $\text{Al}_7\text{Cu}_2\text{Fe}$. This indicates that the η phase has likely dissolved back into the base, showing a trend similar to LCu.

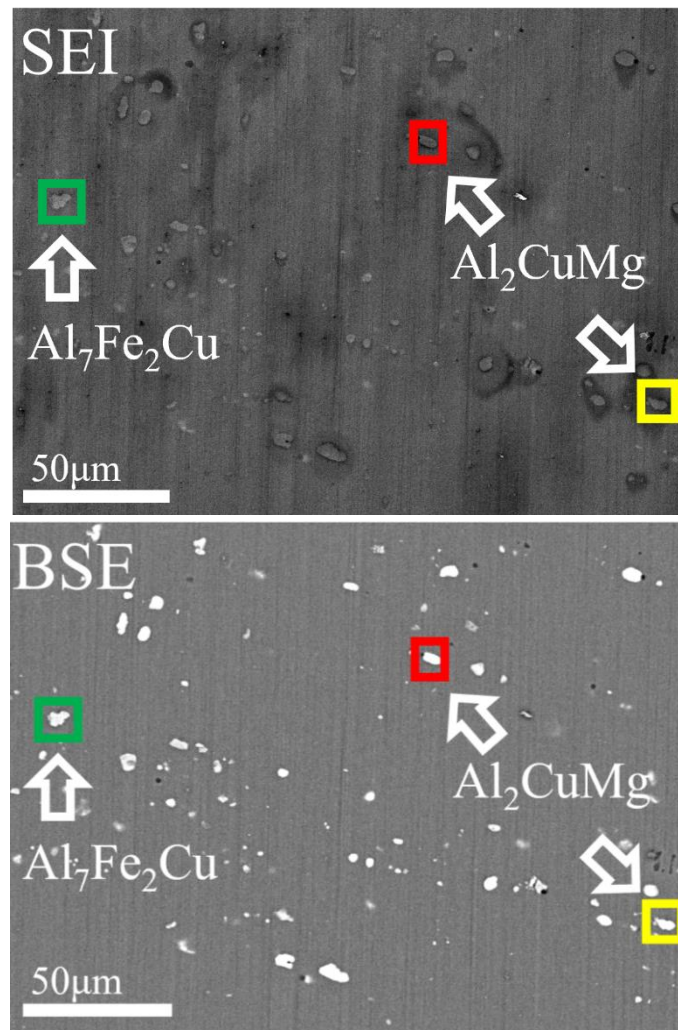


Figure 4-44 SEI and BEI of HCu after 475 °C 3 h solution treatment with peak aging with EDS (red ,yellow : S; green : ω).

Figure 4-45 is EMPA mapping of HCu after 475 °C 3 h solution treatment with peak aging, revealing a concentration of copper elements on the surface, distinguishing between particles aggregating copper and magnesium elements, and those aggregating copper and iron elements, likely corresponding to the S phase and the ω phase, respectively. Most particles on the HCu surface after the first stage of solution treatment showed no iron element signals, identifying them as the S phase. This is in contrast to LCu, where the first stage of solution treatment dissolved more of the second phases. Despite observing some holes on the surface in SEI and BSE modes, elemental scanning indicated they contained significant magnesium and silicon signals, suggesting these holes were caused by polishing with Mg₂Si, not by solution treatment.

Detailed WDS point analysis of the elemental composition in Figure 4-46 revealed that the small spherical second phases were primarily composed of about 50% aluminum and roughly 25% each of copper and magnesium, with almost no other elements present, identifying them as the S phase, Al₂CuMg. The irregularly shaped second phases showed a majority of aluminum signals, about 70%, with iron making up about 10%, and the remainder being copper, zinc, and other elements, identifying these as the ω phase, Al₇Fe₂Cu. In summary, the conclusions drawn for HCu materials are similar to those for LCu, the η phase can be dissolved back into the aluminum alloy base during the 475°C solution treatment stage, but the S phase and ω phase remain, with a greater proportion of the S phase in HCu than in LCu.

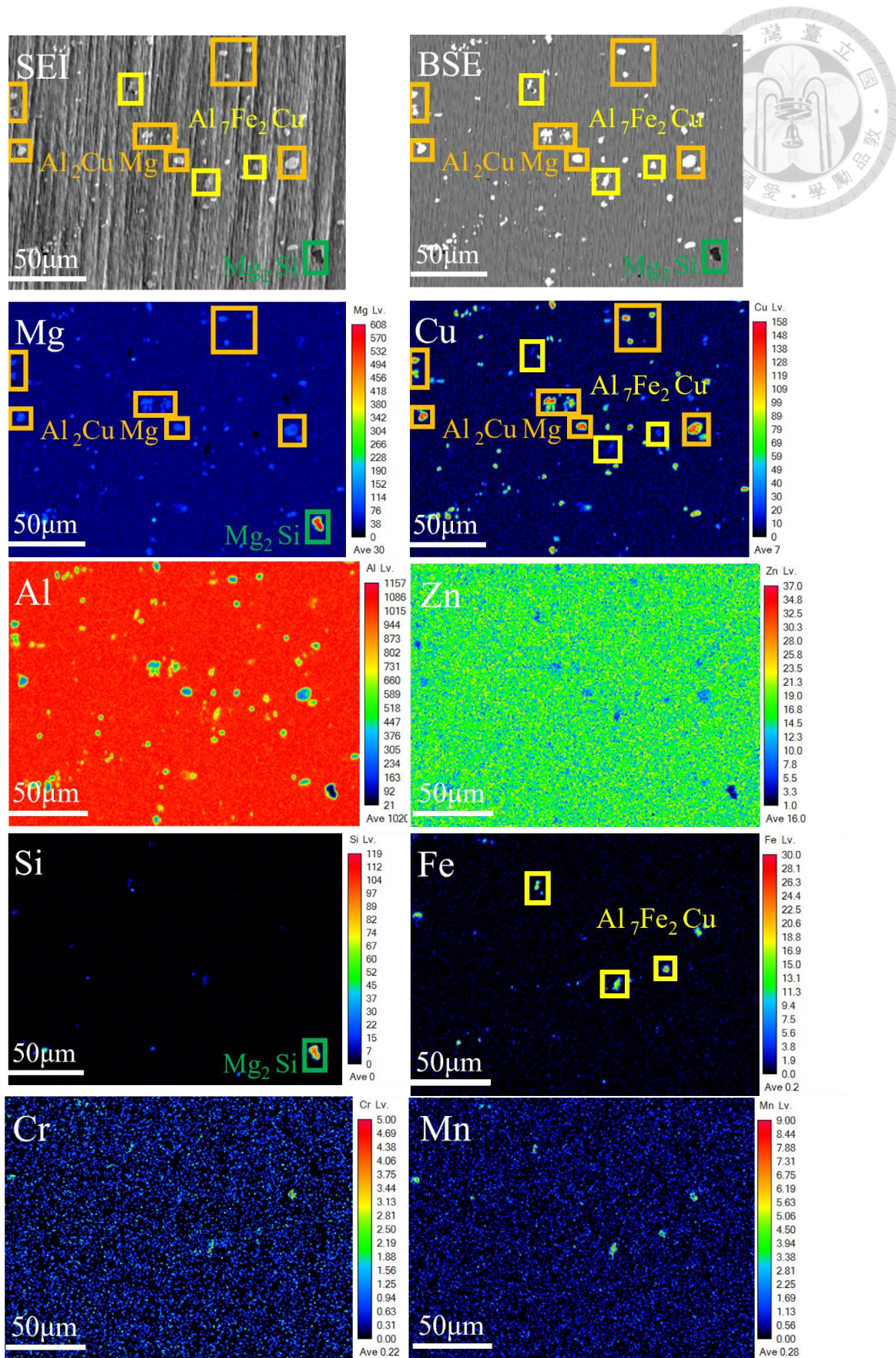
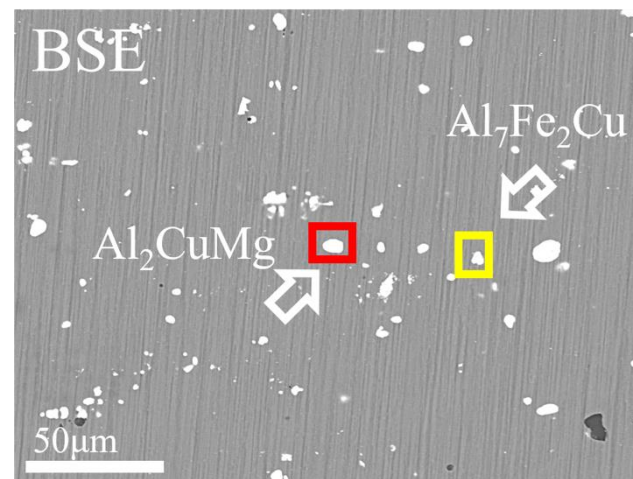
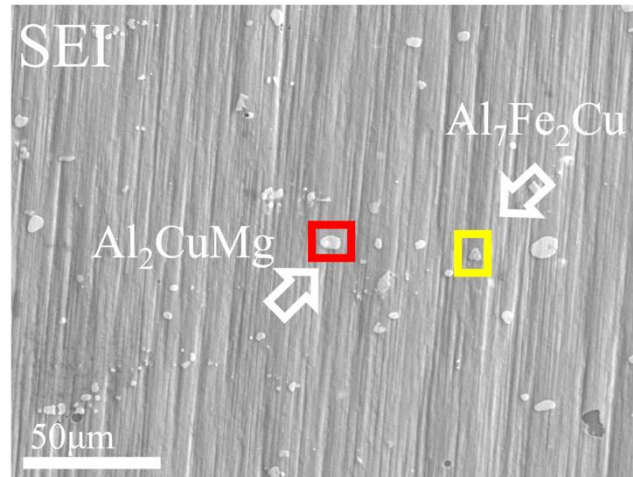


Figure 4-45 EPMA mapping of HCu after 475 °C 3 h solution treatment with peak aging (orange : S; yellow : ω ; green : Mg_2Si holes).



S phase

WDS	at (%)
Al	50.2
Mg	22.4
Cu	25.4
Zn	1.7
Fe	0
Si	0
Mn	0

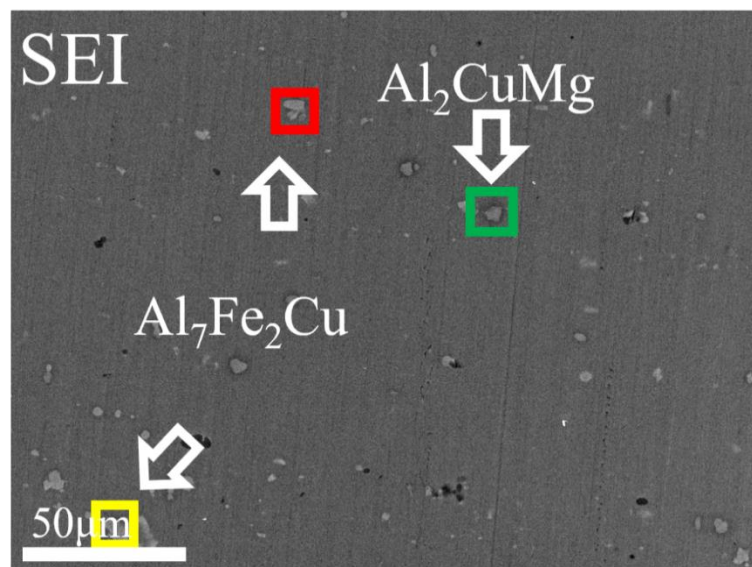
ω phase

WDS	at (%)
Al	75.8
Mg	0.5
Cu	4.9
Zn	1.4
Fe	9.6
Si	1.3
Mn	2.9

Figure 4-46 WDS elemental quantification of HCu after 475 °C 3 h solution treatment with peak aging.

4.4.7 HCu after 490 °C 3 h solution treatment

After the HCu material was subjected to a 490 °C solution treatment for 3 hours, it was observed SEI in Figure 4-47, compared to the condition after a 475 °C solution treatment for 3 hours, the overall number of particles had further decreased, but some holes appeared on the surface of the sample, a trend similar to that observed with LCu. EDX detection of elemental signals from the surface particles indicated a presence of iron-based signals, suggesting that the second phase might be the ω phase. In addition to particles containing iron, a few particles only containing aluminum, magnesium, and copper signals were detected, suggesting these might be incompletely dissolved S phases. This phenomenon differs from LCu, where a 490 °C solution treatment for 3 hours was sufficient to completely dissolve the S phase, suggesting the difference in copper content might cause HCu to have a higher S phase content, hence the residual presence after 490 °C solution treatment for 3 hours.



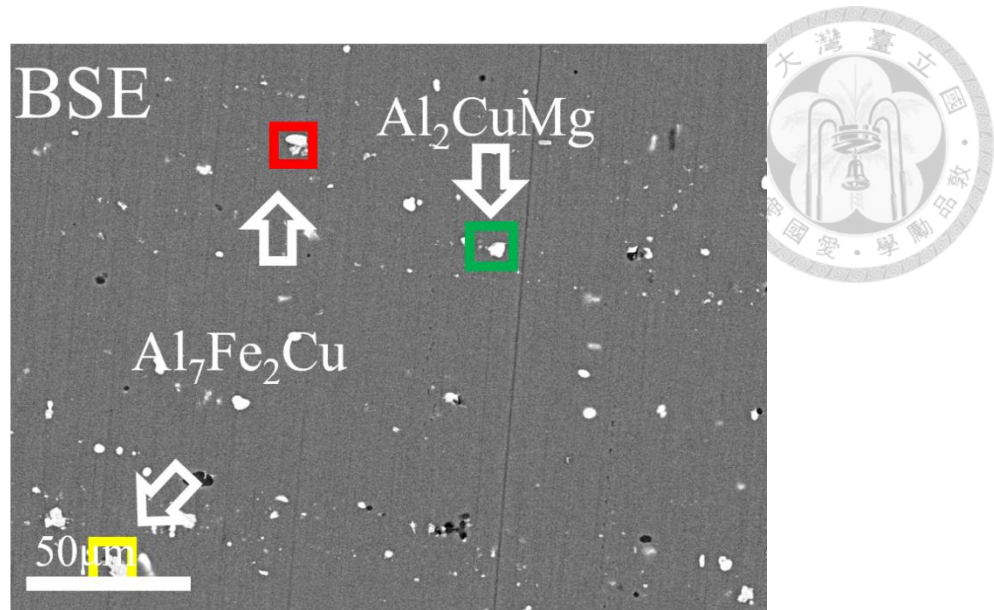


Figure 4-47SEI and BEI of HCu after 490 °C 3 h solution treatment with peak aging with EDS (red ,yellow : ω ; green : S).

EPMA scanning in Figure 4-48 of samples treated at 490 °C for 3 hours revealed a large concentration of copper elements on the remaining particles, as well as magnesium and iron elements, indicating the presence of both the S and ω phases. The treatment was unable to fully dissolve the S phase, although surface S phase particles appeared significantly smaller, suggesting a need to extend the total solution treatment time to fully dissolve the S phase back into the base. Figure 4-49 and Figure 4-50 are WDS point analysis. Former confirmed the presence of the ω phase remaining on the surface.

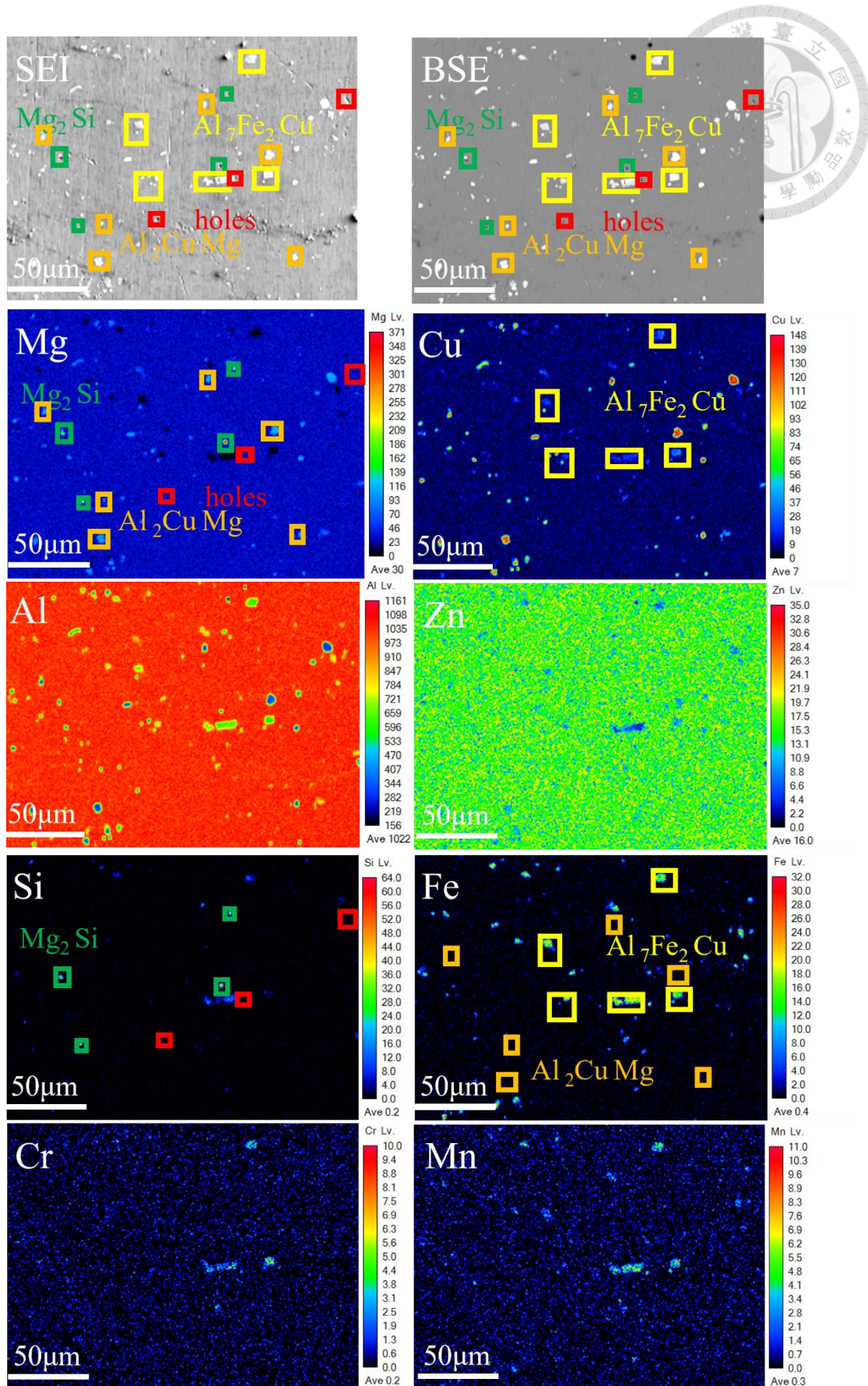
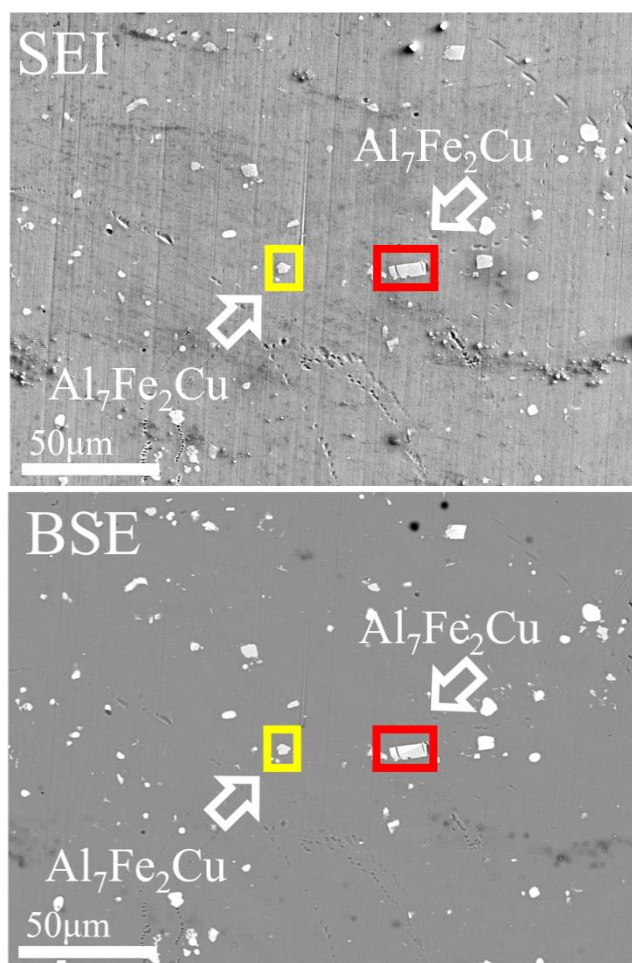


Figure 4-48 EPMA mapping of HCu after 490 °C 3 h solution treatment with peak aging (orange : S; yellow : ω ; green : Mg_2Si ; red : incipient melting).



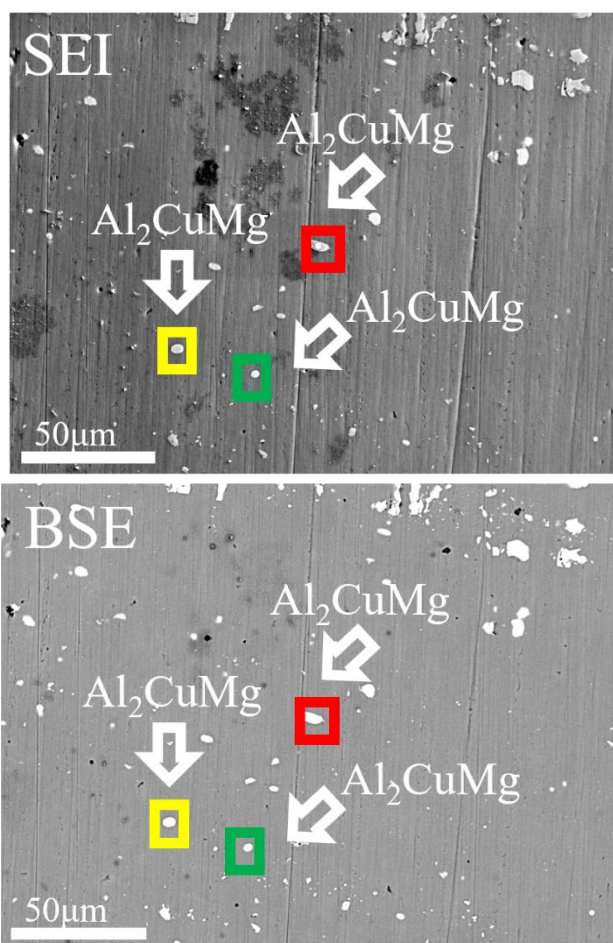
ω phase

ω phase

WDS	at (%)
Al	76
Mg	0.2
Cu	3.4
Zn	0.9
Fe	11.8
Si	2.3
Mn	2.8

WDS	at (%)
Al	81.7
Mg	0.3
Cu	3
Zn	1.4
Fe	11.6
Si	0
Mn	1.5

Figure 4-49 WDS elemental quantification of HCu after 490 °C 3 h solution treatment with peak aging place 1.



S phase		S phase		S phase	
WDS	at (%)	WDS	at (%)	WDS	at (%)
Al	52.0	Al	53.9	Al	53.8
Mg	23.2	Mg	21.8	Mg	22.1
Cu	22.9	Cu	21.8	Cu	21.7
Zn	1.5	Zn	1.8	Zn	1.7
Fe	0	Fe	0.0	Fe	0.0
Si	0.1	Si	0.0	Si	0.0
Mn	0.0	Mn	0.1	Mn	0.1

Figure 4-50 WDS elemental quantification of HCu after 490°C 3h solution treatment with peak aging place 2.

4.4.8 HCu after 475 °C 3 h + 490 °C 3 h Solution Treatment

Under the condition of two-stage solution treatment with 475 °C for 3 hours followed by 490 °C for 3 hours, SEI and BSE in Figure 4-51 showed a low number of particles on the sample surface, and the surface was smoother with fewer holes than samples treated at 490 °C for 3 hours alone. EDS analysis of the surface particles detected iron signals, suggesting the residual particles might be the ω phase, and the S phase might have dissolved back into the base after the additional 475 °C stage. The number of second phases was lower than in samples treated only at 490 °C for 3 hours. In Figure 4-52, the difference in the presence of holes on the surface was less pronounced in HCu compared to LCu, possibly due to a lower proportion of the η phase in HCu, thus resulting in fewer issues related to incipient melting at 490 °C.

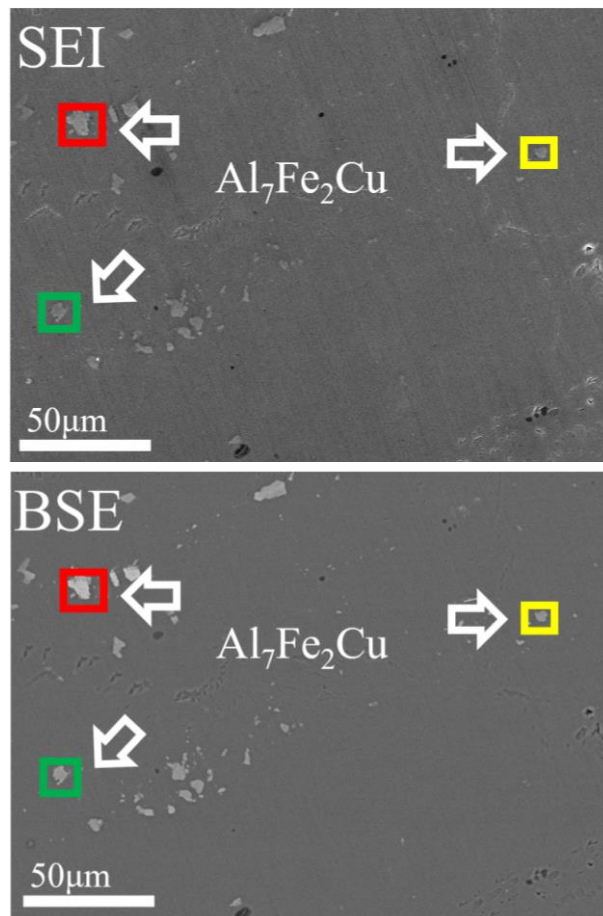


Figure 4-51 SEI and BEI of HCu after 475 °C 3 h + 490 °C 3 h solution treatment with peak aging with EDS (red ,yellow ,green : S).

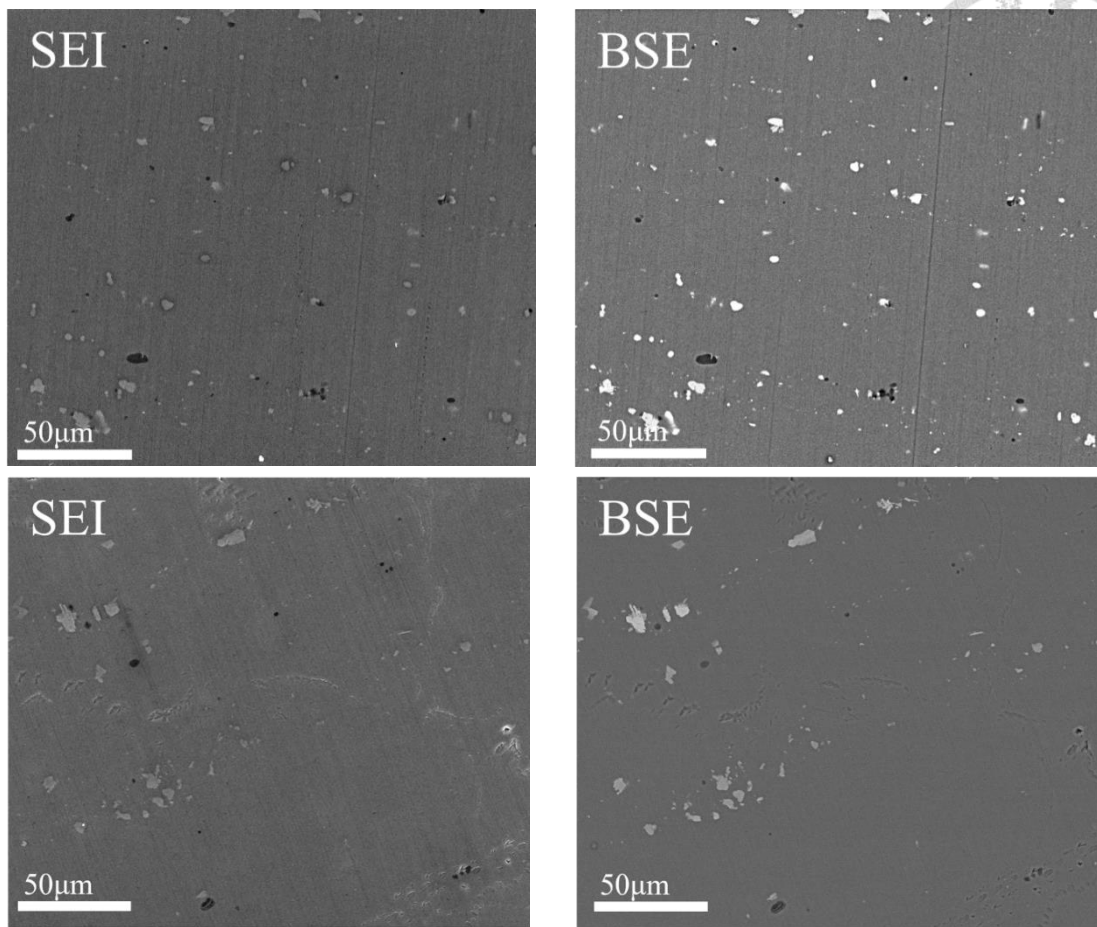


Figure 4-52 SEI and BEI of LCu after 490 °C 3 h and 475 °C 9 h+490 °C 3 h solution treatment with peak aginon condition of mechanical polishing.

EPMA scanning of samples treated with a two-stage solution treatment of 475 °C for 3 hours followed by 490 °C for 3 hours in Figure 4-53 showed fewer particles on the sample surface, and signals for copper and iron elements were detected, suggesting the remaining second phase is the ω phase, $\text{Al}_7\text{Fe}_2\text{Cu}$. Magnesium signals were not detected, indicating the S phase had fully dissolved back into the base. Holes detected on the surface contained magnesium and silicon signals, suggesting they were caused by polishing with Mg_2Si , not by incipient melting. In Figure 4-54, WDS point analysis confirmed the remaining phase as ω phase, $\text{Al}_7\text{Fe}_2\text{Cu}$, with magnesium elements being less than 1%, indicating that both the η and S phases can fully dissolve back into the base after two-stage solution treatment, leaving only the ω phase on the surface without holes caused by incipient melting.

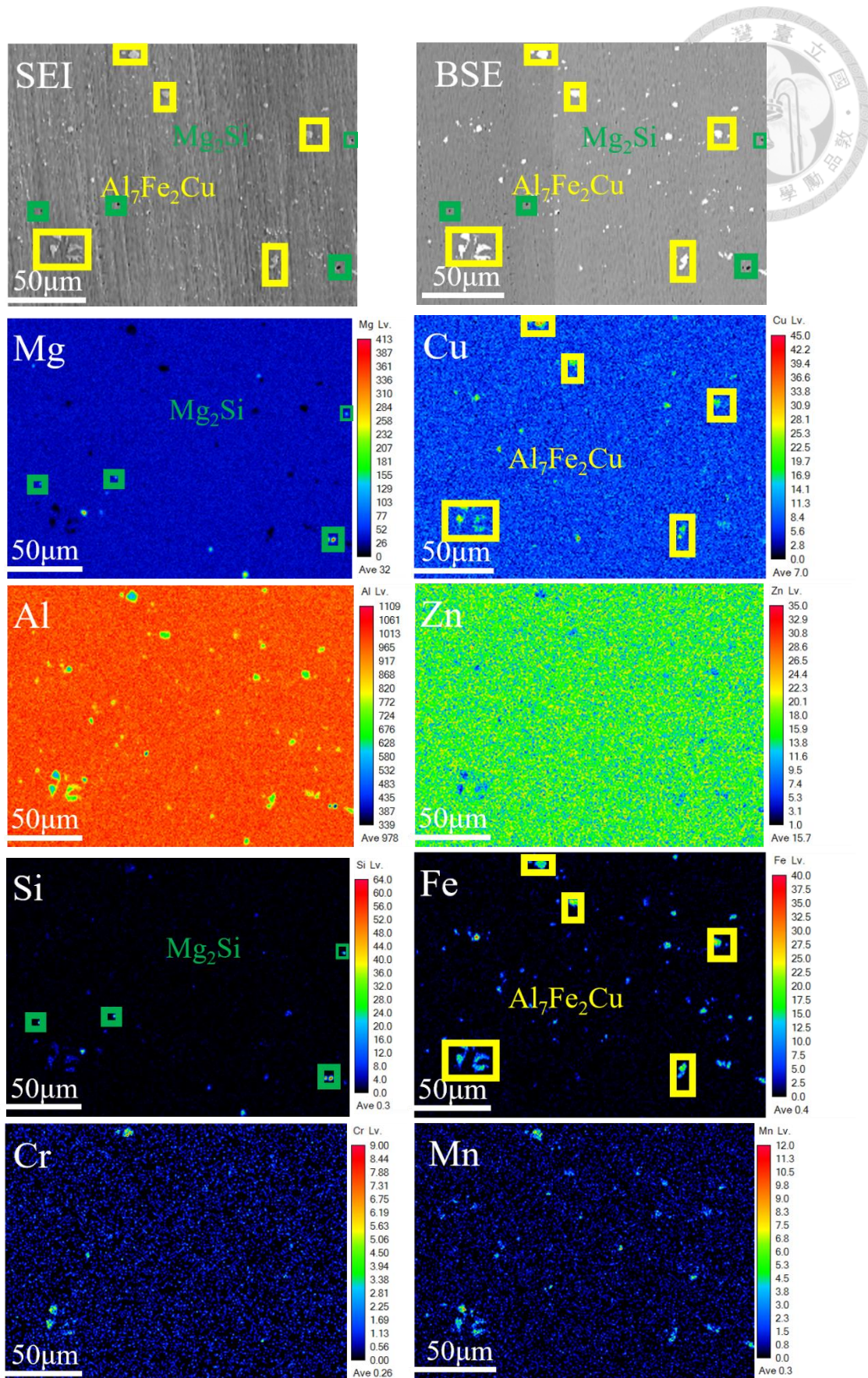
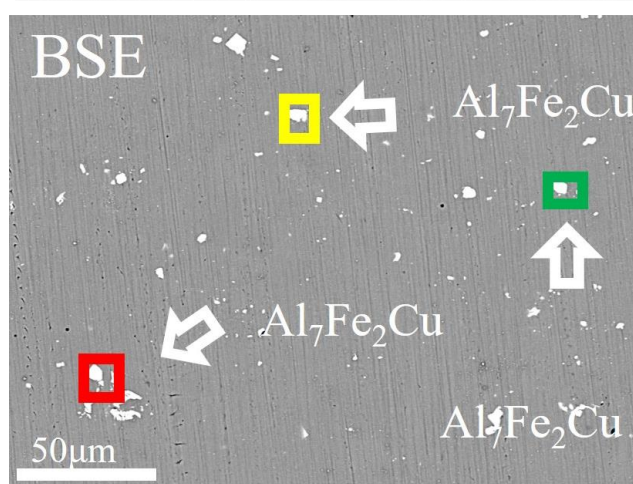
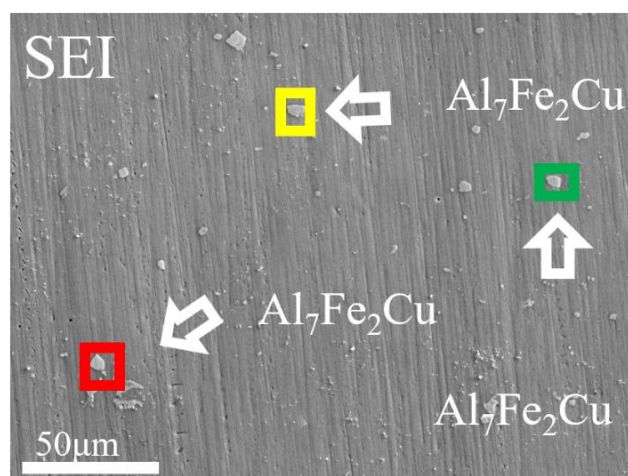


Figure 4-53 EPMA mapping of HCu after 475 °C 3 h + 490 °C 3 h solution treatment with peak aging (yellow : ω ; green : Mg_2Si).



ω phase

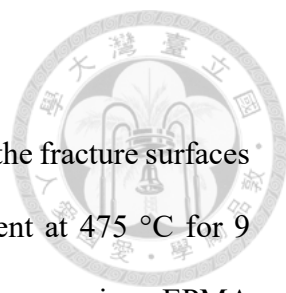
ω phase

ω phase

WDS	at (%)	WDS	at (%)	WDS	at (%)
Al	75.5	Al	81.2	Al	81.1
Mg	0.5	Mg	0.1	Mg	0.1
Cu	5.7	Cu	3.2	Cu	3.2
Zn	1.3	Zn	1.4	Zn	1.3
Fe	8.8	Fe	12.0	Fe	12.4
Si	1.1	Si	0.0	Si	0.0
Mn	3.2	Mn	1.4	Mn	1.4

Figure 4-54 WDS elemental quantification of HCu after 475 °C 3 h + 490 °C 3 h solution treatment with peak aging.

4.4.9 Fracture surface



Shown in Figure 4-55, after tensile testing and fracturing, SEI of the fracture surfaces revealed that in LCu, following the sole first-stage solution treatment at 475 °C for 9 hours, second phases were observed on the fracture surface. Based on previous EPMA analysis, these could likely lead to fracture due to the presence of S phases. In the LCu process involving only the second-stage solution treatment at 490 °C for 3 hours, pores scattered on the fracture surface were visible, potentially caused by incipient melting. Although not all pores might be due to incipient melting, the fracture surface indeed showed more pores compared to other processes, possibly leading to stress concentration reasons. On the fracture surface of LCu after two-stage solution treatment of 475 °C for 9 hours followed by 490 °C for 3 hours, a noticeable reduction in pores was observed compared to the sole second-stage solution, with no second phases observed on the surface, suggesting that the improved ductility could be due to the two-stage solution heat treatment.

The difference was more pronounced in HCu shown in Figure 4-56. Following the sole first-stage solution treatment at 475 °C for 3 hours, a significant amount of second phases was observable on the fracture surface, aligning with the observations from EPMA. This suggests that fractures might occur along grain boundaries where second phases are located during tensile testing, leading to poor mechanical properties. On the fracture surface after the sole second-stage solution treatment at 490 °C for 3 hours, noticeably more surface pores were observed compared to other processes, possibly related to incipient melting, contributing to the higher number of surface pores. Moreover, many second phases interspersed with pores were observed on the fracture surface, potentially undissolved S phases, as indicated by EPMA results. This differs from LCu, possibly due to different Cu contents. In the two-stage solution treatment process of HCu, the surface

appeared smooth with fewer pores and almost no second phases, suggesting this as a reason for better mechanical properties. In summary, the fracture surfaces after different stages of solution treatment exhibit various differences, potentially related to the residue of second phases and incipient melting, supporting the efficacy of two-stage solution treatment.

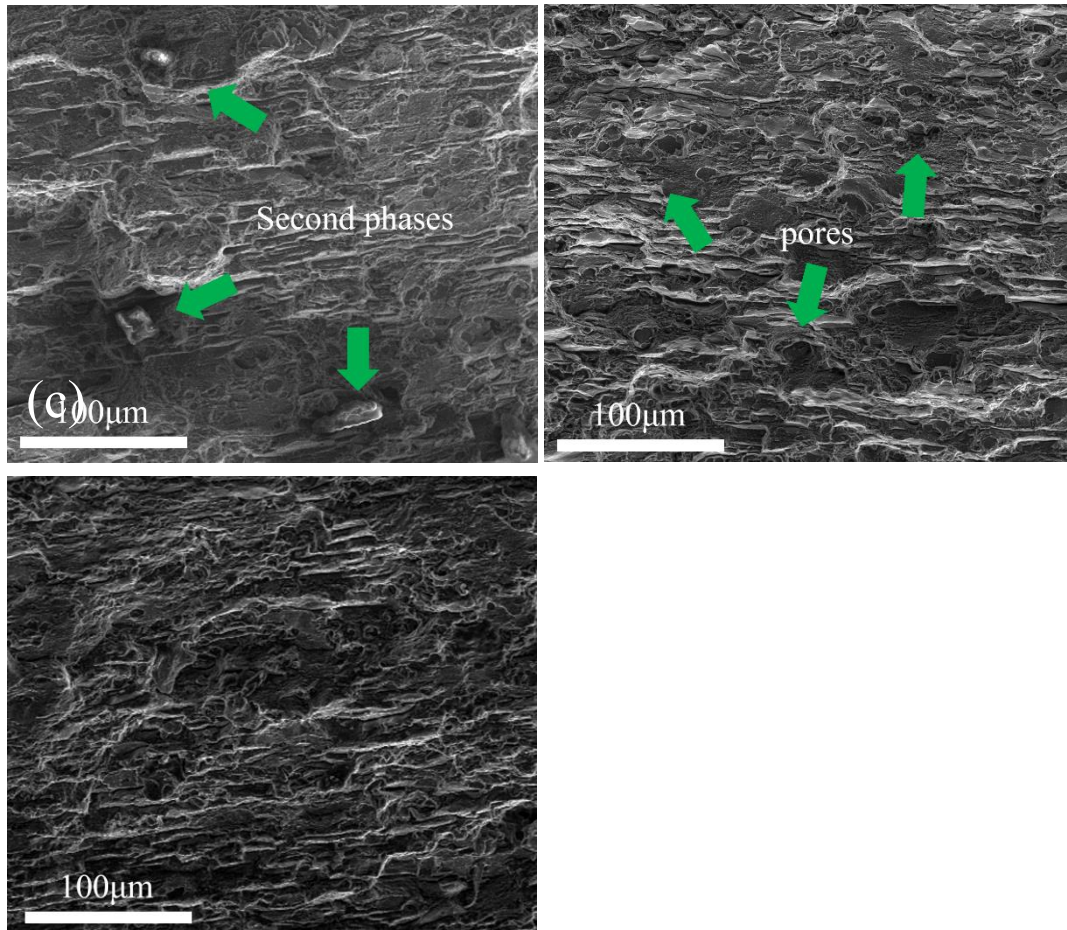
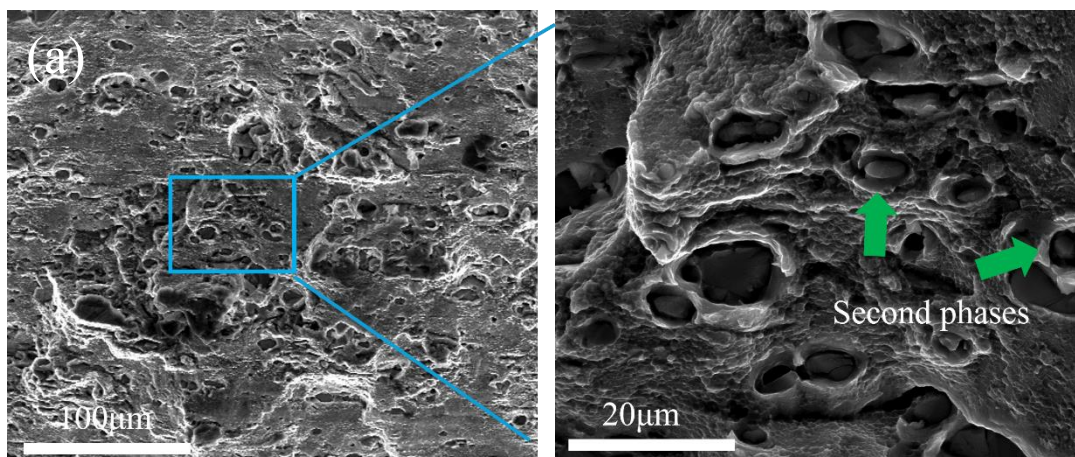


Figure 4-55 Fracture surfaces of LCu after (a) 475 °C 9 h (b) 490 °C 3 h (c) 475 °C 9 h + 475 °C 3 h solution treatment with peak aging.



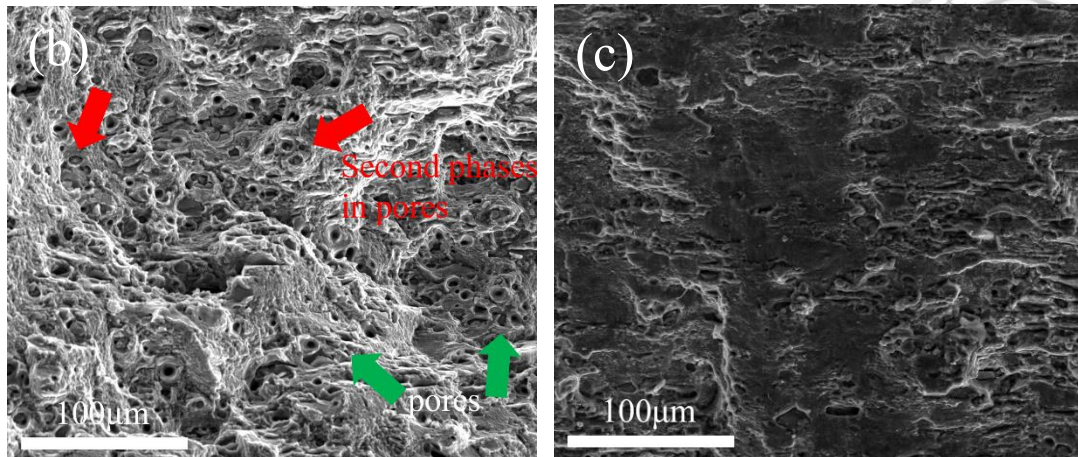


Figure 4-56 Fracture surfaces of HCu after (a) 475 °C 3 h (b) 490 °C 3 h (c) 475 °C 3 h + 490 °C 3 h solution treatment with peak aging.

4.5 EBSD

4.5.1 LCu

EBSD analysis provides crucial insights into the effect of heat treatment on the grain structure of LCu aluminum alloys, specifically focusing on the effective circular diameter of grains under different heat treatment conditions. Figure 4-57 to Figure 4-60 provide EBSD scanning of LCu. In LCu, after a 3-hour solution treatment at 490 °C, the effective grain circle diameter was approximately 61μm. Following a solution treatment of 9 hours at 475 °C and then 3 hours at 490 °C, the grain size was approximately 68μm. After a 12-hour solution treatment at 490 °C, the grain size was about 58μm. From these results, it is evident that different solution treatment stages do not significantly change the grain size. In both single-stage and two-stage solution treatments, the grain size remains within the standard deviation, indicating that in this experiment, grain size is not a variable factor and thus, grain refinement does not affect the mechanical properties. However, upon extending the solution treatment duration to 24 hours, a noticeable growth in grain size was observed, indicating that there were no significant changes in grain size before 12 hours of solution treatment but changing after 12 hours. As the solution treatment time

continued to extend, aluminum atoms accumulated at the grain boundaries, causing the grains to grow outward. The formation of large grains subsequently led to a decrease in strength, consistent with the tensile test results. Table 4-29 shows the grain size of LCu mentioned aboved.

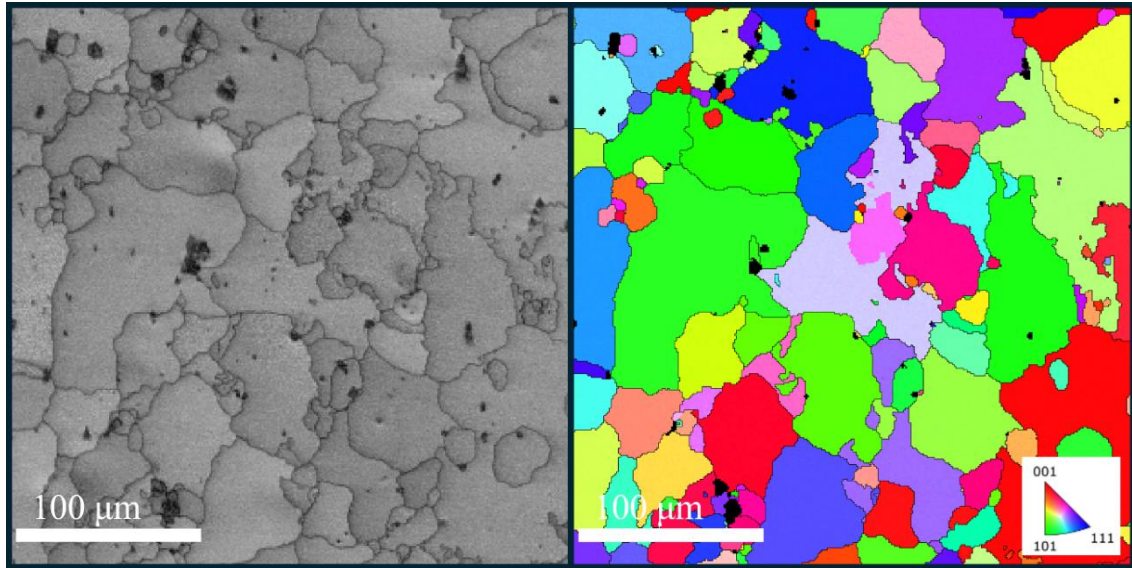


Figure 4-57EBSD analysis of LCu 490 °C 3 h after solution treatment with peak aging.

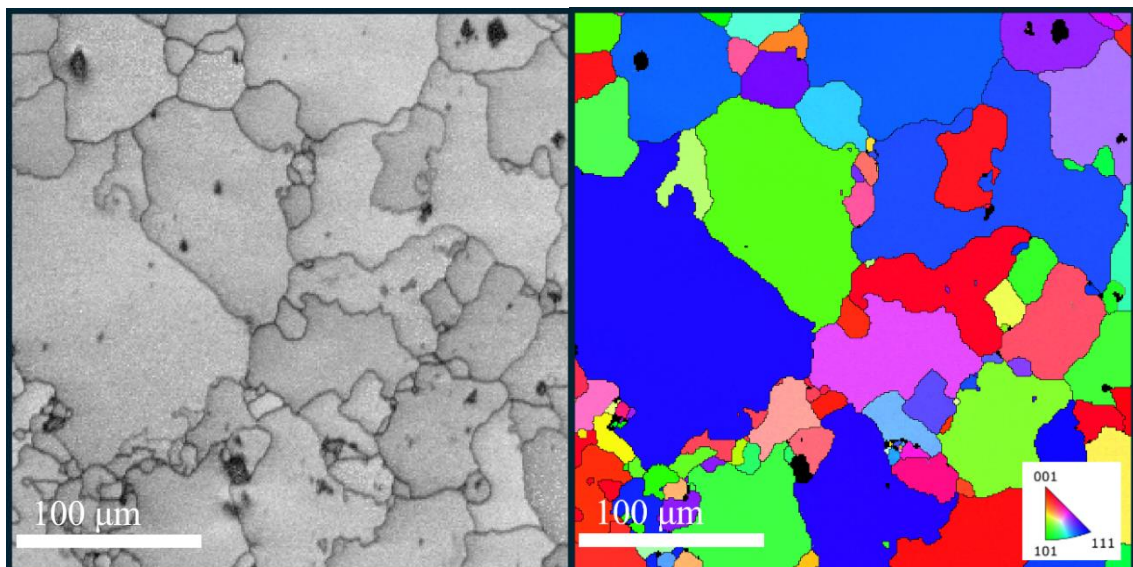


Figure 4-58EBSD analysis of LCu 475 °C 9 h + 490 °C 3 h after solution treatment with peak aging.

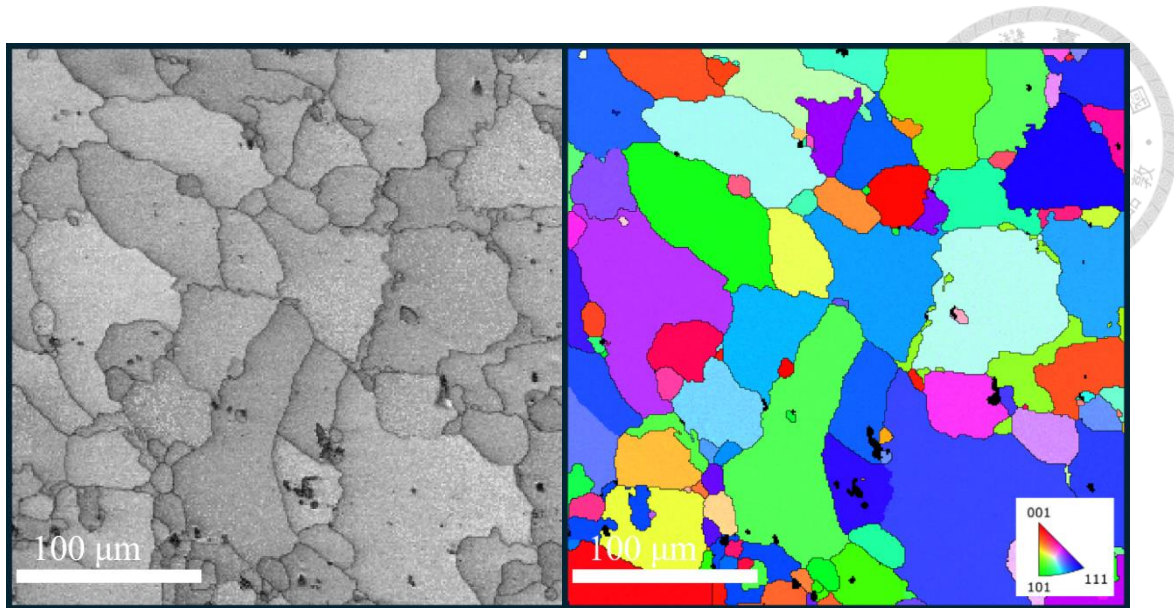


Figure 4-59 EBSD analysis of LCu 490 °C 12 h after solution treatment with peak aging.

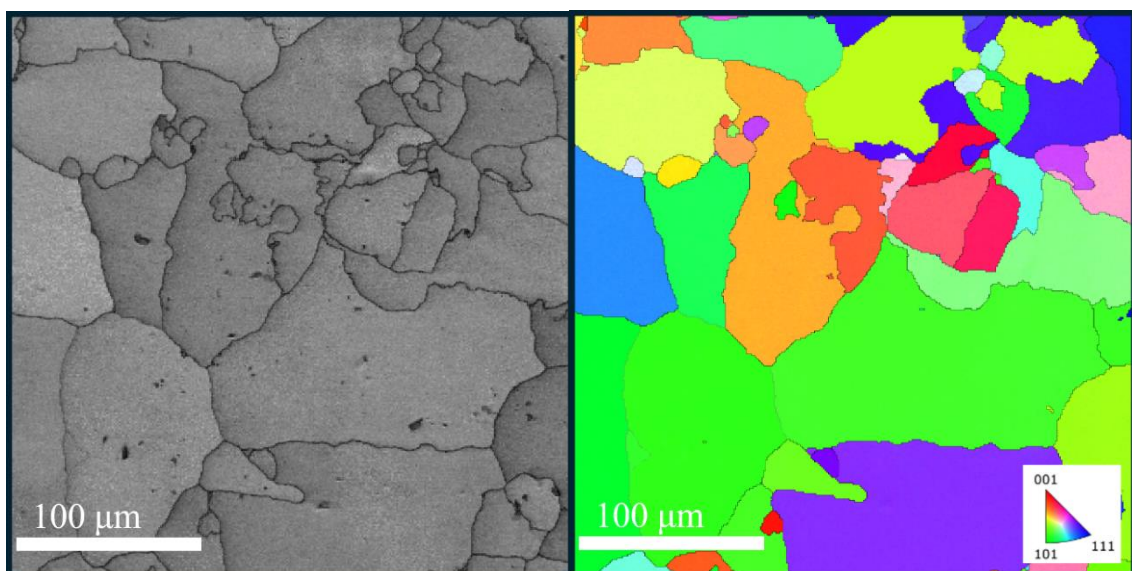


Figure 4-60 EBSD analysis of LCu 490 °C 24 h after solution treatment with peak aging.

Table 4-28 Average grain size of LCu summarized from EBSD.

St. + NA + AA	490 °C 3 h	475 °C 9 h + 490 °C 3 h	490 °C 12 h	490 °C 24 h
Average grain size	64 ± 22 μm	68 ± 23 μm	58 ± 20 μm	96 ± 28 μm

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24 h.

4.5.2 HCu

Figure 4-61 to Figure 4-63 represent the EBSD scanning of HCu. In the EBSD results for HCu, the trend of grain size was similar to that of LCu. In the single-stage solution treatment at 490 °C for 3 hours, the average grain size was 63 μ m. In the two-stage solution treatment of 475 °C for 3 hours followed by 490 °C for 3 hours, the average grain size was 56 μ m. Extending the second stage solution treatment time to 6 hours resulted in an average grain size of 55 μ m. Table 4-29 shows the grain size of HCu. The average grain sizes are close and fall within the standard deviation, indicating that short-term solution treatment does not significantly change the grain size, similar to LCu. Thus, grain refinement does not play a role in the changes in mechanical properties.

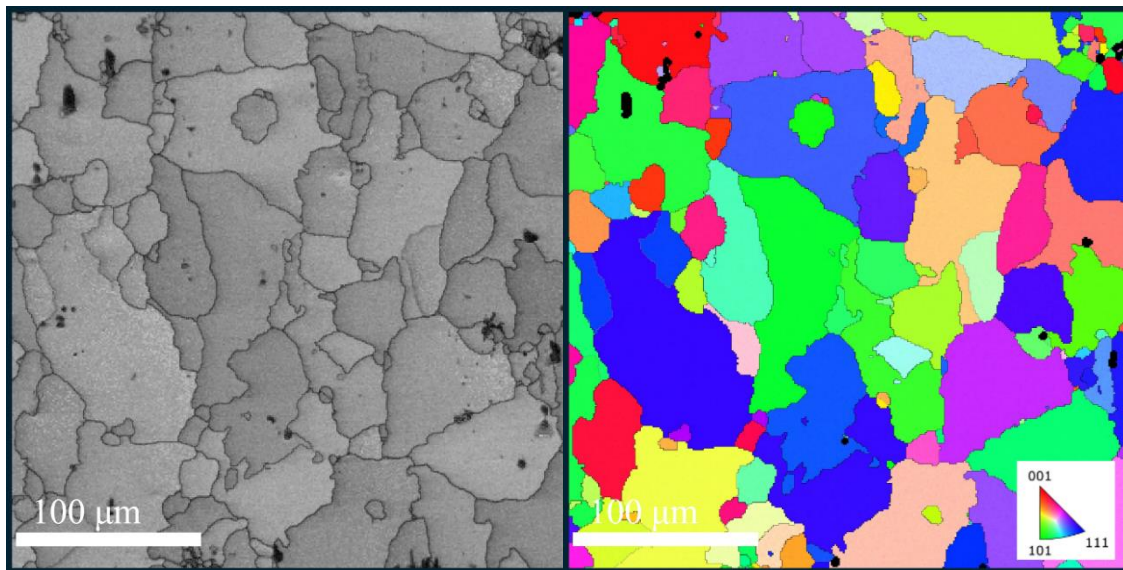


Figure 4-61 EBSD analysis of HCu 490 °C 3 h after solution treatment with peak aging.

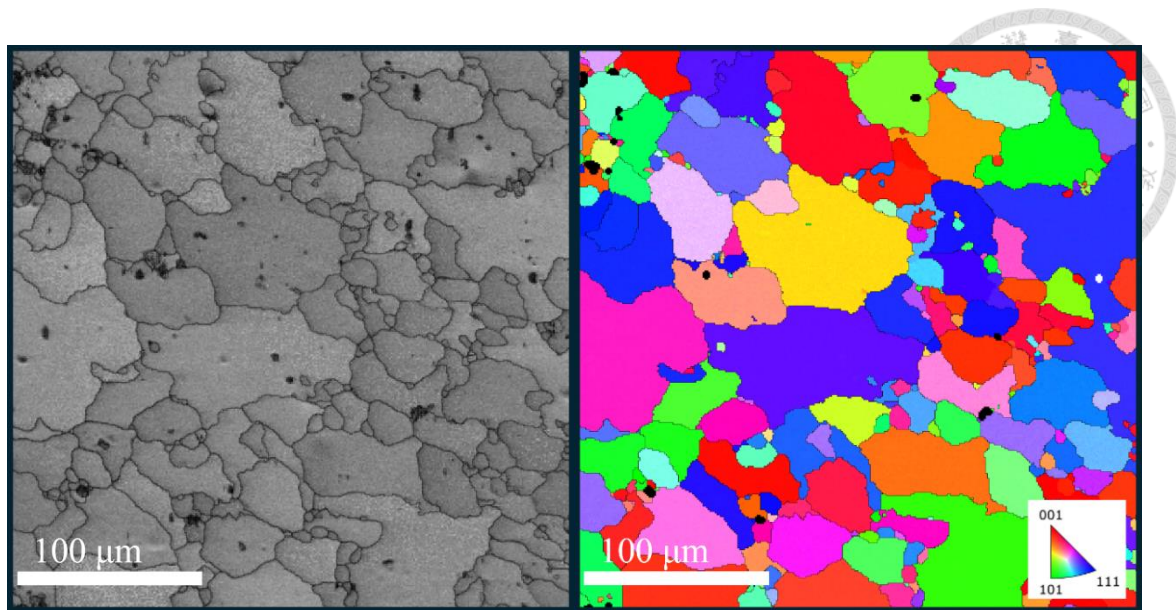


Figure 4-62 EBSD analysis of HCu 490 °C 6 h after solution treatment with peak aging.

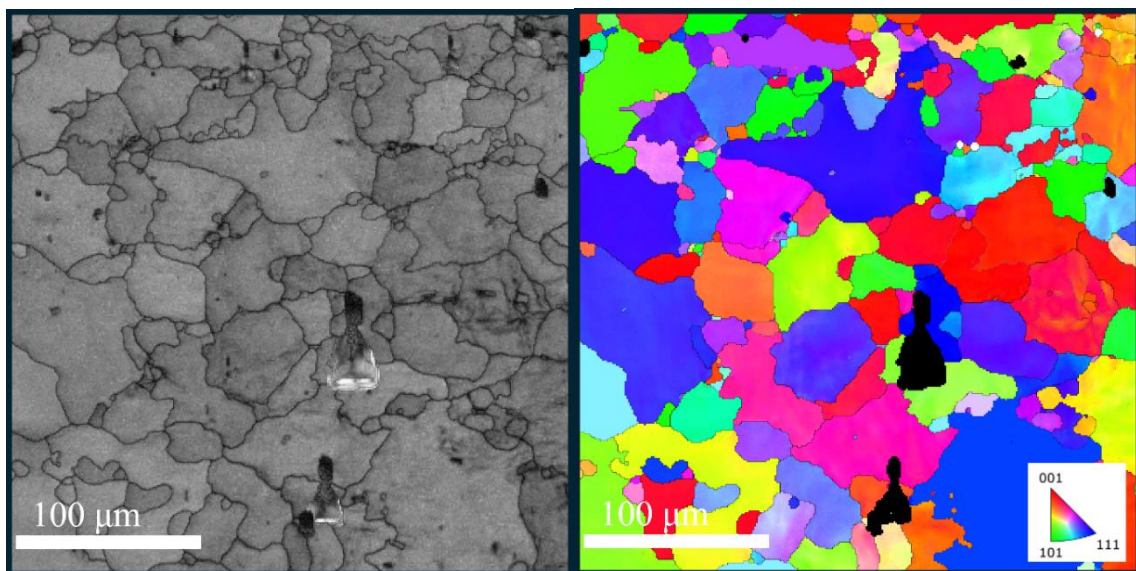


Figure 4-63 EBSD analysis of HCu 475 °C 3 h + 490 °C 3 h after solution treatment with peak aging.

Table 4-29 Average grain size of HCu summarized from EBSD.

St. + NA + AA	490 °C 3 h	475 °C 3 h + 490 °C 3 h	490 °C 6 h
Average grain size	63 ± 21 μm	56 ± 18 μm	55 ± 17 μm

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24 h.

4.6 TEM

4.6.1 LCu after 490 °C 3 h Solution Treatment

Under bright field (BF) observation through the $[110]_{Al}$ axis in TEM, it can be seen that after aging heat treatment, nanoscale precipitates are dispersed within the aluminum alloy, serving as the reason of precipitation hardening. Detailed investigation into different solution treatment processes reveals that the precipitates formed after 3 hours of solution treatment at 490 °C consist of both edge-on and non-edge-on varieties according to statistical results.

In this experiment, ImageJ software was utilized to preprocess bright-field images by removing the background and blurring the images. Afterward, a threshold was set to count precipitates with an area greater than 2 nm² and a circularity greater than 0.7, in order to eliminate noise and edge-on precipitates. The software then statistically analyzed the area of the precipitates. The size of the precipitates was calculated as equivalent diameters by projecting the angle between the $[110]$ direction and the (111) plane, according to the software's statistical data.

Figure 4-64 shows the bright field of single-stage of LCu with peak aging. Based on bright-field imaging statistics, under the condition of 490 °C solution heat treatment for 3 hours, the average diameter of precipitates is about 4.408 nm, with at least several hundred precipitates counted.



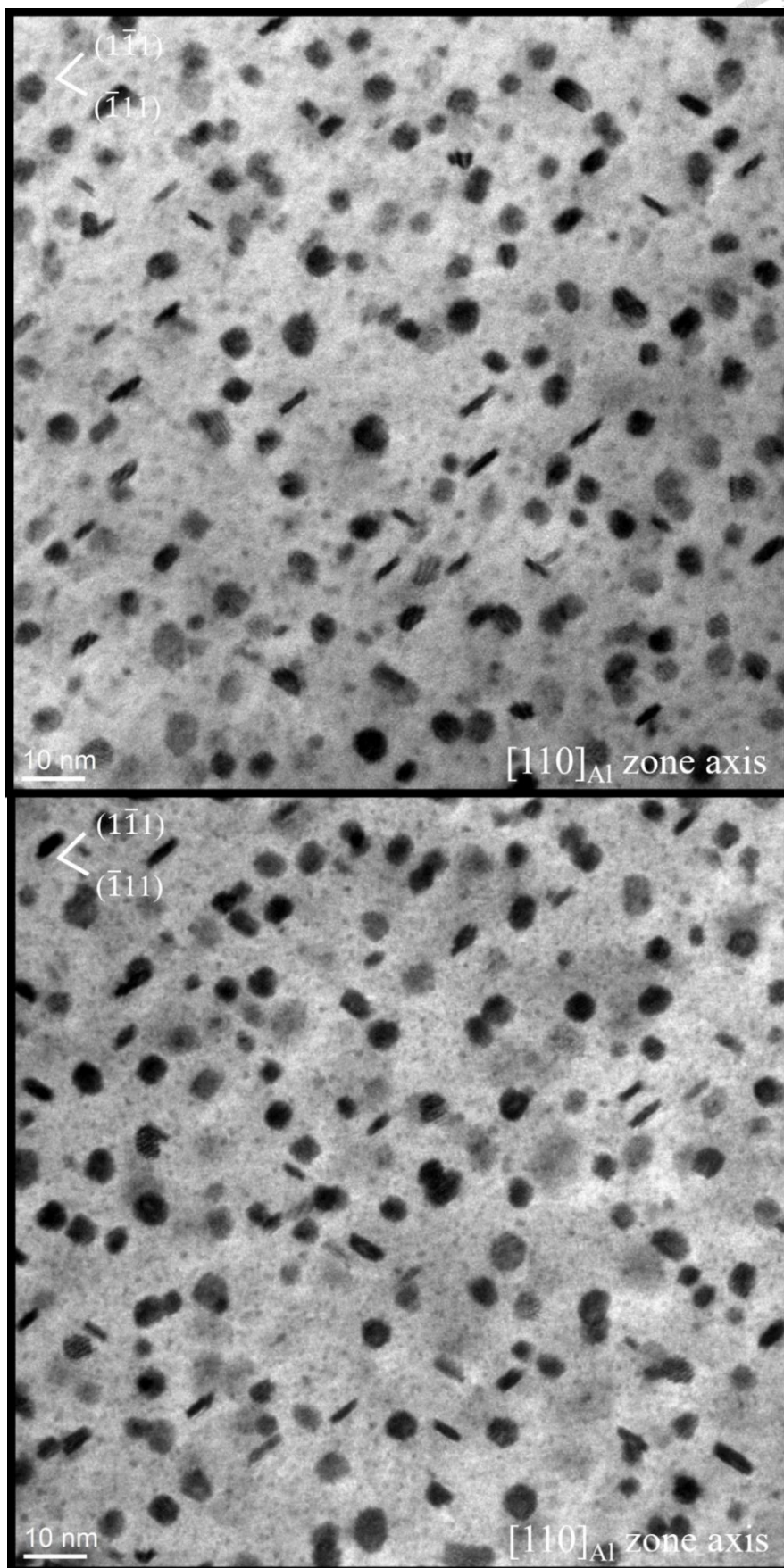


Figure 4-64 TEM-BF images of LCu after 490 °C 3 h Solution Treatment with peak aging.

Figure 4-65 to Figure 4-67 show the HR image single-stage of LCu with peak aging. Based on the HR images, which are formed by the interference of electron beams passing through atoms, the areas with clear contrast indicate the locations of precipitates. Using Fast Fourier Transform (FFT), the types of precipitates can be identified based on their diffraction patterns.

In the condition of solution treatment at 490 °C for 3 hours, both η' and η precipitates are observed. According to FFT analysis, most of the precipitates are identified as η_2 , with the remainder being η_1 , η_3 , and η_4 .

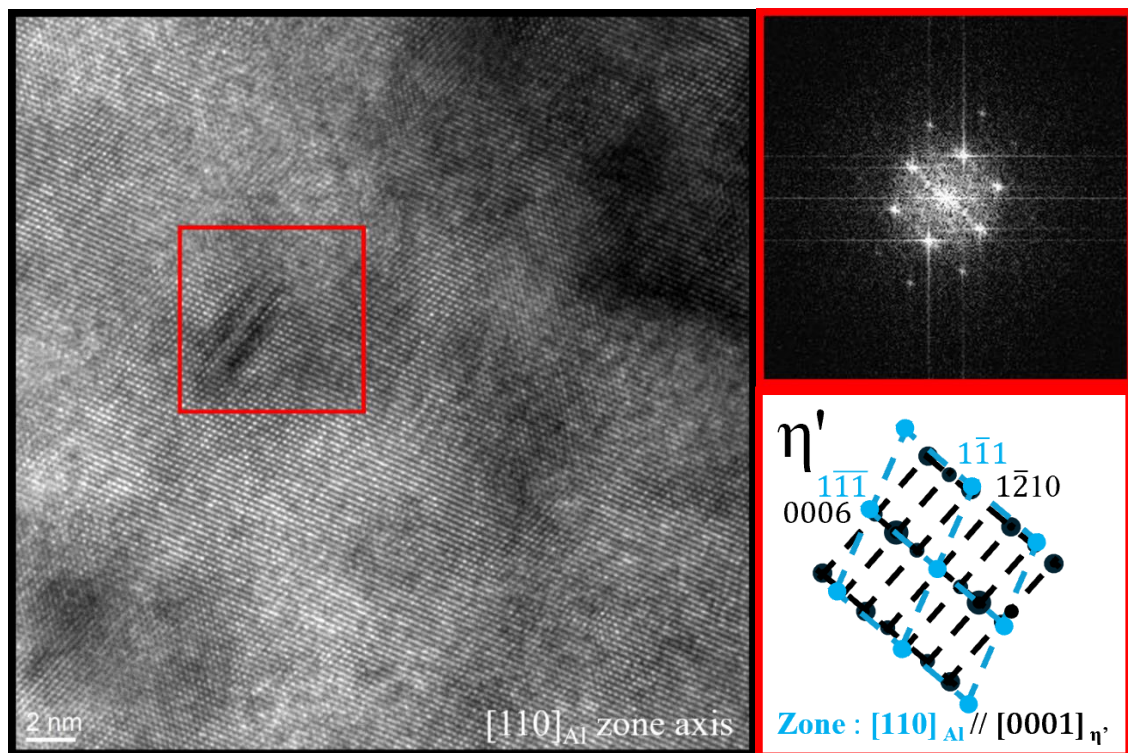


Figure 4-65 HRTEM image of η' precipitate in 490 °C 3 h Solution Treatment with peak aging.

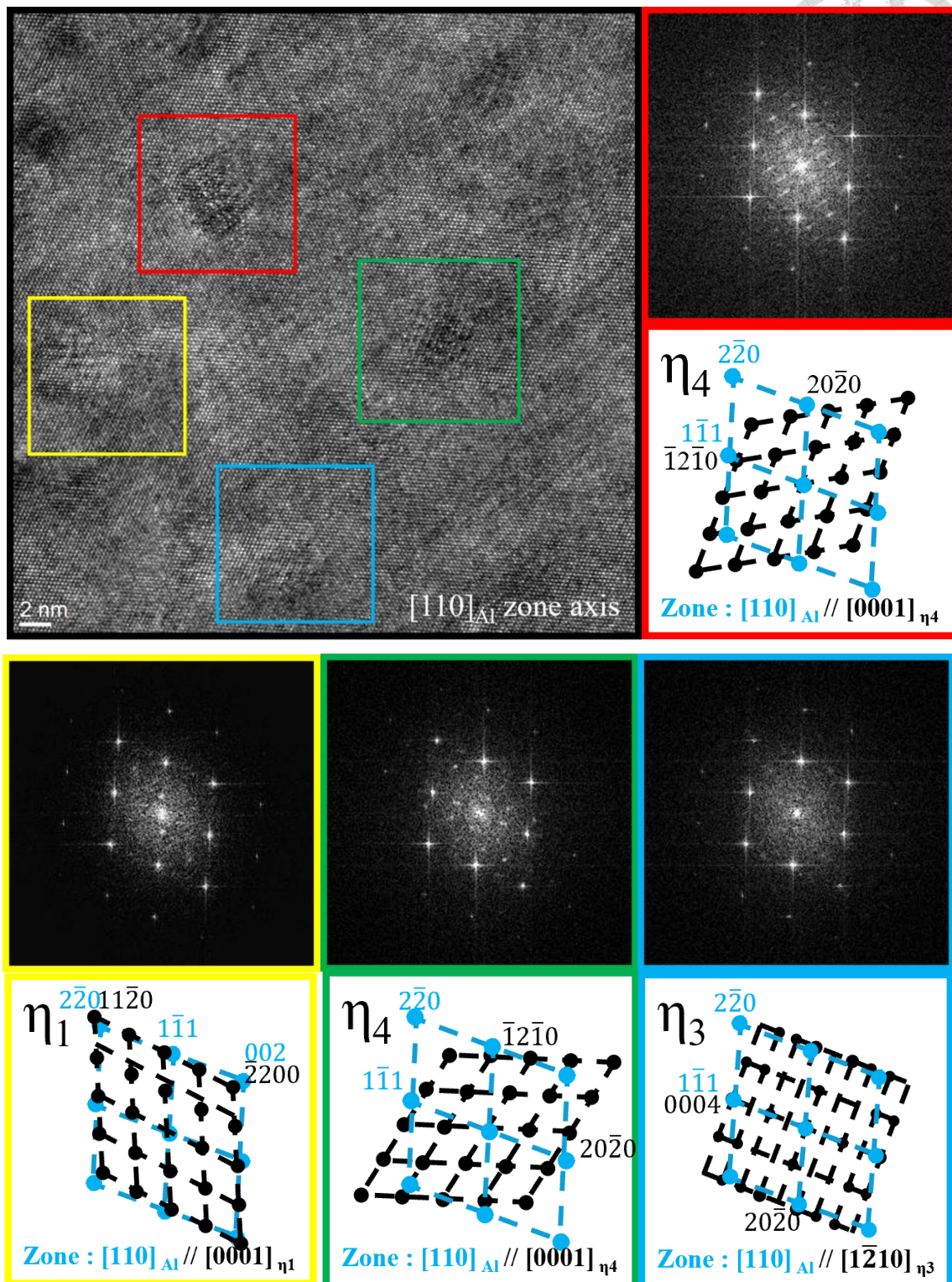


Figure 4-66 HRTEM image of η precipitate in 490 °C 3 h Solution Treatment with peak aging.

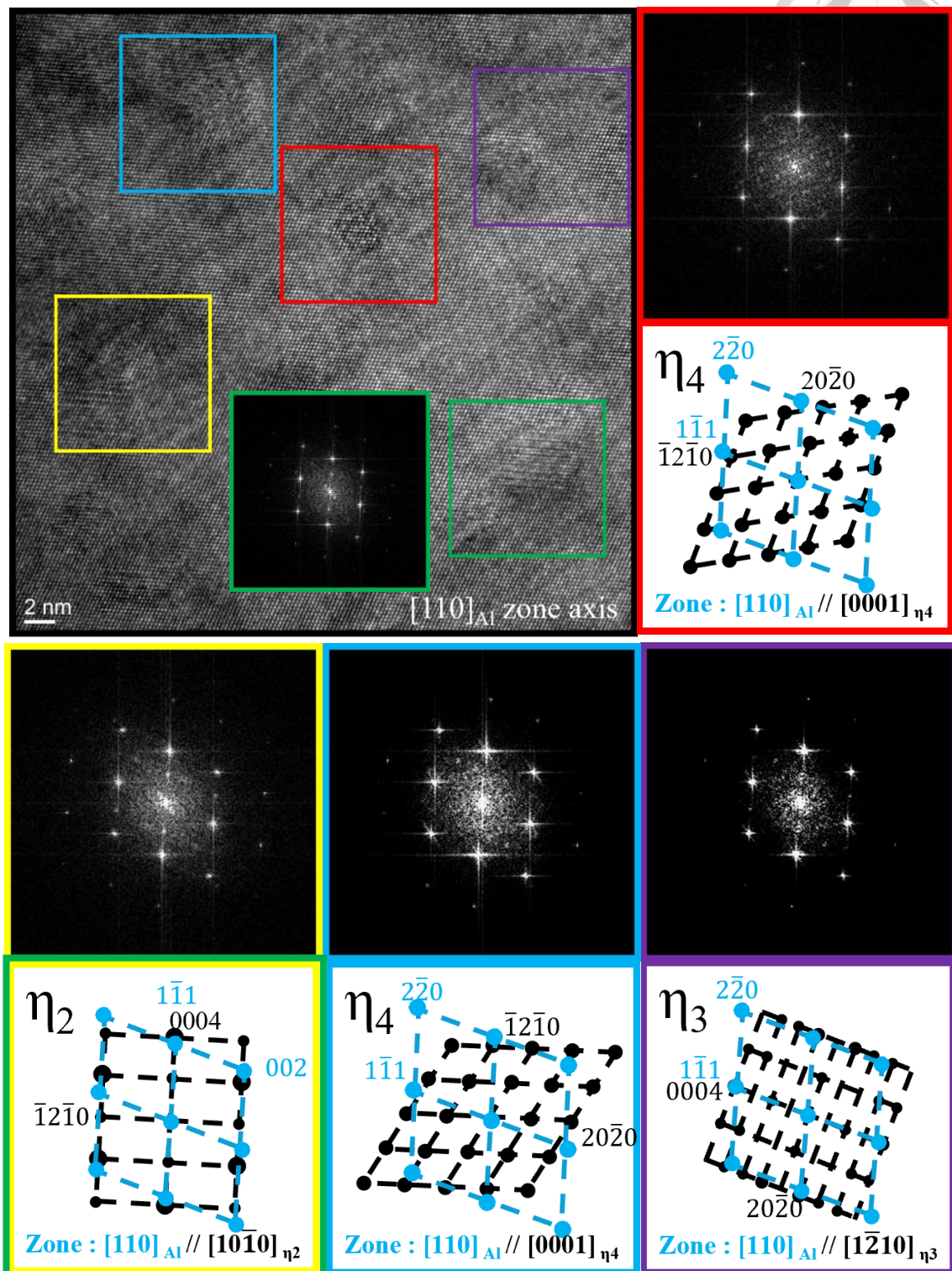
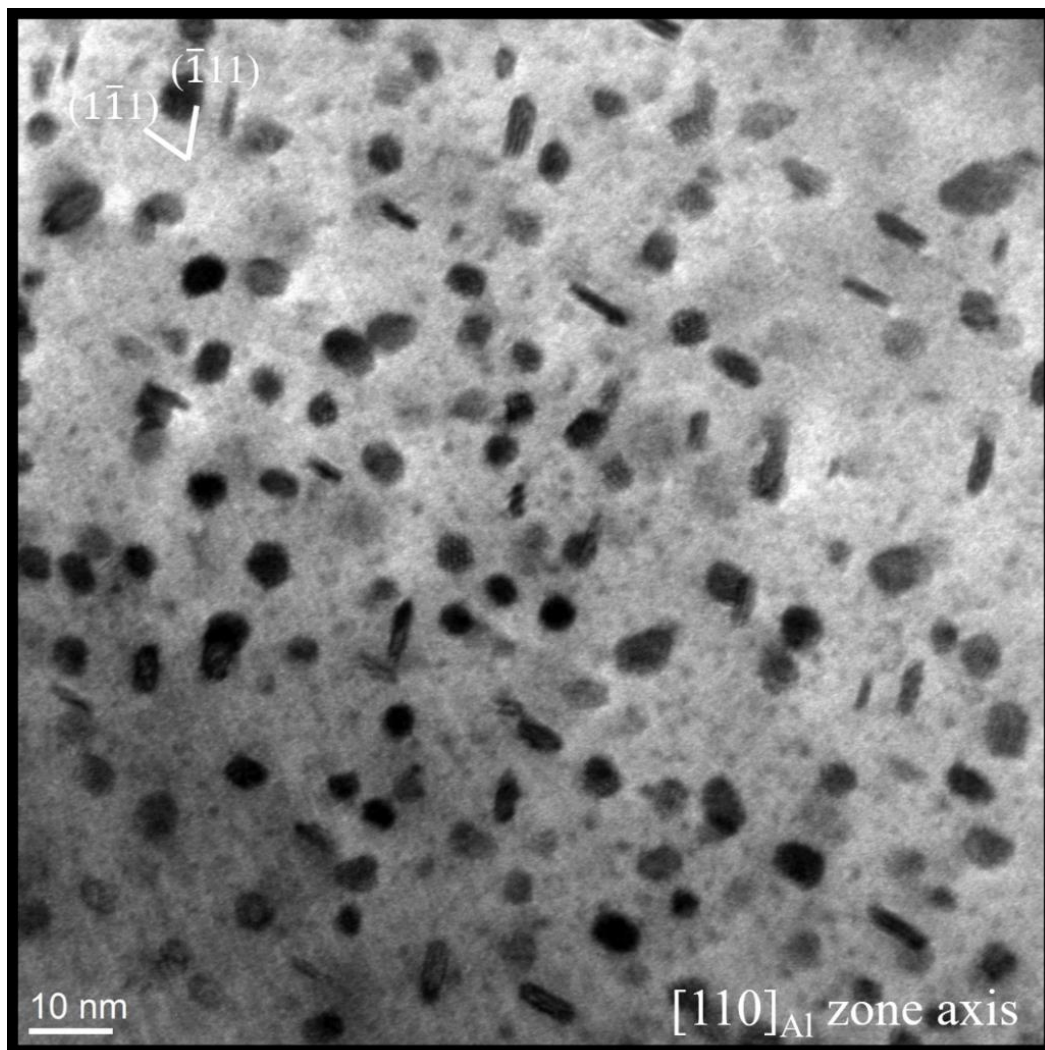


Figure 4-67 HRTEM image of η' precipitate in 490 °C 3 h Solution Treatment with peak aging.

4.6.2 LCu after 475 °C 9 h + 490 °C 3 h Solution Treatment

Figure 4-68 shows the bright field of two-stage of LCu with peak aging. Under bright-field imaging of two-stage solution heat-treated samples, precipitates were still observable. According to software analysis, the average size of these precipitates was approximately 4.376 nm, similar to the size of precipitates found in single-stage solution-treated samples, as detailed in the Table 4-30 below.



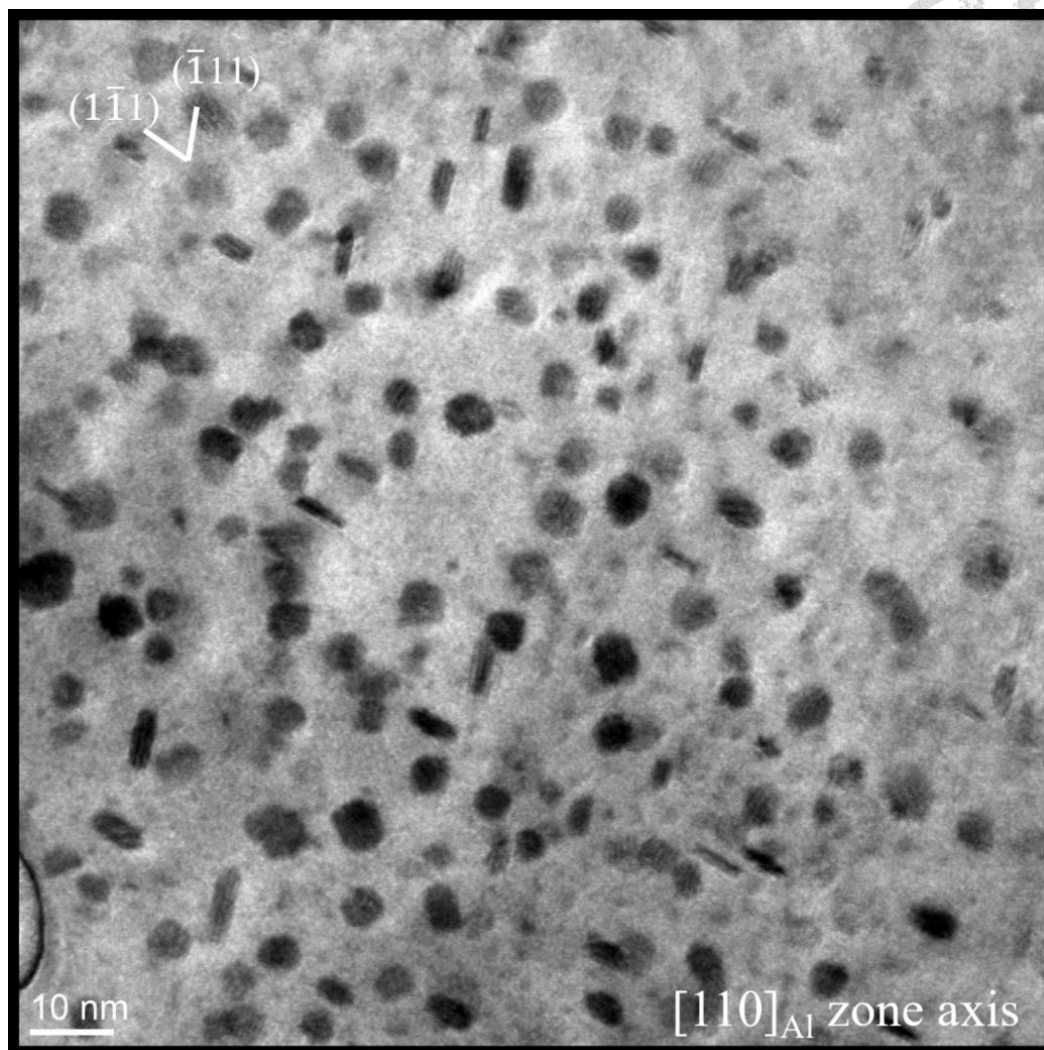


Figure 4-68 TEM-BF images of LCu after 475 °C 9 h + 490 °C 3 h solution treatment with peak aging.

Table 4-30 Analyzed result of precipitates in LCu of single-stage and two-stage solution treatment with peak aging.

St. + NA + AA	490 °C 3 h	475 °C 9 h + 490 °C 3 h
Average precipitate diameter	4.408 ± 1.425 nm	4.376 ± 1.409 nm

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24 h.

Figure 4-69 and Figure 4-70 show the HR image two-stage of LCu with peak aging. Under the two-stage solution treatment condition of 475 °C for 9 hours followed by 490 °C for 3 hours, the analysis of precipitates via HR images and FFT reveals that the main types of precipitates are η' and η_2 , with others being η_1 , η_3 , and η_4 . The variety of precipitates is similar to that found in the single-stage solution treatment process.

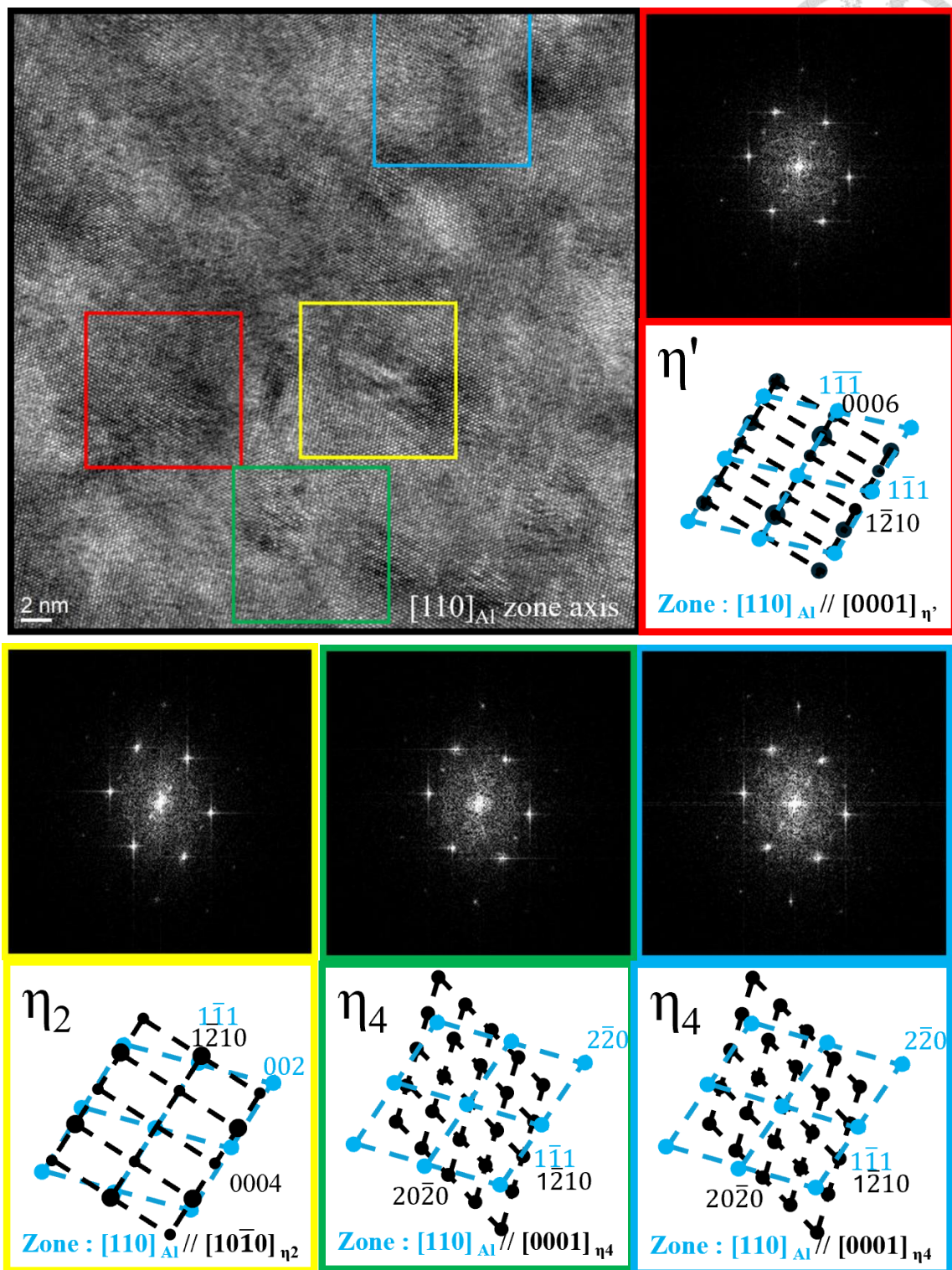


Figure 4-69 HRTEM image of η' and η precipitate after 475 °C 9 h + 490 °C 3 h Solution Treatment with peak aging.

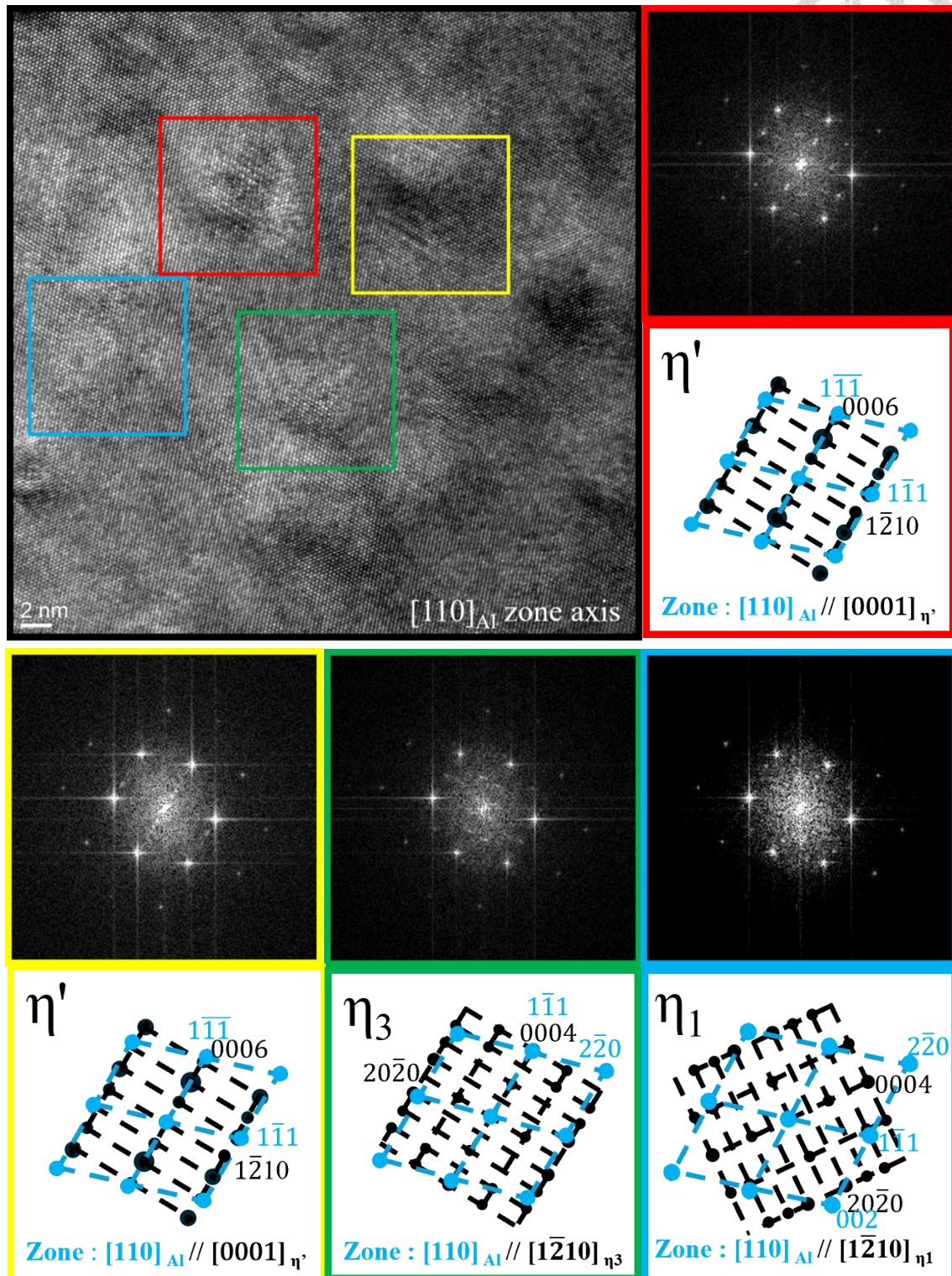


Figure 4-70 HRTEM image of η' and η precipitate after 475 °C 9 h + 490 °C 3 h Solution Treatment with peak aging.

In summary, there are no significant differences in the size, morphology, and distribution of precipitates among the various solution treatment processes.

4.7 Discussion

Based on the research results summarized in Figure 4-71, it is clear that for LCu material, the original material's mechanical properties were poor, with low strength and elongation, primarily due to the presence of a large amount of second phases detectable by DSC and XRD. SEM observations revealed the primary second phases in the original material as the η phase, the S phase, and the ω phase. The presence of these un-dissolved second phases is considered the main reason for the poor mechanical properties without solution treatment.

After a 475 °C solution treatment for 9 hours, there was a significant improvement in mechanical properties. DSC detection showed that the η phase had dissolved back into the base, and no η phase particles were detected in SEM, suggesting that at 475 °C conditions, the η phase could be eliminated, increasing the amount of precipitating elements and improving mechanical properties. However, the DSC still showed signals for the S phase, and SEM, EDX, and WDS detected aluminum, magnesium, and copper signals, indicating that the S phase remained after the 475 °C treatment, contributing to poorer mechanical properties. ω phase particles were also detected in SEM. EPMA detected magnesium and silicon signals in holes, suggesting that the 475 °C treatment does not cause incipient melting.

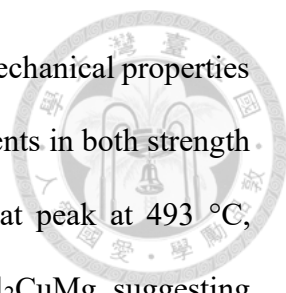
The 490 °C solution treatment for 3 hours resulted in no detection signals for the η and S phases in DSC, and no particles of these phases were detected in SEM, only iron-based signals were detected in EDX and WDS, likely the ω phase. Thus, compared to the 475 °C for 9 hours treatment, there was a further improvement in mechanical properties, both in strength and ductility. However, EPMA showed many holes, some of which had elemental signals identical to the base, indicating holes caused by incipient melting,

suggesting that while mechanical properties improved, there was room for further enhancement.

The two-stage solution treatment of 475 °C for 9 hours followed by 490 °C for 3 hours showed no signals for η and S phases in DSC, and none were detected in SEM, similar to the 490 °C for 3 hours treatment, only detecting iron-based second phases confirmed as ω phase by WDS. After the 490 °C treatment, η and S phases could be fully dissolved, improving mechanical properties. The most significant difference between single-stage and two-stage solution treatments was the smoother surface of the samples from two-stage solution treatment, with fewer holes detected in SEI and BSE. EPMA scans showed that the majority of holes contained magnesium and silicon signals, indicating they were caused by polishing rather than incipient melting, leading to a significant improvement in mechanical properties, especially elongation.

In HCu, a large amount of second phases detectable by XRD were present in the original material, with DSC signals indicating a significant heat release at the melting point of the S phase, stronger than that of LCu. SEM detected η , S, and ω phases, with a notably higher proportion of S phase compared to LCu, hence the lower elongation of HCu in the original material.

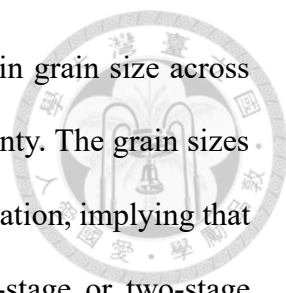
After HCu underwent a 475 °C solution treatment for 3 hours, there was a significant improvement in mechanical properties, likely due to the complete dissolution of the η phase back into the base, as indicated by the absence of η phase signals in DSC and SEM. However, compared to LCu treated only at 475 °C for the first stage, HCu had poorer mechanical properties, possibly due to a higher copper content in HCu, resulting in a higher proportion of the S phase. EPMA evidence confirmed that most second phases were Al_2CuMg , indicating that although mechanical properties improved, there was still room for improvement due to the excessive remaining S phase.



After HCu underwent a 490 °C solution treatment for 3 hours, mechanical properties improved further compared to just the 475 °C stage, with improvements in both strength and elongation. However, DSC scanning revealed a significant heat peak at 493 °C, indicating residual S phase, and EPMA confirmed the presence of Al₂CuMg, suggesting that a 490 °C treatment for 3 hours was insufficient to fully dissolve the S phase in HCu, which might also contribute to poor mechanical properties. Therefore, if the S phase can be completely dissolved, the strength and elongation rate may be further improved.

The two-stage solution treatment of 475 °C for 3 hours followed by 490 °C for 3 hours in HCu led to significant improvements in mechanical properties, including elongation. DSC scans showed no second phase signals, and EPMA confirmed the absence of η and S phases, indicating they had fully dissolved back into the material. The surface was smoother, with fewer holes than the 490 °C for 3 hours treatment, suggesting the two-stage solution treatment effectively addressed issues related to incipient melting observed in single-stage treatments, and being able to completely dissolve the S phase back into the matrix is a reason for the improvement in mechanical properties.

On the fracture surface, the material exhibits direct fracture morphology, and comprehensive observation on the SEI can integrate the results previously observed on SEM. In the condition of only the first-stage solution treatment, second phases can be seen on the fracture surface, which are residues from the solution treatment and are related to poor material properties. On the fracture surface of only the second-stage solution treatment, holes can be observed, possibly related to incipient melting, which is also a reason for poor mechanical properties of the material. In HCu, residual second phases can also be seen, unlike in LCu, consistent with previous experimental results. Finally, under two-stage solution treatment conditions, fewer holes and no observed second phases are present.



The EBSD analysis reveals that there is no significant change in grain size across the different heat treatment regimes within the experimental uncertainty. The grain sizes across all lower than 12 hours conditions fall within the standard deviation, implying that grain size is not a variable significantly affected by either single-stage or two-stage solution heat treatments under the specific conditions tested in this experiment. Consequently, the mechanical properties observed to improve or change across these heat treatment conditions are not attributed to grain refinement or significant changes in grain size but likely result from alterations in the dissolution of second phases, the distribution of alloying elements, and the avoidance of incipient melting defects.

Analyzing the precipitates in bright-field images, it was found that there are no significant differences in the size of the precipitates between single-stage and two-stage solution heat treatments. The average diameter of the precipitates during peak aging was approximately 4 nm. Therefore, the change in mechanical properties is likely not related to the precipitation strengthening mechanism. Furthermore, HR imaging analysis indicated that the types of precipitates are similar in both single-stage and two-stage solution heat treatments, suggesting that the enhancement in strength and ductility is more closely associated with the second phases.

The comprehensive experimental results show that both L_{Cu} and H_{Cu} improved under the two-stage solution treatment, but the degree of improvement was greater in L_{Cu} than in H_{Cu}. The reason is that the increased copper content forms S phase, which cannot be fully dissolved at 490 °C. The added 475 °C stage mainly targets the η phase. Thus, the mechanical properties of L_{Cu} improve more than those of H_{Cu}. It can be concluded that increasing the copper content in aluminum alloys to significantly produce S phase leads to a decline in mechanical properties. Although two-stage solution treatment can slightly enhance the properties diminished by the excessive addition of copper, generally,

the mechanical properties of high-copper aluminum alloys remain inferior to those of low-copper aluminum alloys. Therefore, if only considering the two-stage solution treatment, LCu material is superior to HCu.

Overall, the research demonstrated that two-stage solution treatments can significantly improve the mechanical properties of aluminum alloys by dissolving critical second phases and minimizing defects such as incipient melting, leading to optimal mechanical performance, and the details are illustrated below. In this study, both strength and elongation showed significant improvement, especially in elongation, without the need to sacrifice strength to achieve it.

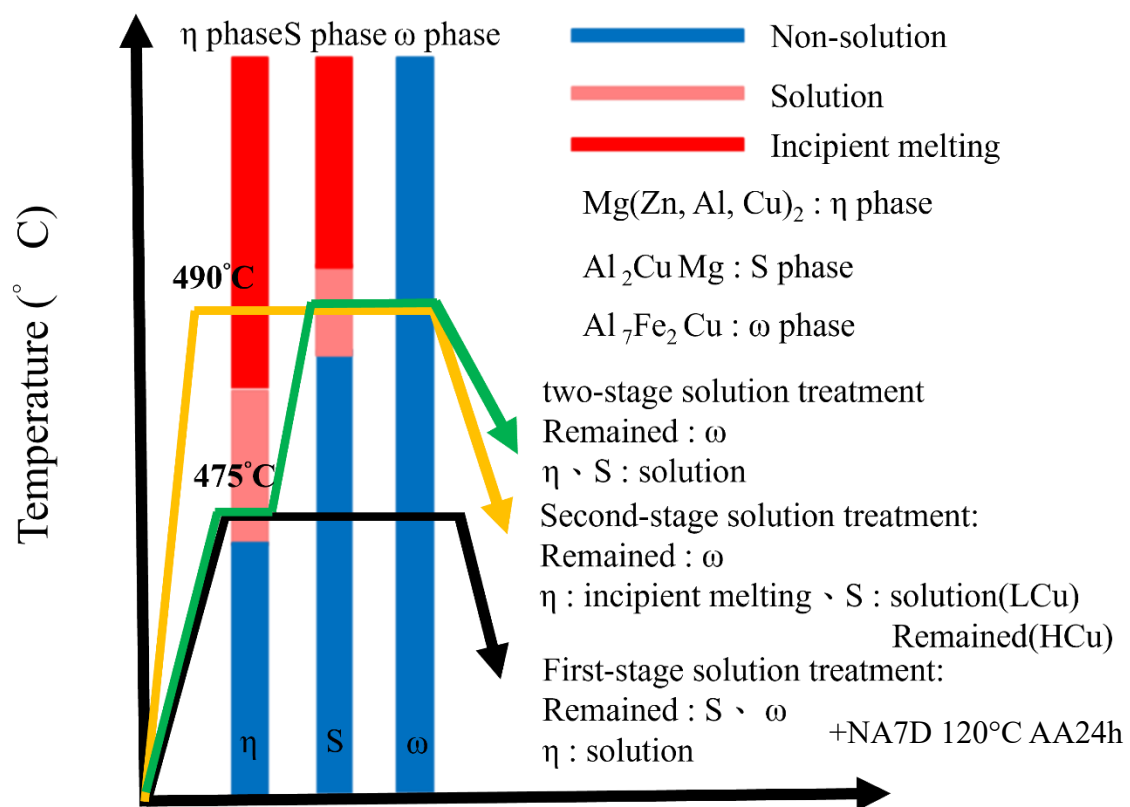


Figure 4-71 Summarize of two-stage solution treatment.

Chapter 5 Three-Stage solid solution treatment



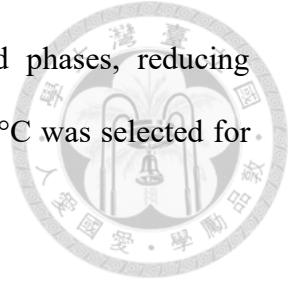
5.1 Introduction

As discussed in Chapter 4, after the two-stage solution treatment, the overall mechanical properties of HCu did not improve completely compared to LCu. Therefore, to enhance HCu, a third stage of solution treatment at 500 °C for one hour was decided upon, and to maintain a fixed total solution time, the duration at 475 °C was reduced by one hour due to the lack of significant benefits from increasing the 475 °C solution time as observed in Chapter 4.

The addition of a 500 °C solution for one hour could offer two potential benefits to improve HCu's mechanical properties. First, it could directly dissolve residual small S phases in the HCu matrix. As there is a higher amount of S phase in the HCu matrix, it's possible that not all S phase was completely dissolved in the previous single-stage and two-stage processes and has become too small to be observed at previous magnifications. Thus, dissolving these small S phases directly could release alloying elements from the precipitates, enhancing mechanical properties with minimal impact from any incipient melting pores formed during the process. Secondly, literature [121] suggests that higher temperature quenching can create more nucleation sites for precipitates, and a greater distribution of precipitates can lead to improved mechanical properties. Therefore, based on these two factors, it is believed that a 500 °C solution treatment could enhance mechanical properties without needing prolonged exposure; reaching thermal equilibrium for a short duration should suffice. Since there are more S phases in HCu, it is projected that the three-stage solution treatment could offer greater benefits for HCu.

The choice of 500 °C is based on literature suggesting that solution temperatures too high, exceeding 515 °C, could lead to the formation of a network-like eutectic structure

between the 7000 series aluminum alloy matrix and the second phases, reducing mechanical properties. Therefore, considering all these factors, 500 °C was selected for the third stage of the solution treatment.



5.2 Tensile test

In the two-stage solution heat treatment, an improvement in mechanical properties was observed compared to the single-stage solution heat treatment. However, the extent of improvement in HCu was significantly less than that in LCu. To further enhance the mechanical properties of HCu, it was decided to add third stage of solution heat treatment at 500 °C for 1 hour. Based on experimental results, the 475 °C stage had minimal effect on HCu; thus, to keep the total solution time constant, the duration at 475 °C was reduced by 1 hour, transitioning to a three-stage solution heat treatment process in Figure 5-1.

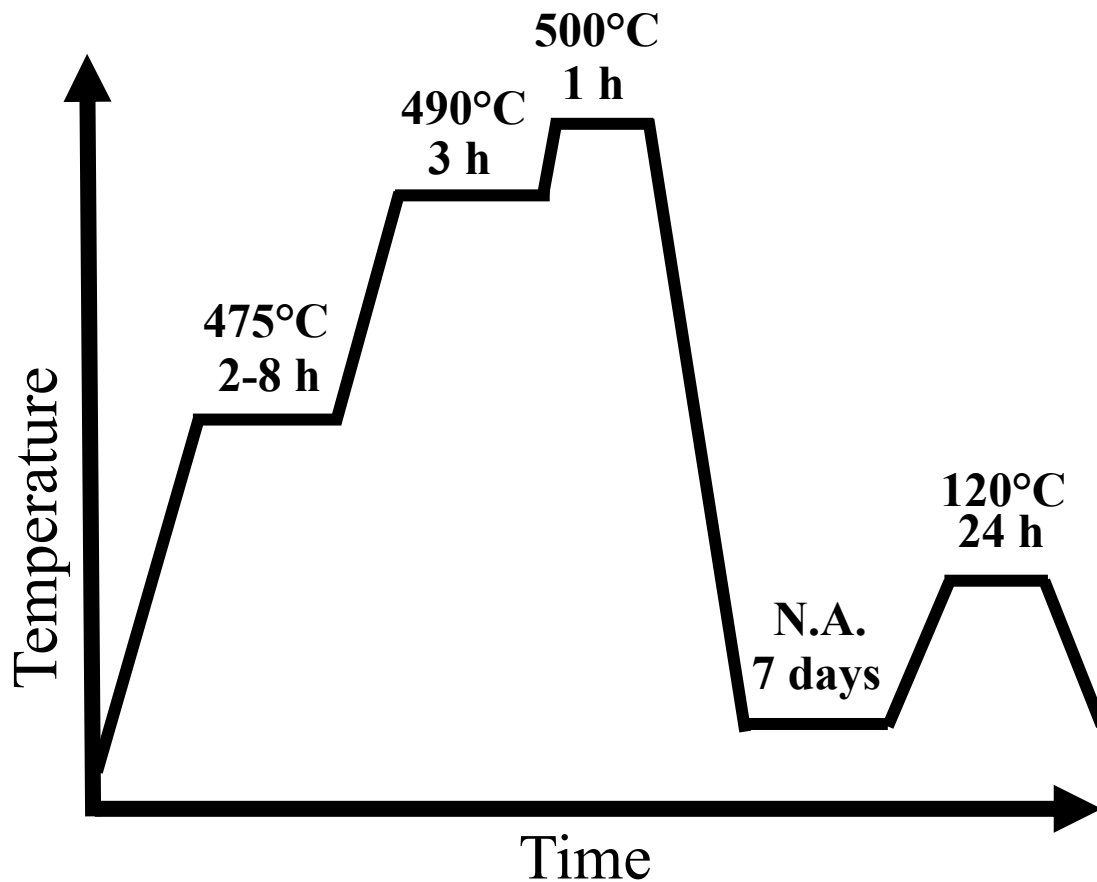


Figure 5-1 Illustration of three-stage solution treatment used for the experiment.

According to literature, quenching from higher temperatures can lead to a higher strength [121]. There may be denser distribution of precipitates, thereby improving mechanical properties. Figure 5-2 and Table 5-1 to Table 5-2 shows the tensile curve and mechanical properties of LCu and HCu. The results demonstrated that the addition of a third solution stage at 500°C significantly enhanced the mechanical properties of HCu, especially in terms of elongation, while the effect was not pronounced in LCu.

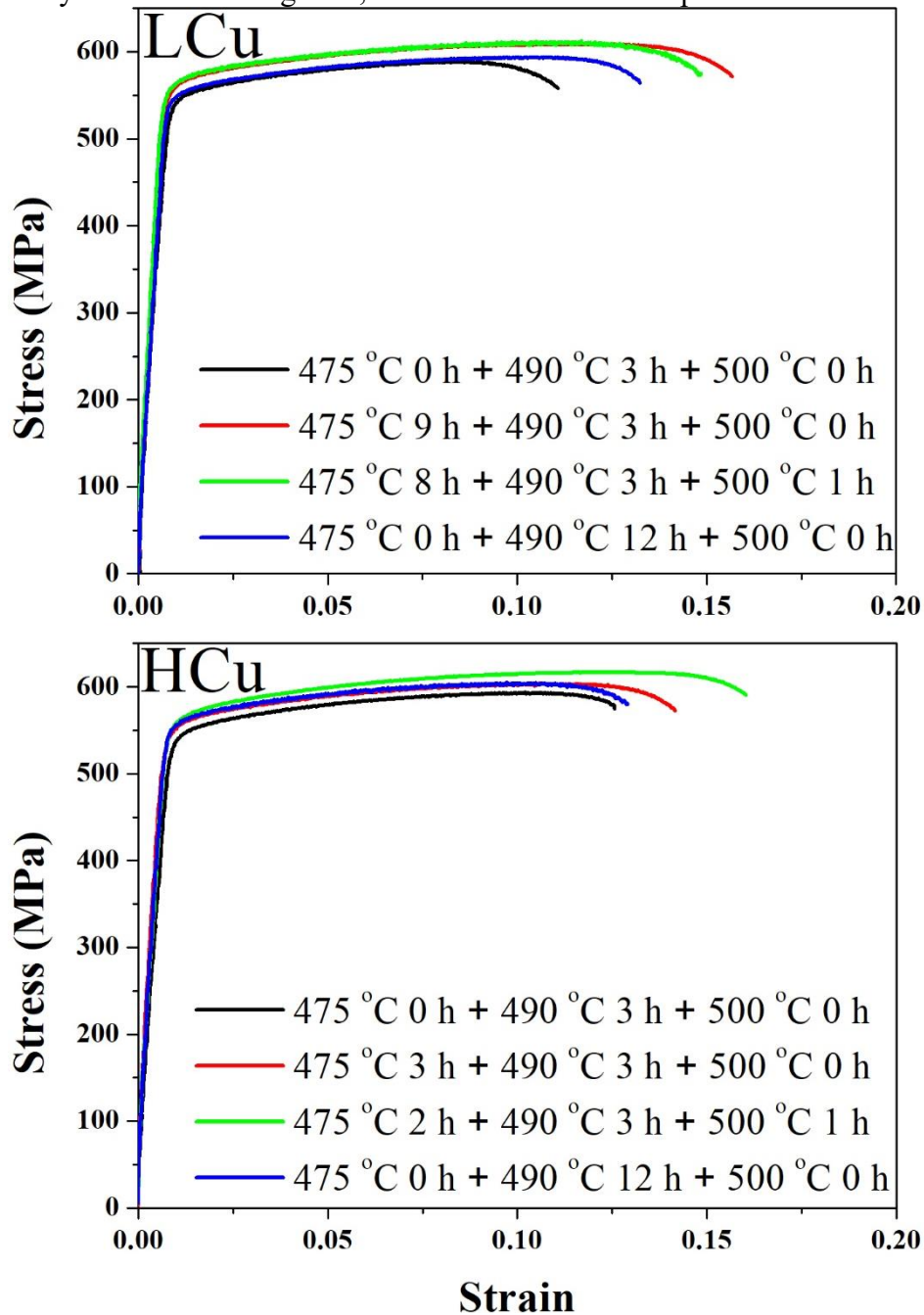


Figure 5-2 Tensile curves of three-stage, two-stage, and single-stage solution treatment for LCu and HCu on the condition peak ageing at 120 °C.

Table 5-1 Mechanical properties of three-stage, two-stage, and single-stage solution treatment for LCu on the condition peak ageing at 120 °C.

LCu	YS(MPa)	UTS(MPa)	EL(%)
St. + NA + AA			
3h/490 °C	548 ± 3.4	597 ± 2.6	11.8 ± 0.42
9h/475°C + 3h/490°C	551 ± 3.6	606 ± 1.4	15.0 ± 0.46
8h/475°C + 3h/490°C+ 1h/500°C	555 ± 1.0	610 ± 1.7	14.7 ± 0.35
12h/490°C	549 ± 1.5	599 ± 0.66	13.5 ± 0.27

Table 5-2 Mechanical properties of three-stage, two-stage, and single-stage solution treatment for HCu on the condition peak ageing at 120 °C.

HCu	YS(MPa)	UTS(MPa)	EL(%)
St. + NA + AA			
3h/490°C	540 ± 1.7	597 ± 2.7	12.6 ± 0.056
3h/475°C + 3h/490°C	543 ± 3.7	603 ± 4.0	14.3 ± 0.95
2h/475°C + 3h/490°C+ 1h/500°C	548 ± 3.5	613 ± 4.0	15.2 ± 0.8
6h/490°C	540 ± 2.2	598 ± 1.5	13.3 ± 0.42

St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 °C 24 h.

Therefore, it is inferred that a three-stage solution heat treatment is effective for aluminum alloys with a high copper content. The detailed mechanisms behind the strengthening process still require further investigation.

5.3 DSC, XRD and EPMA Result

Given the improvements in mechanical properties observed in HCu following the three-stage solution treatment, with significant enhancements in strength and ductility compared to the two-stage solution process, while the changes in LCu compared to the two-stage solution treatment were not significant, future analyses will primarily focus on HCu.

Regarding the second phases, results from Chapter 4 suggest that following the two-stage solution treatment, there are no significant η phases or S phases remaining within the aluminum alloy matrix, indicating the absence of macroscopic second phases affecting mechanical properties.

Experimental findings illustrated in the DSC thermal analysis in Figure 5-3 show no significant endothermic peaks, indicating that no second phases were detected. Additionally, in Figure 5-4, the XRD analysis, only signals from the aluminum base were received, with theoretical peaks for η and S phases indistinguishable from noise. Therefore, it is concluded that there are no significant second phases present in HCu following the three-stage solution treatment. This suggests that the treatment effectively eliminates such phases.

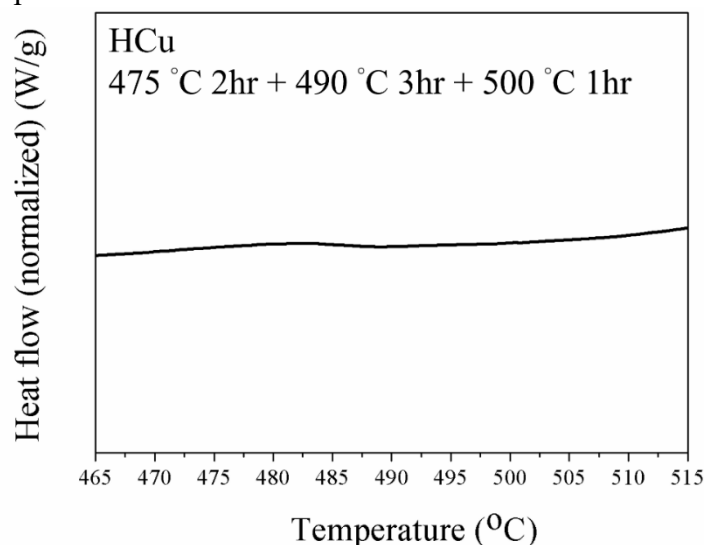


Figure 5-3 DSC curves of three-stage solution treatment on HCu.

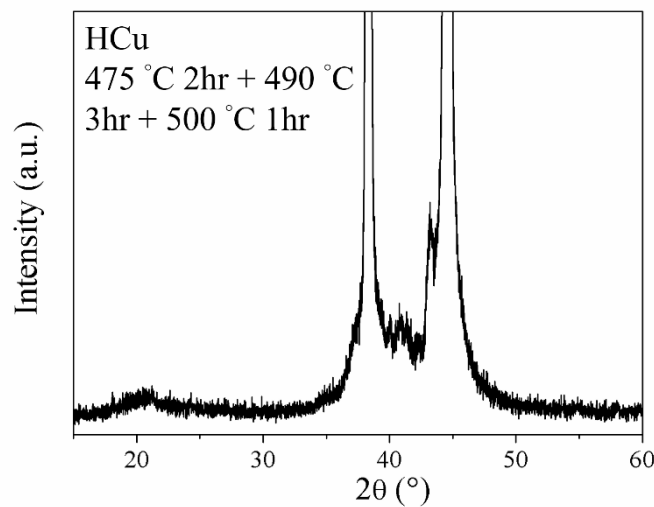


Figure 5- 4 XRD curves of three-stage solution treatment on HCu.

After conducting an EPMA scan shown in Figure 5-5, the surface of the sample three-stage solution treatment in HCu appeared clean and similar to that of the two-stage solution treatment, with a low number of pores. The elemental distribution revealed iron signals in the secondary phases, suggesting the presence of the ω phase, $\text{Al}_7\text{Fe}_2\text{Cu}$. This indicates that the η and S phases likely dissolved back into the matrix, consistent with the findings from the two-stage solution treatment. Further quantitative analysis using WDS in Figure 5-6 confirmed the presence of the ω phase, thereby confirming that the η and S phases are not present in the matrix.

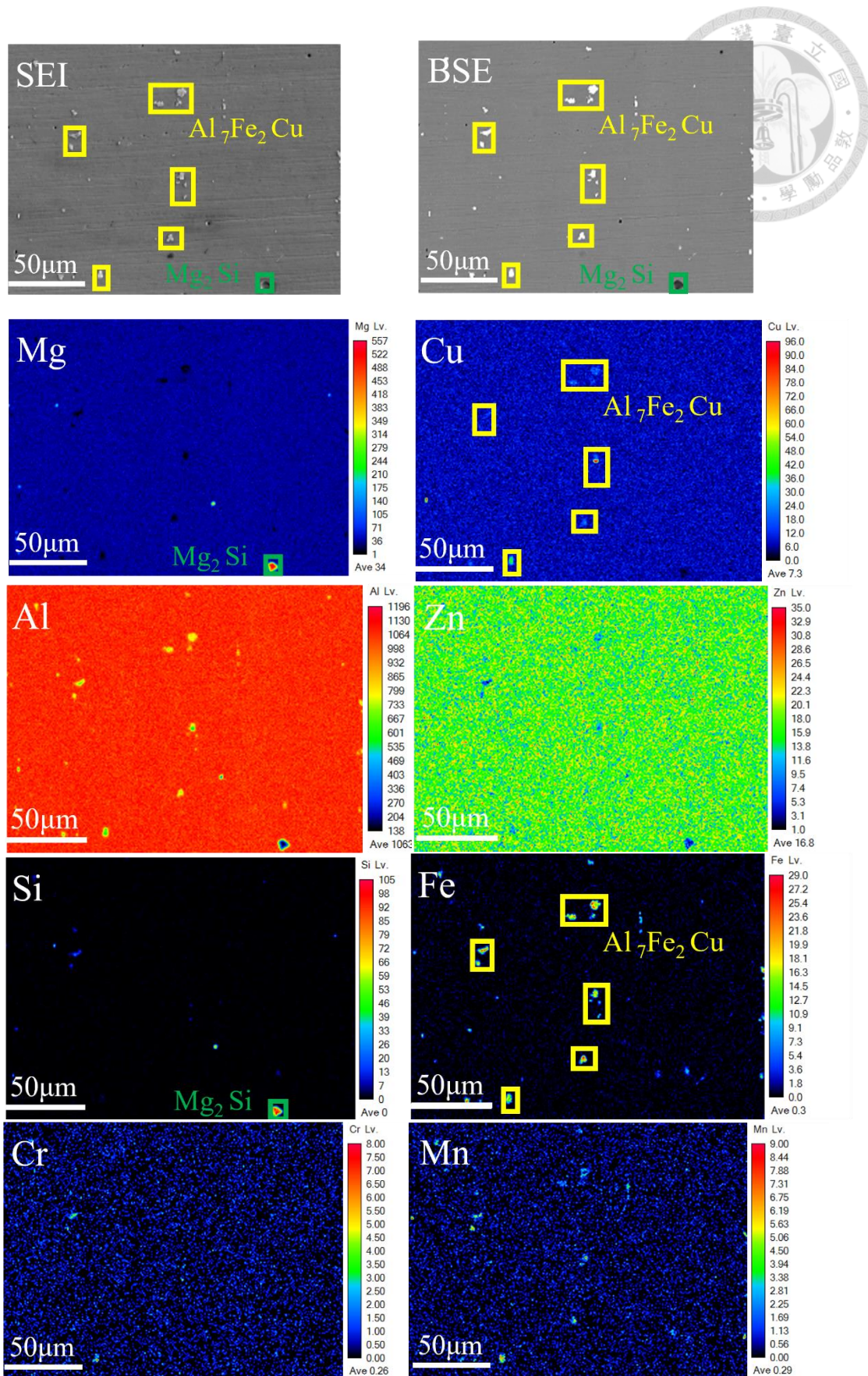
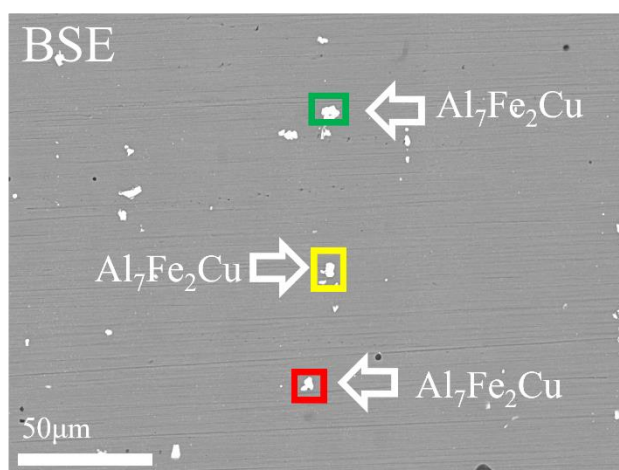
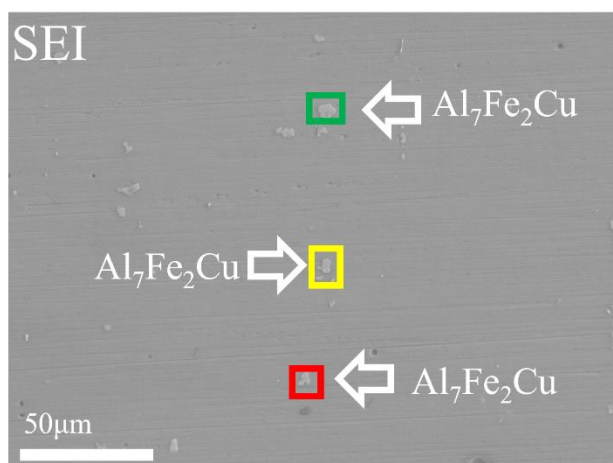


Figure 5-5 EPMA mapping of HCu after 475 °C 2 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging (yellow : ω ; green : Mg_2Si).



ω phase

ω phase

ω phase

WDS	at (%)	WDS	at (%)	WDS	at (%)
Al	81.8	Al	82.2	Al	85.5
Mg	0.18	Mg	0.37	Mg	1.2
Cu	1.3	Cu	1.3	Cu	1.6
Zn	2.9	Zn	2.8	Zn	2.3
Fe	11.0	Fe	11.1	Fe	7.85
Si	0.02	Si	0.03	Si	0.03
Mn	2.55	Mn	2.03	Mn	1.2

Figure 5-6 WDS elemental quantification of HCu after 475 °C 3 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging.

5.4 TEM Bright Field

Figure 5-7 shows the bright field of three-stage of LCu with peak aging. In the TEM bright field, a dense distribution of precipitates can be seen. Statistics show that the average size of precipitates is about 3.64 nm, with a standard deviation of 1.28 nm, based on at least several hundred precipitates analyzed. The calculation reveals that the size of precipitates is slightly lower than the results of previous experiments, but the strength of the material has increased, suggesting that the increase in precipitate density may have contributed to this improvement.

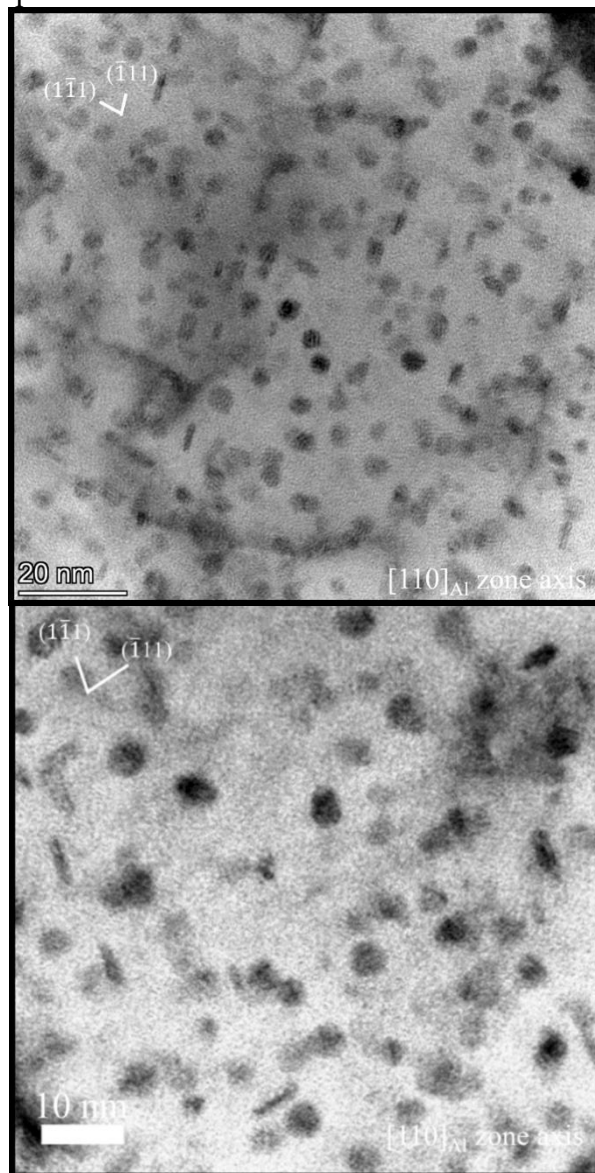
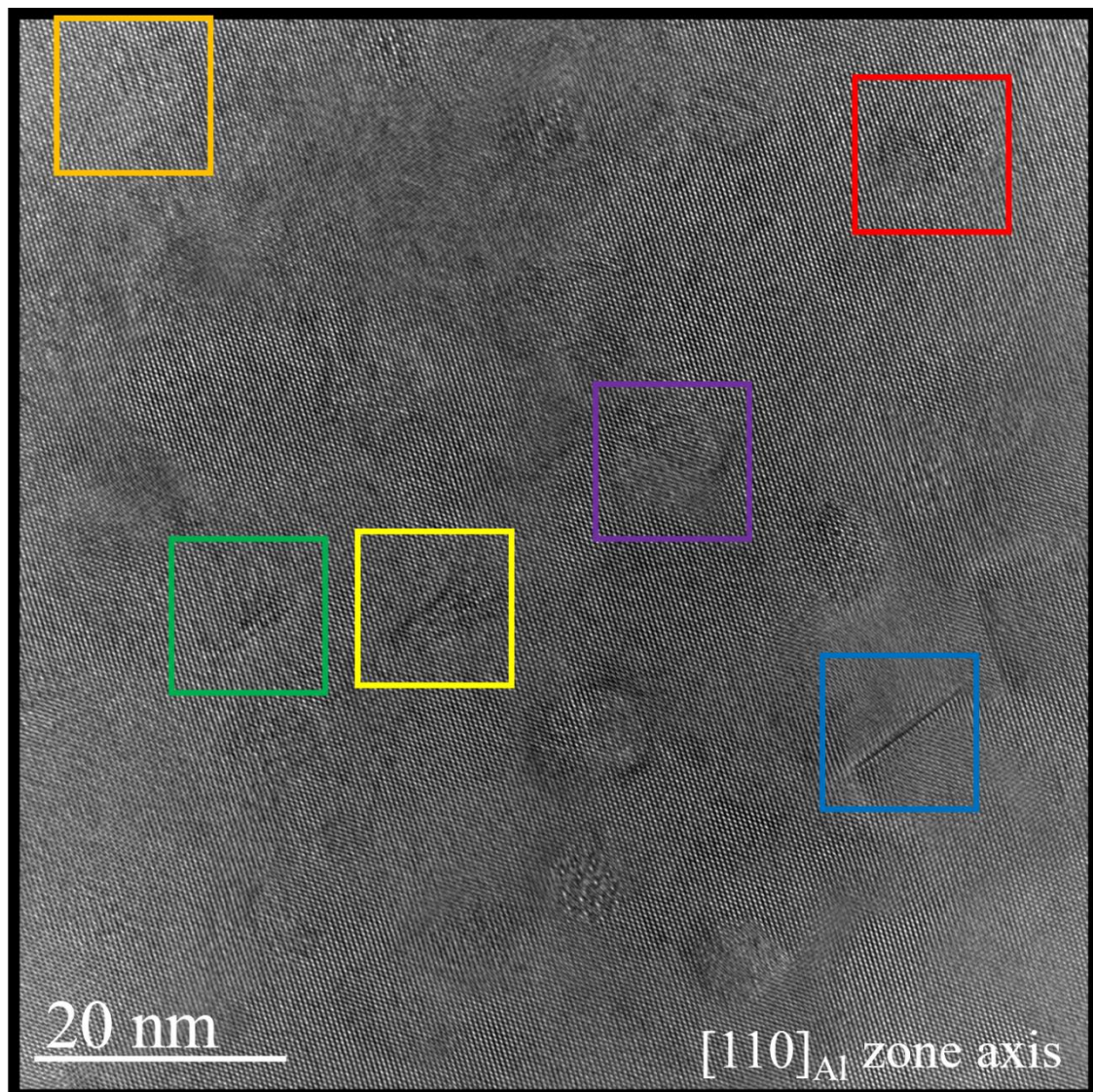


Figure 5-7 TEM-BF images of HCu after 475 °C 2 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging.

5.5 HRTEM image

Figure 5-8 shows the HR image three-stage of LCu with peak aging. Detailed analysis of the HRTEM images identified the types of precipitates present. According to FFT analysis, the main precipitates are η' and η_2 , with the remainder being η_1 and η_3 , and GP Zones were also observed. The types of precipitates are similar to those in the single-stage and two-stage solution treatment processes, which aligns with previous conclusions, suggesting that changing the solution treatment stages has minimal impact on the precipitates. The diameter of precipitates measured in the HR images is approximately less than 4 nm, consistent with the results from bright field statistics.



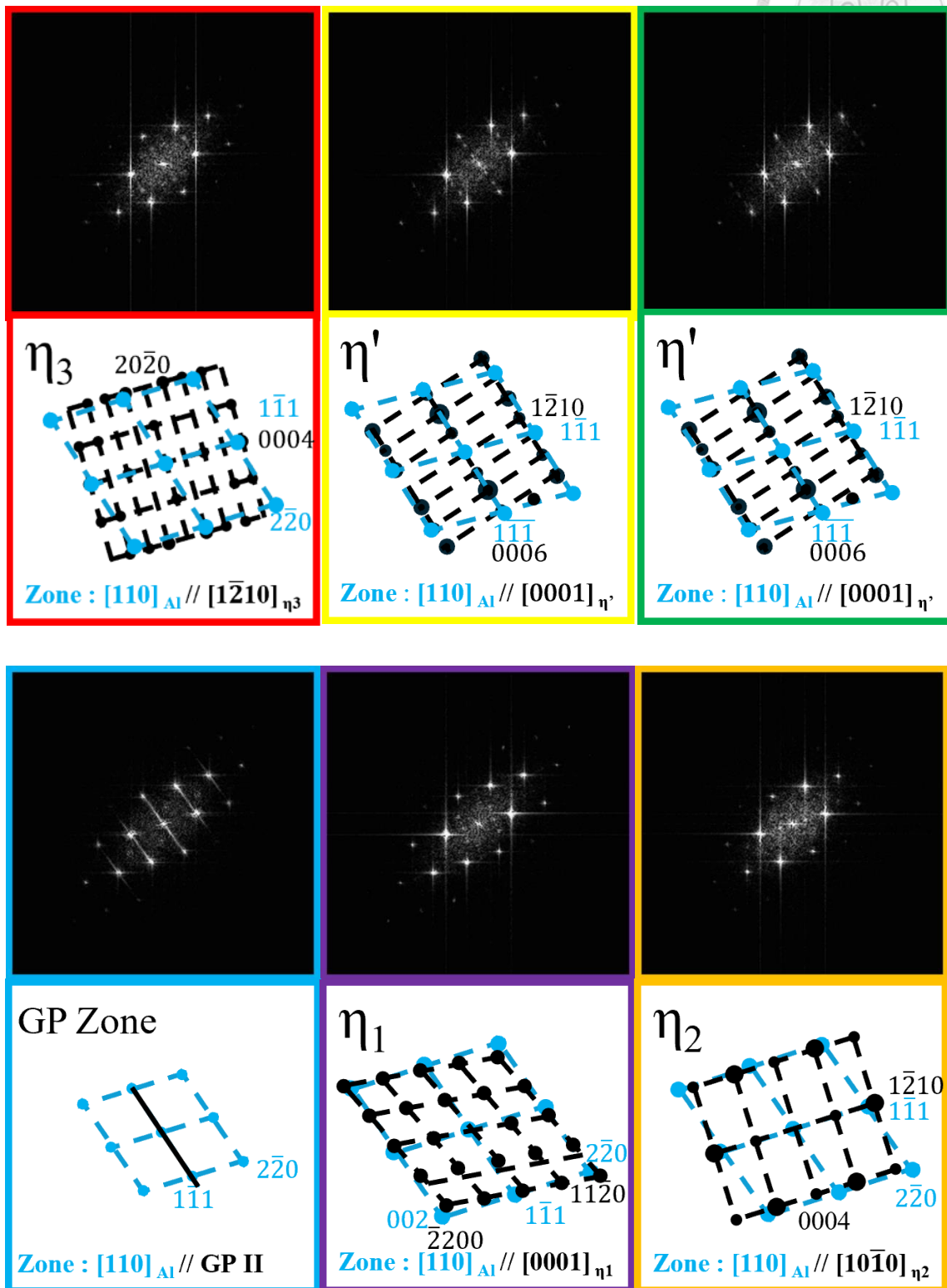
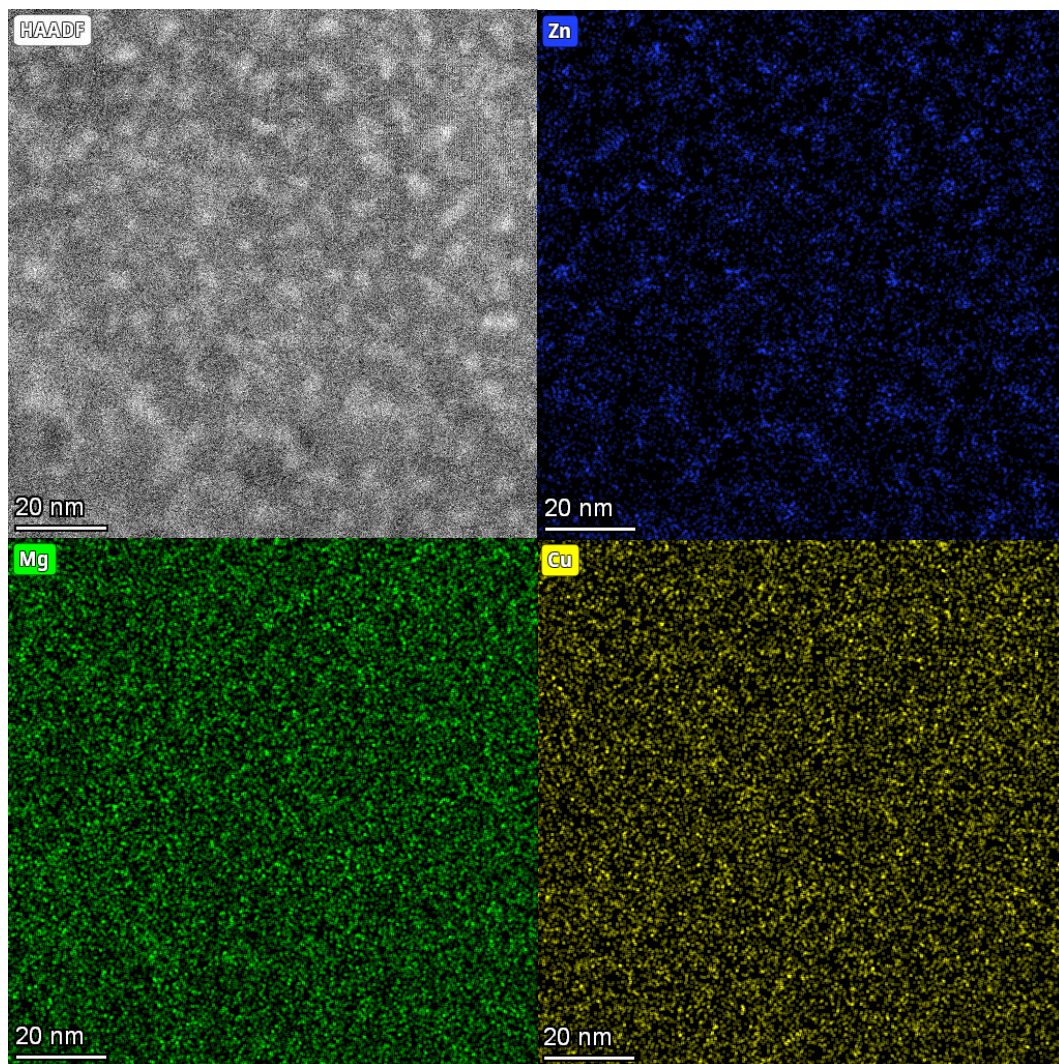


Figure 5-8 HRTEM image of precipitate in HCu after 475 °C 2 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging.

5.6 HAADF and Atomic-Resolution Image Analysis

In HAADF images shown in Figure 5-9, precipitates are visible, and zinc element distribution observed with EDS scanning matches the precipitates. Additionally, the η phase composition is MgZn_2 . Copper element distribution indicates some copper incorporation into the precipitates, differing from previous study results [119] and potentially contributing to the improvement in mechanical properties. The detailed mechanism remains to be clarified.



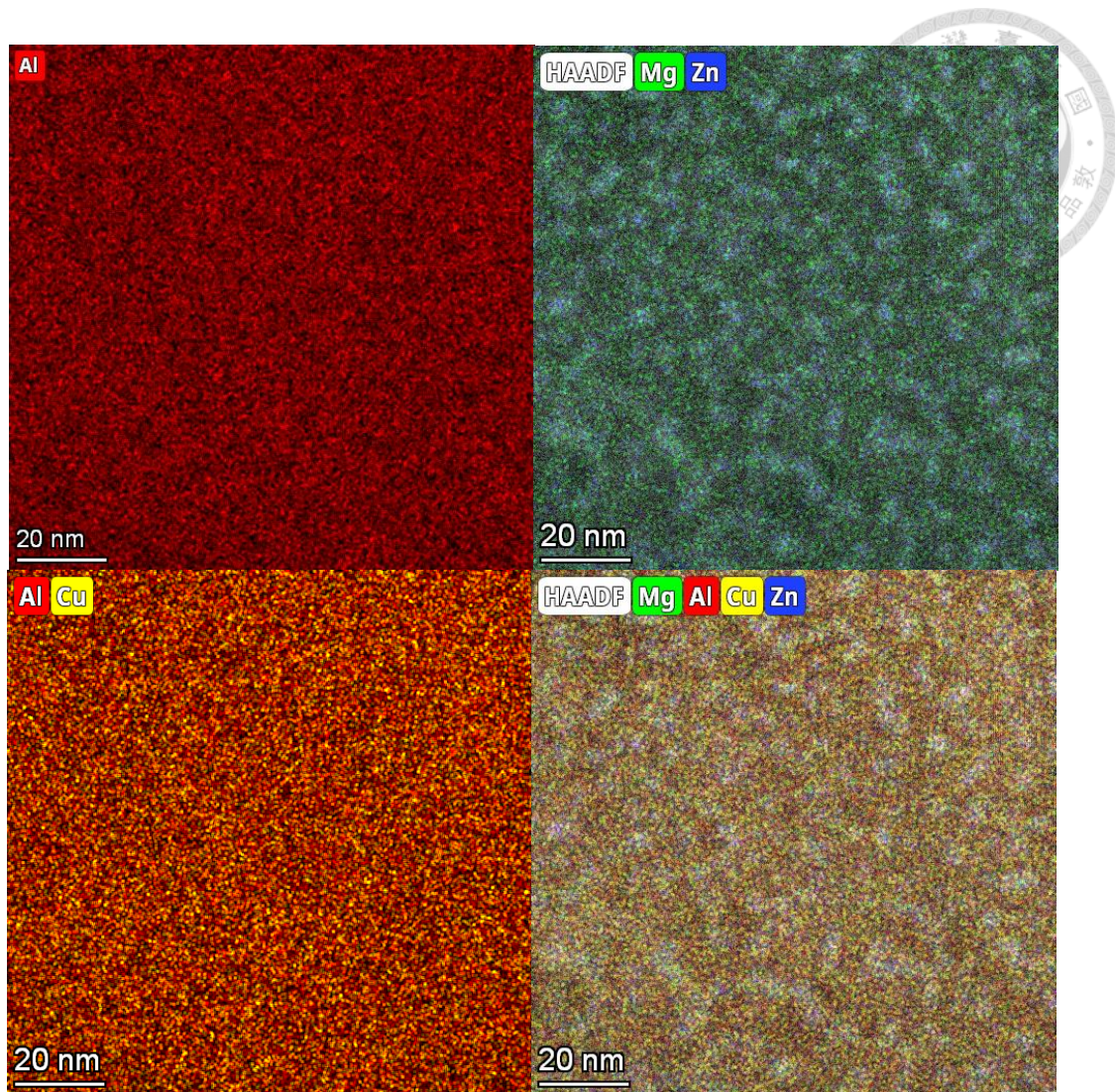


Figure 5-9 HAADF and EDS mapping of HCu after 475 °C 2 h + 490 °C 3 h+ 500 °C 1 h Solution Treatment with peak aging.

In atomic-resolution images shown in Figure 5-10, complete precipitates are visible, and the types of precipitates, identified as η_1 and η_2 based on FFT diffraction spots, are measured to be approximately less than 4 nm in size, consistent with previous statistical results. This suggests that the three-stage solution treatment process likely results in smaller precipitates.

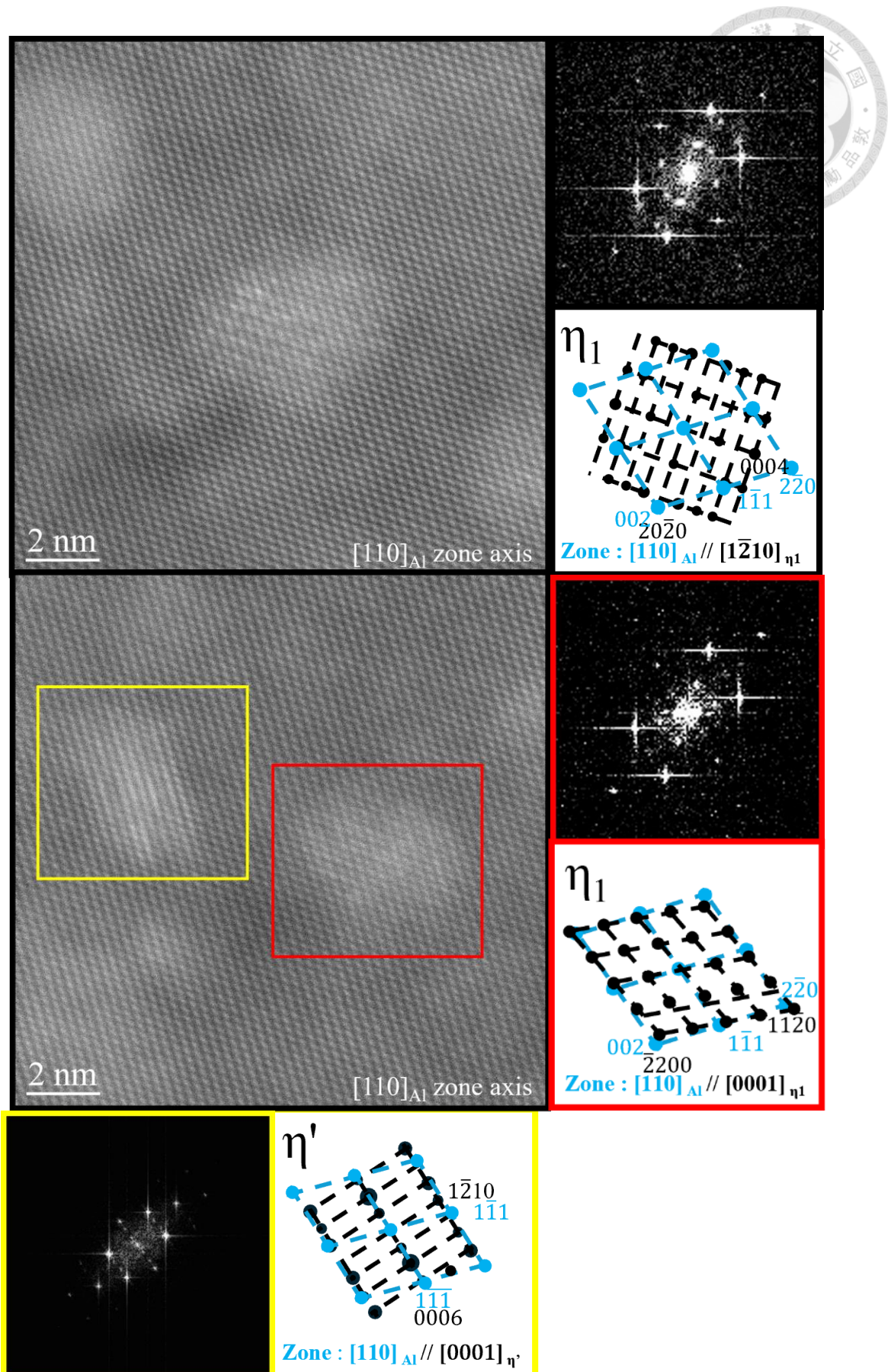


Figure 5-10 Atomic-resolution image and FFT diffraction of HCu after 475 °C 2 h + 490 °C 3 h + 500 °C 1 h solution treatment with peak aging.



To summarize, after the introduction of the third stage of solution treatment, there is a significant improvement in the mechanical properties of HCu, and this improvement may not be attributed solely to the influence of second phases. Furthermore, TEM data reveals that the precipitates in this process are slightly smaller than those in earlier processes, which could be a reason for the enhancement in mechanical properties. However, the exact mechanism behind this improvement remains to be clarified.

5.7 Comparison between LCu and HCu

Based on a review of the literature, it is evident that as the copper content reaches a certain threshold, the mechanical properties of aluminum alloys tend to decline with an increase in the proportion of copper, mainly due to second phases. Table 5-3 and Table 5-4 summarize the mechanical properties of single-stage, two-stage, and three-stage solution treatment in LCu and HCu. The experimental results comprehensively show that in single-stage solution treatment processes, LCu samples exhibit superior mechanical properties compared to HCu, especially in terms of ductility where HCu consistently lags behind LCu.

To address the issue of second phases, the application of two-stage solution treatment resulted in certain improvements in the mechanical properties of both LCu and HCu. However, the enhancement in mechanical properties was still more pronounced in LCu, with a similar trend observed in elongation rates. This discrepancy is likely due to differences in copper content. Notably, LCu showed significant improvements in elongation rates.

In efforts to further enhance the mechanical properties of HCu, a three-stage solution treatment was employed. This treatment led to substantial improvements in both strength and elongation rates for HCu, with the improvements being more significant than those

observed in LCu. The speculated reasons for this might be due to an increase in the density of precipitates and the dissolution of minute S phases, though the exact mechanisms still need to be clarified.

In summary, two-stage solution treatment is suitable for LCu materials, while three-stage solution treatment fits better for HCu materials, owing to differences caused by varying copper content. Increasing copper results in more copper-containing secondary phases, which degrade mechanical properties in a single-stage solution treatment. However, by utilizing two-stage and three-stage solution treatments, the issues associated with second phases can be effectively addressed. Moreover, HCu, which originally had poorer mechanical properties, can achieve levels comparable to LCu after the three-stage solution treatment.

Table 5-3 Mechanical properties of LCu on the condition peak ageing at 120 ° C.

LCu	YS(MPa)	UTS(MPa)	EL(%)
St. + NA + AA			
490°C 12h	549.3 ± 1.5	599.4 ± 0.6	13.5 ± 0.27
475°C 9h + 490°C 3h	551.0 ± 3.6	606.0 ± 1.4	15.0 ± 0.46
475°C 8h + 490°C 3h + 500°C 1h	555.0 ± 1.0	610.9 ± 1.7	14.7 ± 0.35

Table 5-4 Mechanical properties of HCu on the condition peak ageing at 120 ° C.

HCu	YS(MPa)	UTS(MPa)	EL(%)
St. + NA + AA			
490°C 6h	540.5 ± 2.2	597.0 ± 1.1	12.9 ± 0.13
475°C 3h + 490°C 3h	543.2 ± 3.7	604.2 ± 3.6	14.3 ± 0.71
475°C 2h + 490°C 3h + 500°C 1h	547.4 ± 3.5	613.8 ± 0.4	15.2 ± 0.80

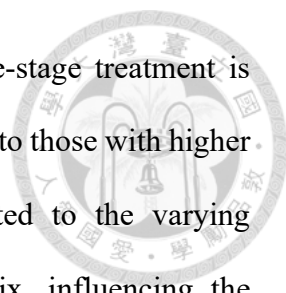
St. denoted to solution treatment, NA denoted to nature aging 7 days, and AA denoted to artificial aging 120 ° C 24h.

Chapter 6 Conclusion



The research findings provide a comprehensive understanding of the effects of multi-stage solution heat treatment on the mechanical properties and microstructure of aluminum alloys, specifically focusing on the contrast between low and high copper content alloys (LCu and HCu respectively). Here is a summary based on the insights:

1. **Enhancement of Mechanical Properties:** The multi-stage solution heat treatment significantly increased the mechanical properties, particularly the elongation rates, compared to single-stage heat treatment. This improvement indicates that the multi-stage process is more effective in optimizing the microstructure for better overall performance. According to statistics, the two-stage solution treatment increased the strength by approximately 10MPa and the elongation by about 3.2% in LCu. In HCu, the strength increased by approximately 10MPa, and the elongation by about 1.8%. The three-stage solution treatment increased the strength by approximately 15MPa and the elongation by about 2% in LCu. In HCu, the strength increased by approximately 15MPa, and the elongation by about 2.5%.
2. **Incomplete Solution of S Phase at 475 °C:** The heat treatment at 475 °C alone is insufficient to fully dissolve the S phase in the alloy, leaving S phase remained, leading to suboptimal mechanical properties, which can act as stress concentrators, potentially reducing ductility and strength.
3. **Formation of Incipient Melting at 490 °C:** The single-stage heat treatment at 490 °C can lead to incipient melting voids, which are detrimental to the material's mechanical properties.
4. **Merit of Two-Stage Solution Heat Treatment:** The two-stage solution heat treatment can solve the problems of S phase solution and incipient melting. However,



the extent of mechanical property improvement through three-stage treatment is more pronounced in alloys with lower copper content compared to those with higher copper content. This differential response might be attributed to the varying solubility and distribution of copper in the aluminum matrix, influencing the effectiveness of second phase dissolution.

5. Precipitates and Grain Size: The two-stage solution heat treatment does not significantly alter the size of precipitates and grains within the alloy. This observation suggests that while the treatment effectively modifies the distribution and solubility of second phases, it does not have a marked impact on the overall microstructural scale, such as precipitate or grain size, which are also crucial factors influencing mechanical properties. However, the three-stage solution treatment would decrease the precipitate size in HCu.

6. Improvement of high copper content aluminum: By our design, applying three-stage solution treatment, high copper content aluminum can reach better mechanical properties. Solving the problem caused by second phase. The strength and elongation are not less than low copper content aluminum.

These insights offer valuable guidance for the development of optimized heat treatment protocols aimed at maximizing the performance of aluminum alloys, particularly in applications where superior mechanical properties are critical. The findings underscore the importance of carefully selecting heat treatment conditions based on the specific alloy composition and the desired property enhancements.

Chapter 7 Future Work

1. Measure the thickness of the specimens using EELS to quantify the differences in precipitate density resulting from various solution treatment processes.
2. Delve into the mechanisms by which the addition of a third stage to the solution treatment process alters mechanical properties, and optimize the three-stage solution treatment process.
3. Utilize TEM to capture the diffraction spots of the second phases to establish their crystal structures.
4. Clarify the dissolution mechanism of the S phase in high copper aluminum alloys.



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