

CHARACTERISATION OF CREEP AND CREEP
CRACK GROWTH PROPERTIES IN A NOVEL HEAT
RESISTANCE STEEL

Volume 2



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Chapter 2

Table 2.1 Typical Grade 91 heat treatment process and tensile strength. [7]

	C	Si	Mn	Cr	Mo	W	Nb	V	N	B
Nominal composition	0.09	0.25	0.50	9.0	0.50	1.80	0.05	0.20	0.050	0.003
ASME*	0.07/0.13	0.50 max	0.30/0.60	8.50/9.50	0.30/0.60	1.50/2.00	0.04/0.09	0.15/0.25	0.030/0.070	0.001/0.006

* American Standard of Mechanical Engineering/ as P92, T92 in code case [1995].

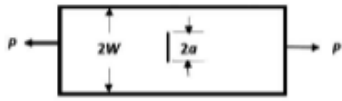
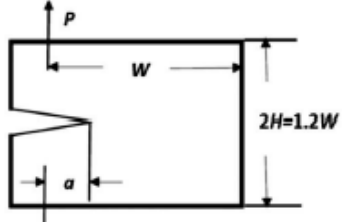
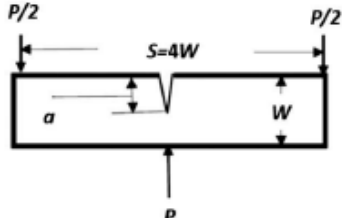
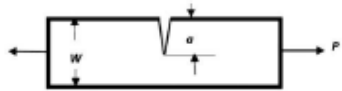
Phosphorus content is restricted to 0.02 % mass maximum.

Ni content is restricted to 0.04 % mass maximum and it is under discussion to revise it to less than 0.02 % mass maximum (from 2009).

Table 2.2 Heat treatments and tensile requirements for Gr:91 steels [7].

	Grade	Normalizing (°C)	Tempering (°C)	Tensile strength (MPa)	Yield strength (MPa)
Seamless tube	SA213M T91	1040–1080	730–800	585 min	415 min
Seamless pipe	SA335M P91	1040–1080	730–800	585 min	415 min
Plate	SA387M 91	1040–1080	730–800	585–760	415 min

Table 2.3 K calculations of different geometries. [58-59]

Specimen Geometry	$F\left(\frac{a}{W}\right) = \frac{K}{P}BW^{1/2}$
 Center Crack Tension, CC(T)	$\frac{1}{2} \sqrt{\pi \left(\frac{a}{W}\right) \sec \frac{\pi a}{2W}} \left[1 - 0.025 \left(\frac{a}{W}\right)^2 + 0.06 \left(\frac{a}{W}\right)^4 \right]$
 Compact Type, C(T)	$\left[\frac{2 + a/W}{(1 - a/W)^{1.5}} \right] \left(0.886 + 4.64 \left(\frac{a}{W}\right) - 13.32 \left(\frac{a}{W}\right)^2 + 14.72 \left(\frac{a}{W}\right)^3 - 5.6 \left(\frac{a}{W}\right)^4 \right)$
 Single Edge Crack Bend, SEC(B)	$\frac{3(S/W)(a/W)^{1/2} [1.99 - a/W(1 - a/W)] [2.15 - 3.93(a/W) + 2.7(a/W)^2]}{2(1 + 2a/W)(1 - a/W)^{3/2}}$
 Single Edge Crack Tension, SEC(T)	$\sqrt{\pi \left(\frac{a}{W}\right)} \left[1.1176 + 0.1297 \left(\frac{a}{W}\right) + 3.8146 \left(\frac{a}{W}\right)^2 - 3.9246 \left(\frac{a}{W}\right)^3 + 3.2687 \left(\frac{a}{W}\right)^4 - 0.7024 \left(\frac{a}{W}\right)^5 \right]$
 Double Edge Crack Tension, DEC(T)	$\left(\frac{\pi(a/W)}{(1 - a/W)} \right)^{1/2} \left[1.122 - 0.561 \left(\frac{a}{W}\right) - 0.205 \left(\frac{a}{W}\right)^2 - 0.471 \left(\frac{a}{W}\right)^3 - 0.7024 \left(\frac{a}{W}\right)^4 \right]$

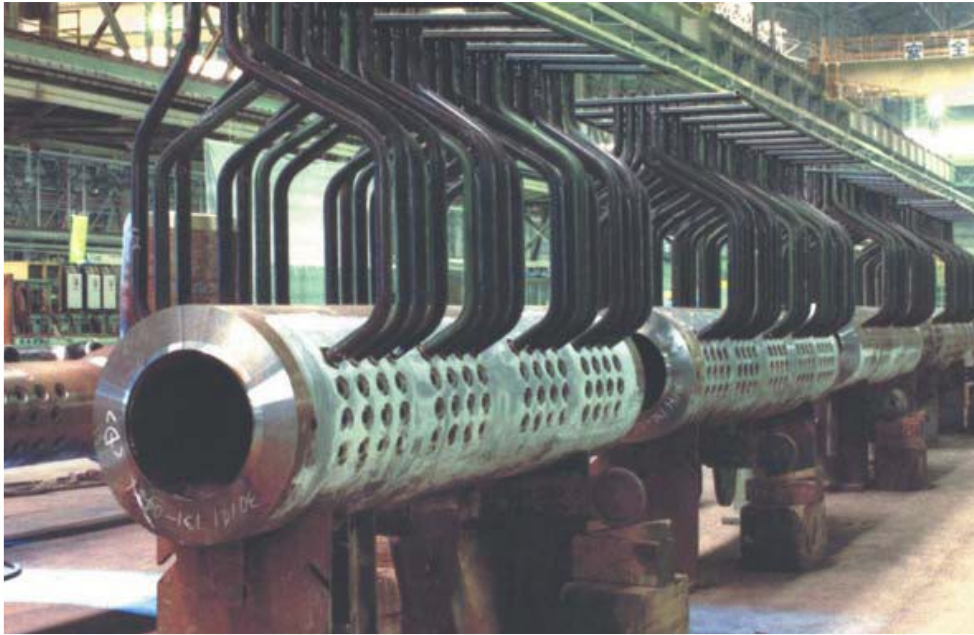


Figure 2.1 Picture of a typical header.[1]

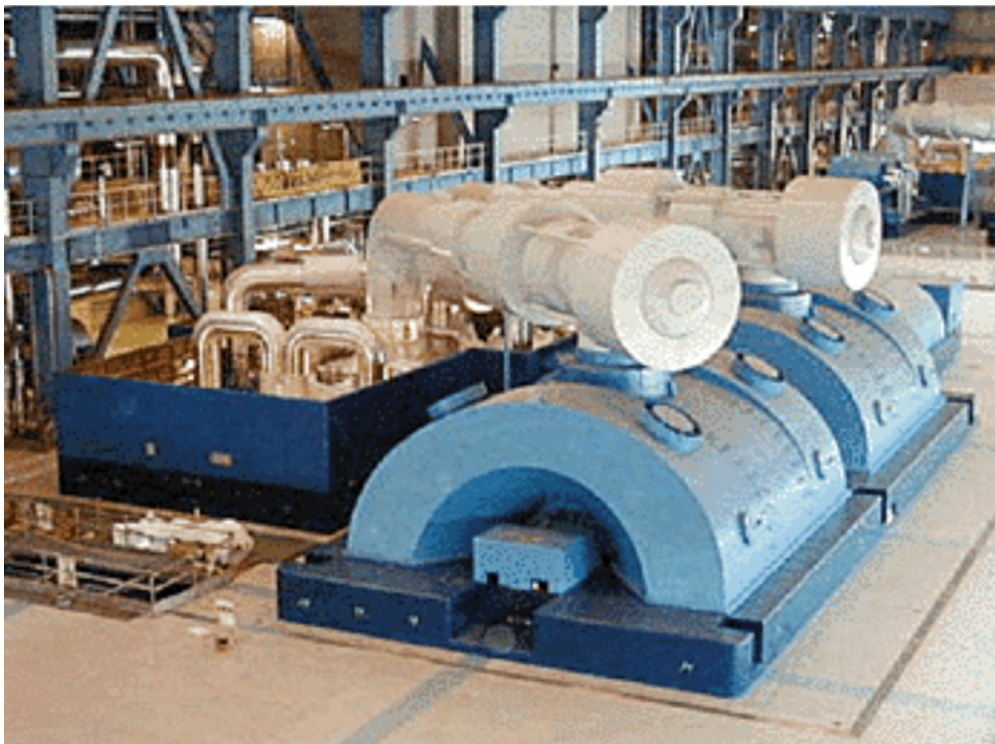


Figure 2.2 A classic steam turbine in a power plant (service temperature: 600°C , stress: 25.1 MPa). [2]

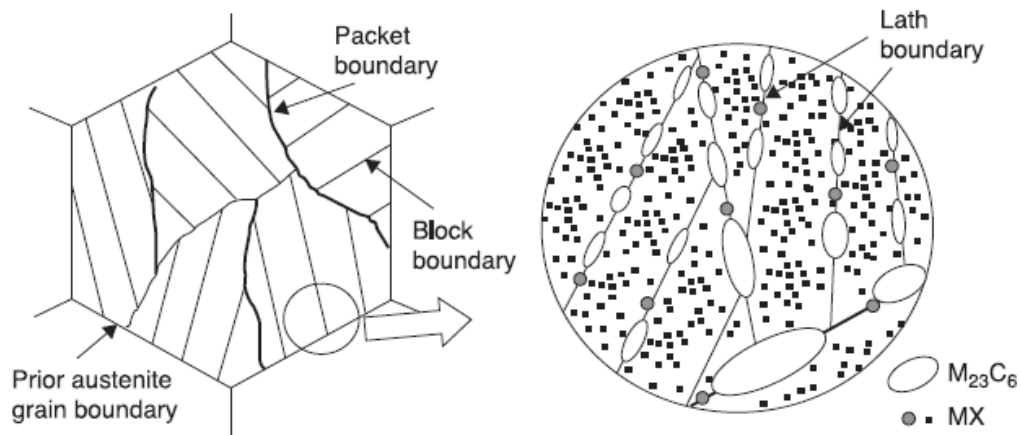


Figure 2.3 Schematic drawing of a typical martensite ferritic steels' microstructure (tempered martensitic lath structure was involved). [7]

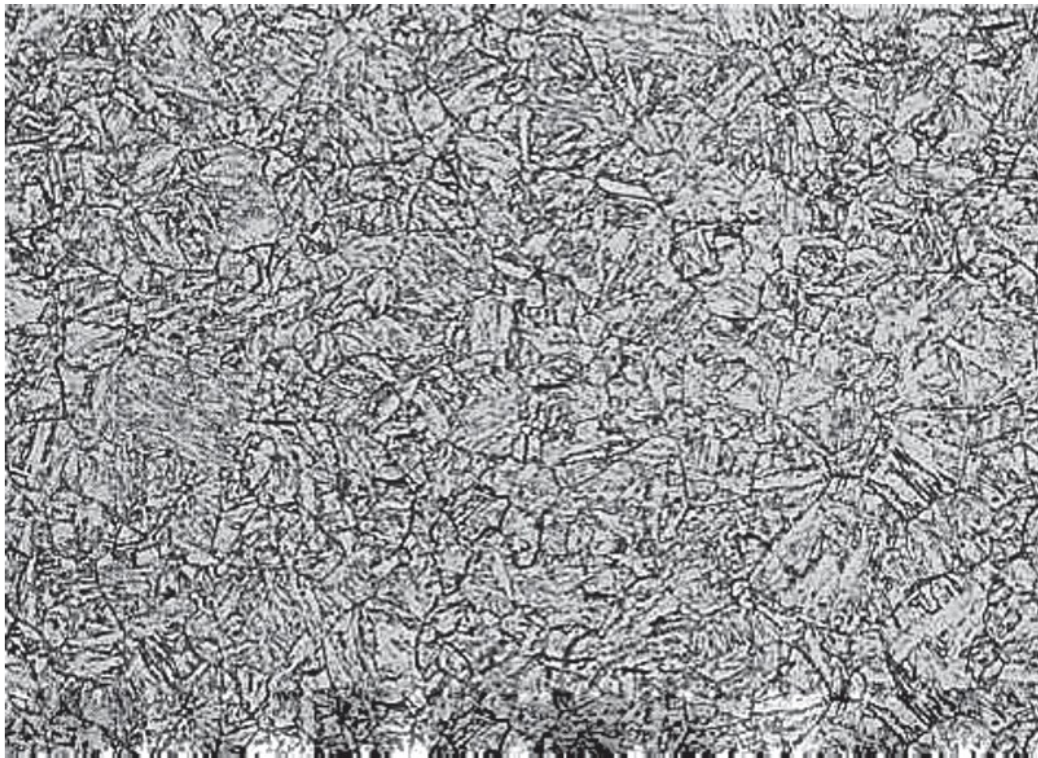


Figure 2.4 Typical optical micrograph of Grade 92 material. [7]

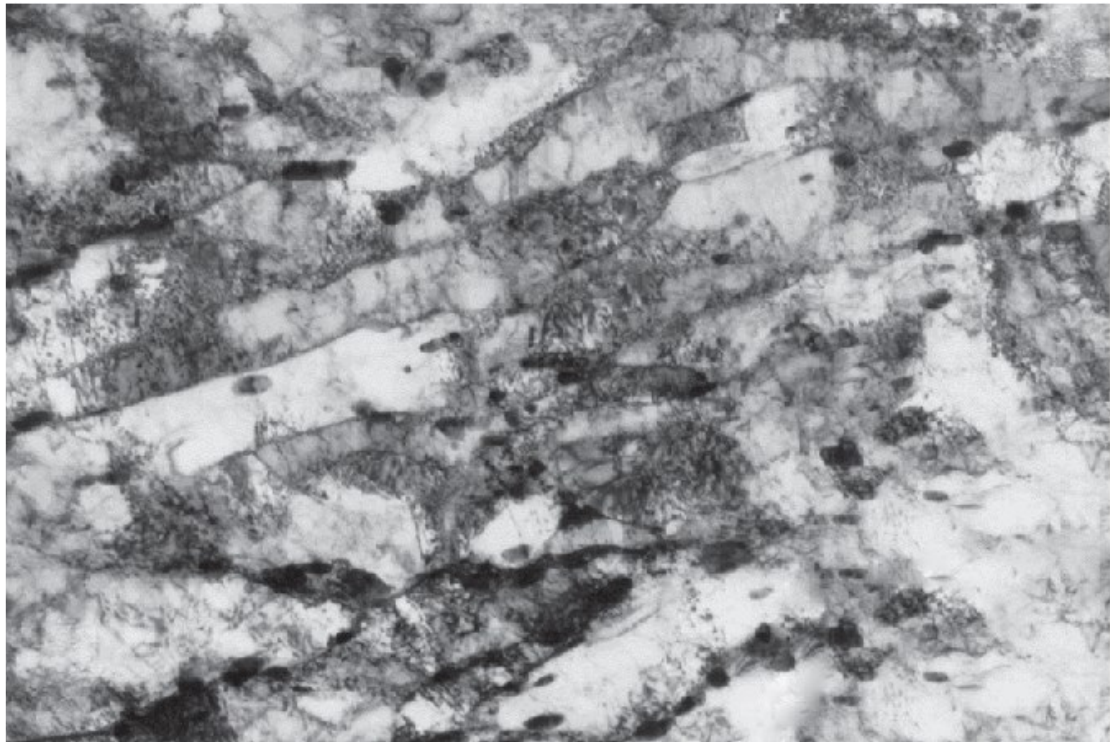


Figure 2.5 TEM image of Grade 91's microstructure after heat treatment. [7]

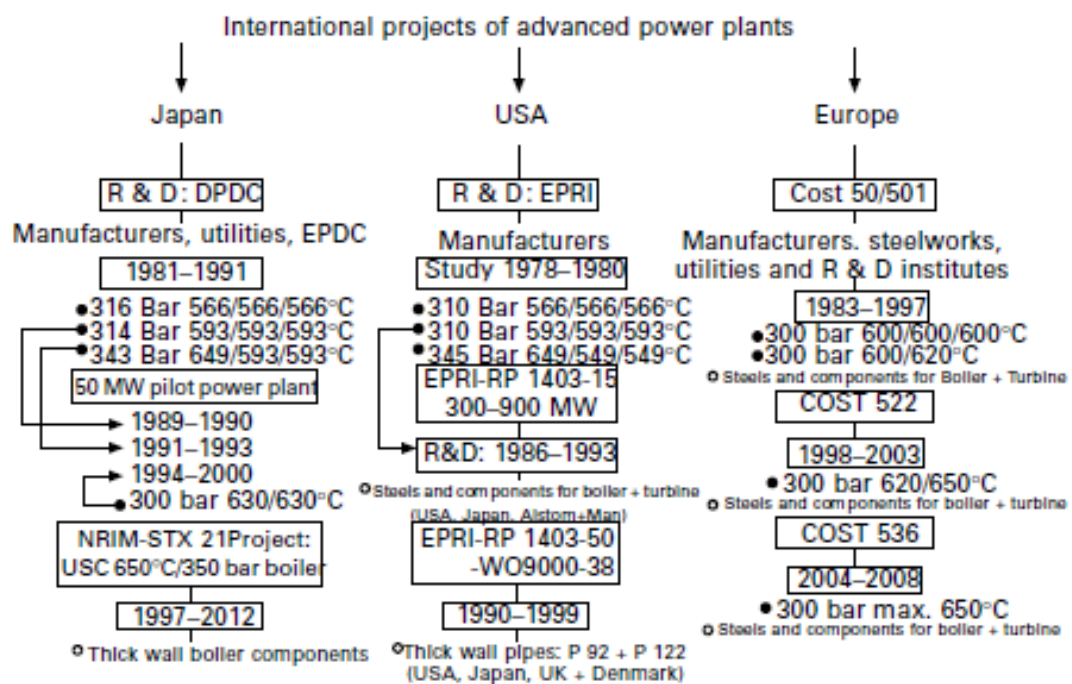


Figure 2.6 Overview of the evolution in the development of heat-resistant steels applied for advanced power plants since 1978. [14]

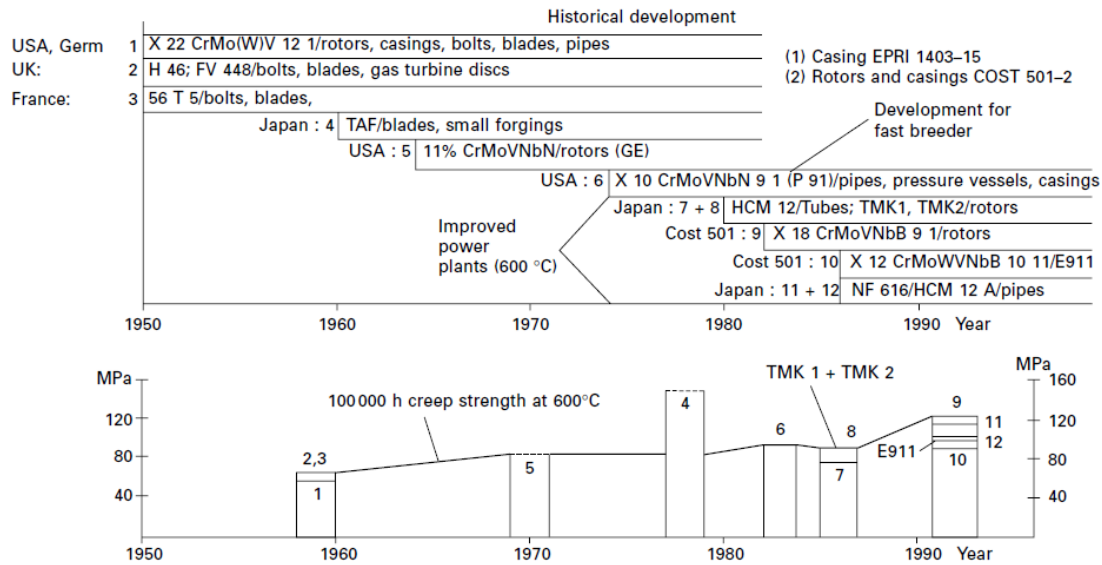


Figure 2.7 The development of high chromium (9-12%) steels from 1950 to 1995 and its creep rupture strength under 600°C for long period (100,000 hours). [14]

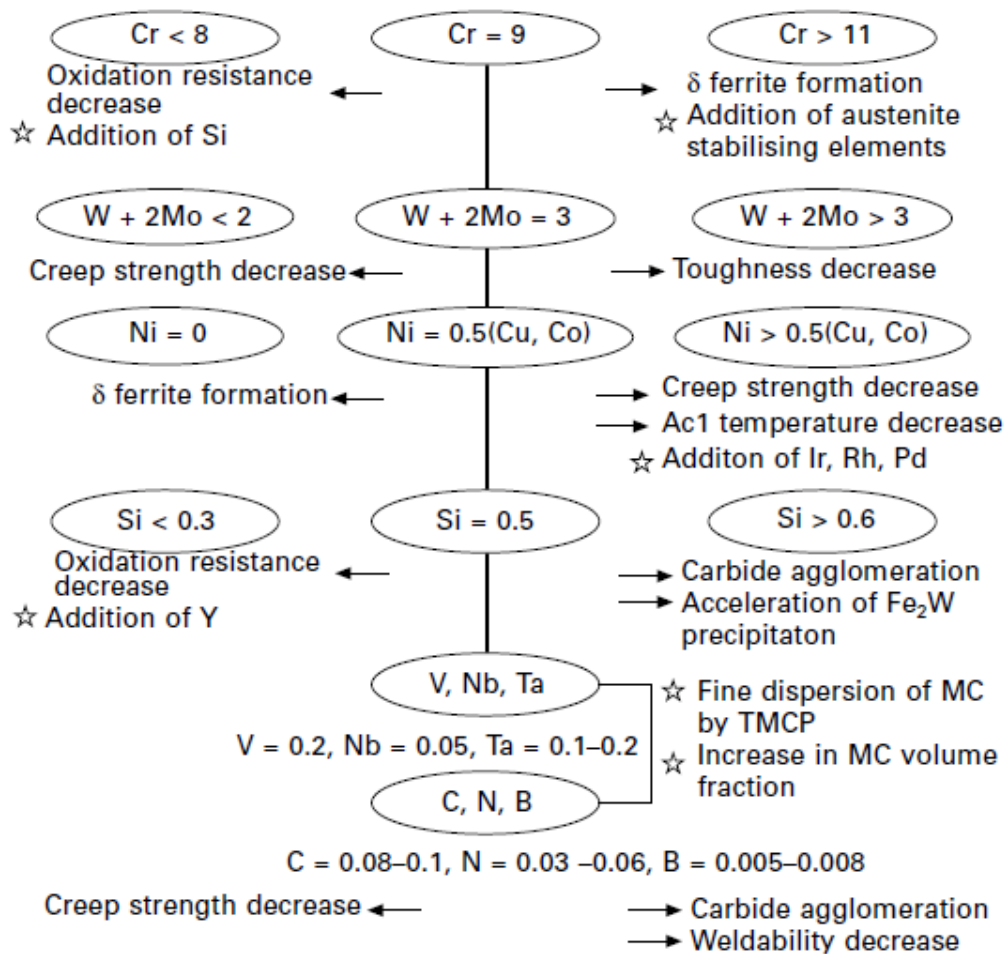


Figure 2.8 Illustration of guiding principles in the development of high chromium content ferritic heat resistant steels for ultra-supercritical (USC) boilers (650°C).[14]

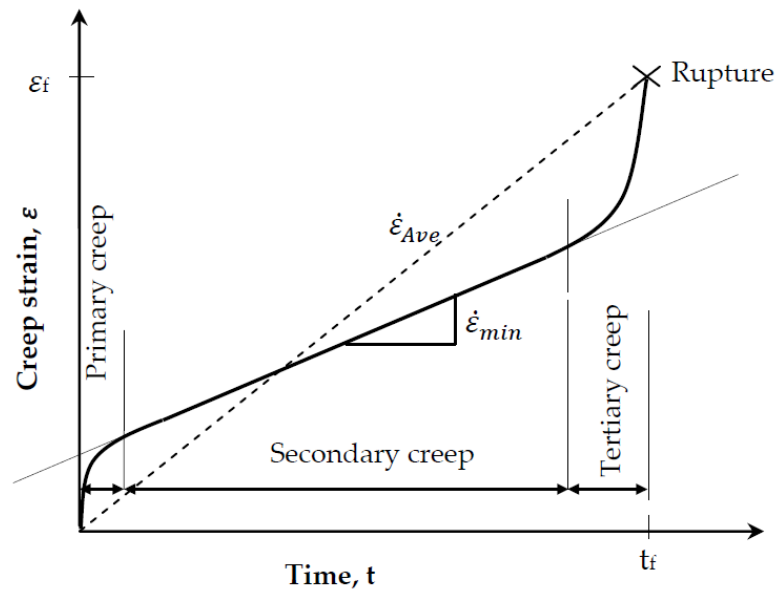
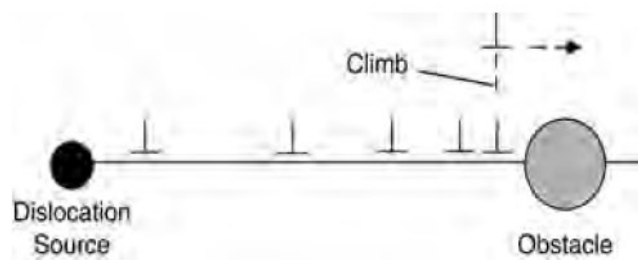
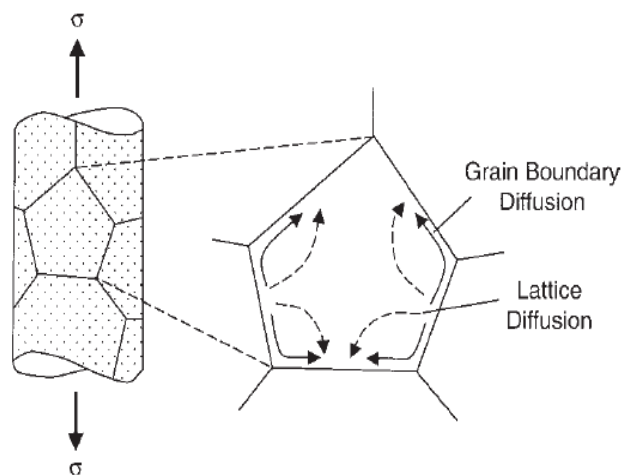


Figure 2.9 Typical uniaxial creep strain curve



(a) Dislocation creep mechanism



(b) Diffusion creep mechanism

Figure 2.10 Basic creep mechanisms

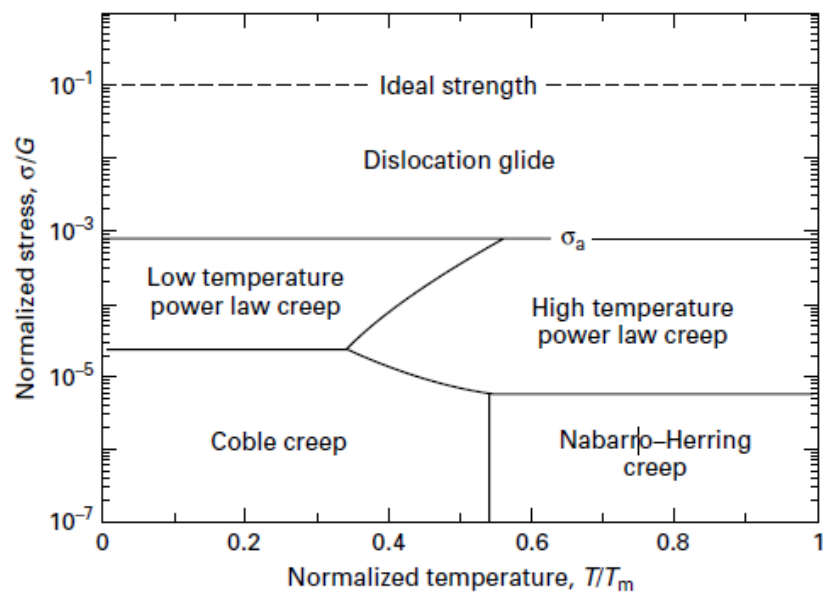


Figure 2.11 Schematic drawing of creep deformation mechanism map. [14]

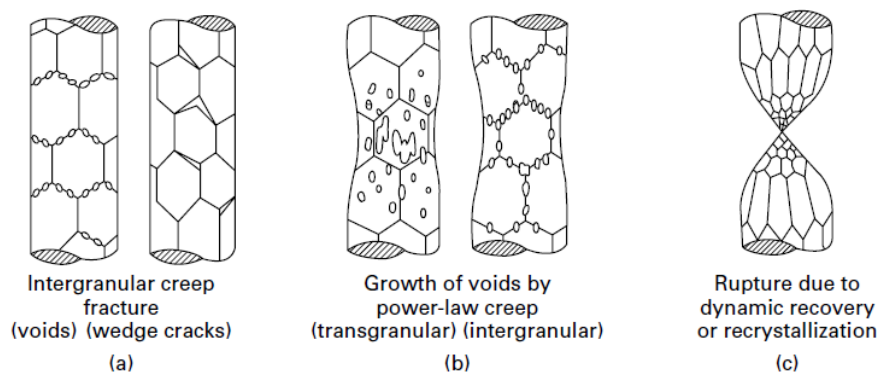


Figure 2.12 Schematic drawing of creep fracture mechanisms.[14]

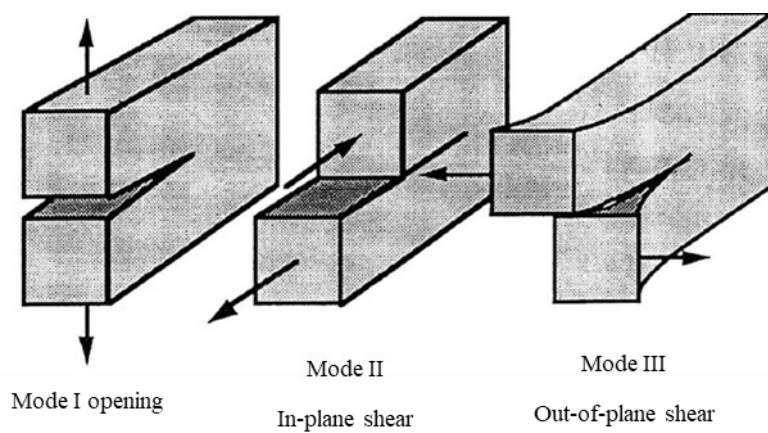


Figure 2.13 Three modes of loading a cracking body.

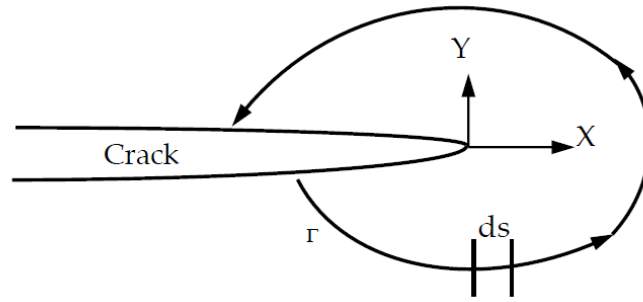


Figure 2.14 Schematic of the arbitrary contour used to get the integral J . [42]

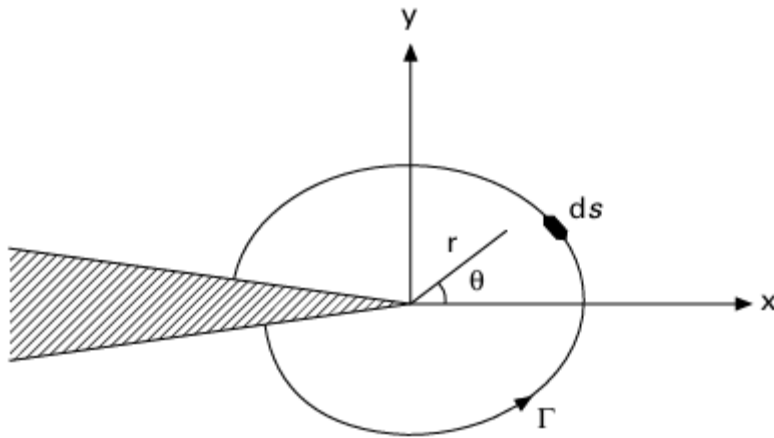


Figure 2.15 Contour integral around the crack tip for the definition of non-linear fracture mechanic C^* parameter. [60]

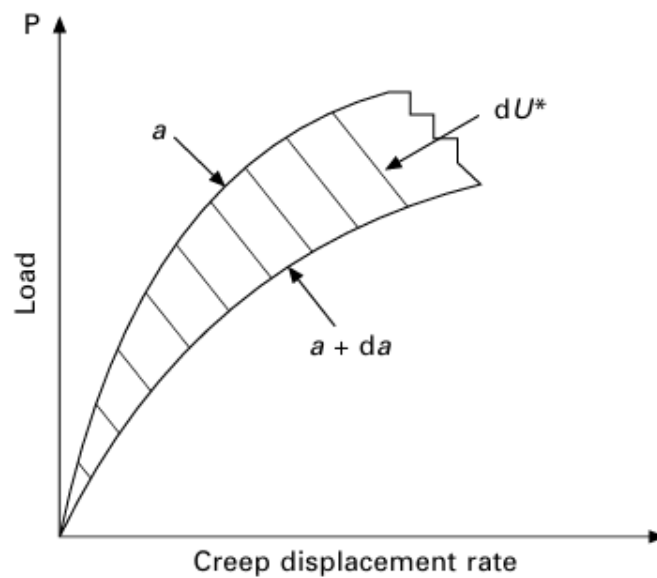


Figure 2.16 Load versus creep displacement rate curves with different crack sizes of non-linear materials. [60]

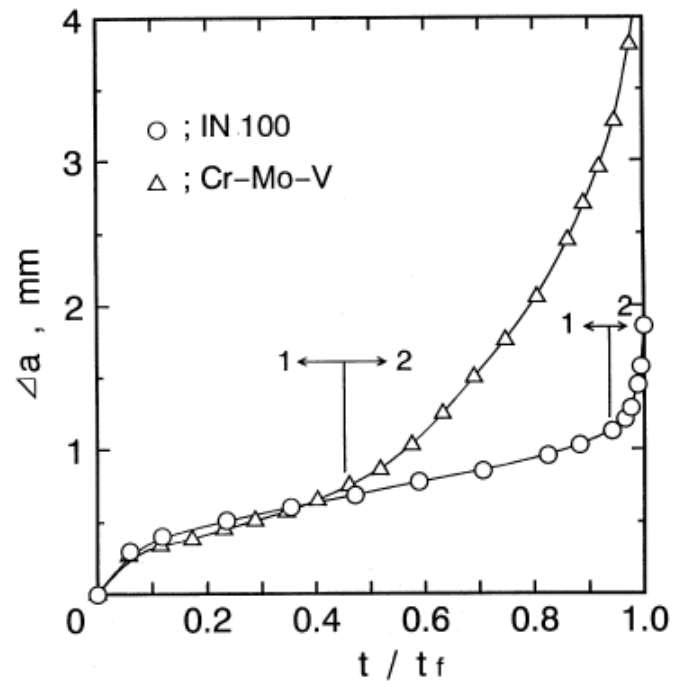


Figure 2.17 An example of creep crack growth curves for Cr-Mo-V alloy and IN100 indicating the difference between creep ductile material and creep brittle material.[45]

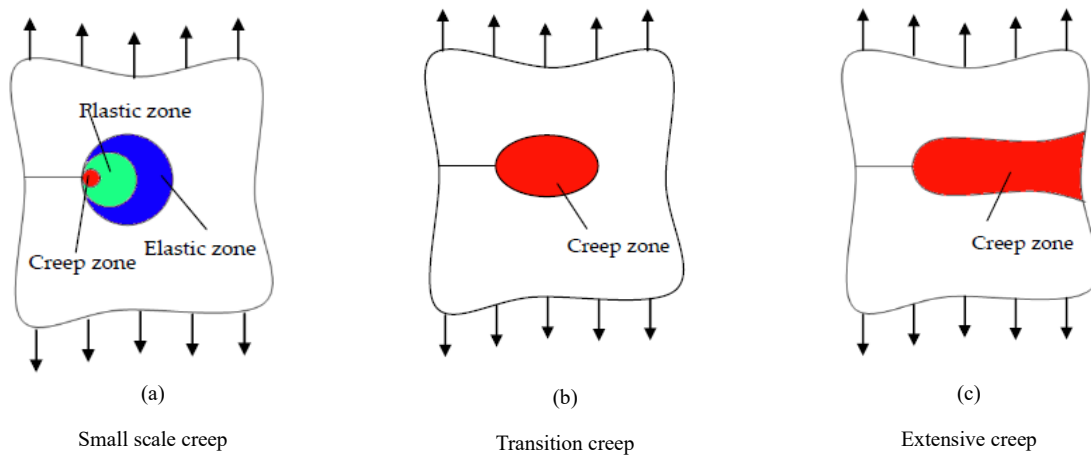


Figure 2.18 Three creep deformation regimes with a crack defect. [46]

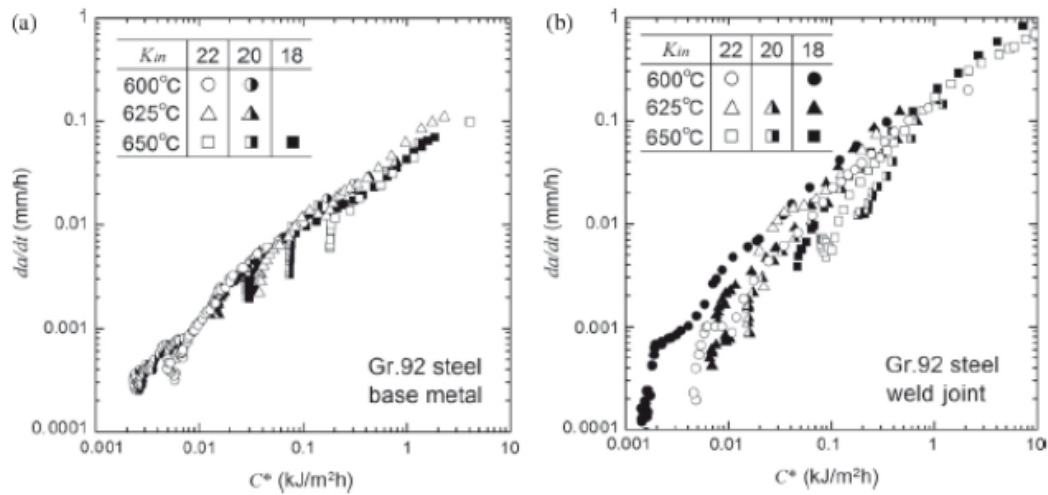


Figure 2.19 Creep crack growth resistance curves with C^* parameter at different temperatures and loading conditions on P92 and weld materials. [52]

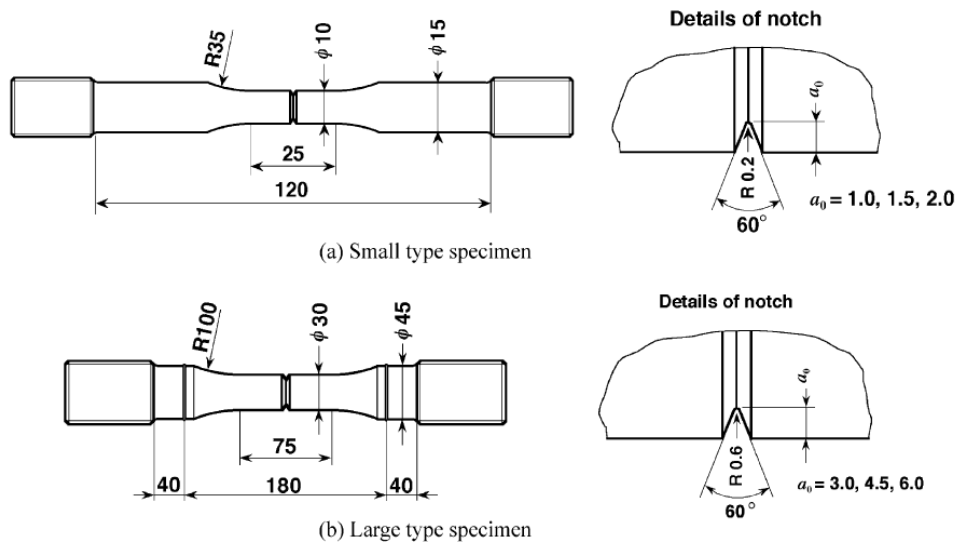


Figure 2.20 Schematic drawing of circular notched testpiece with different notch depth versus diameter ratio. [52]

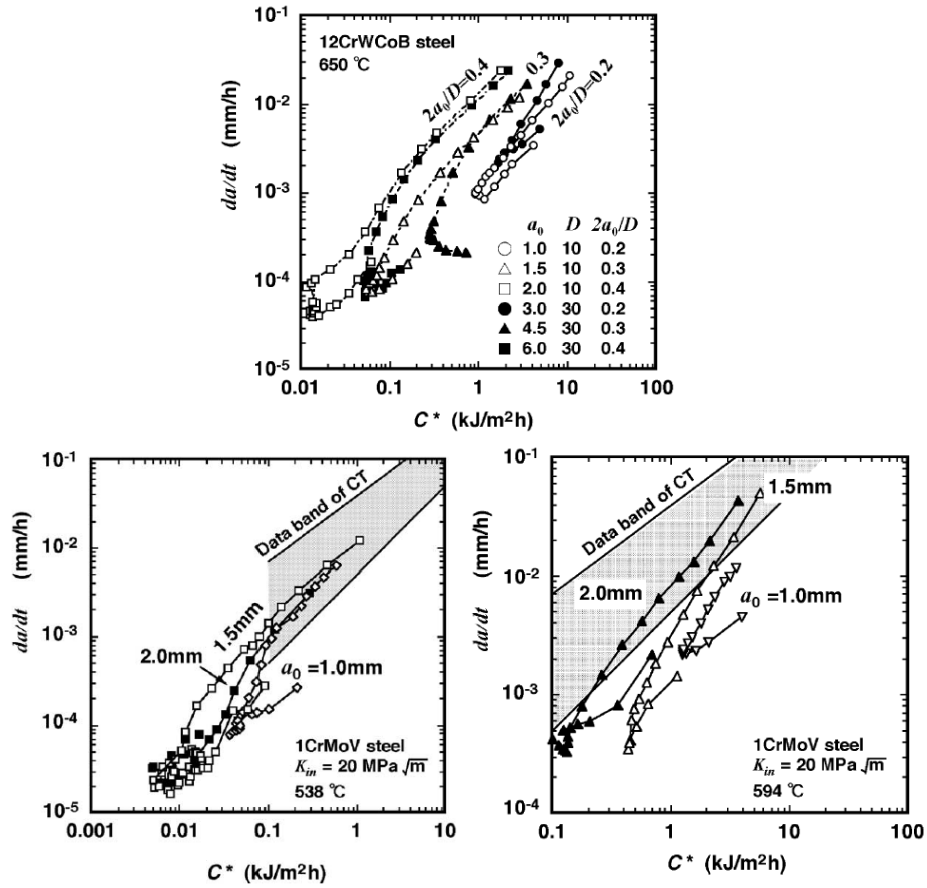


Figure 2.21 Creep crack resistance curves with C^* parameter of two different Cr heat resistant steels under different temperatures and loading conditions. [52]

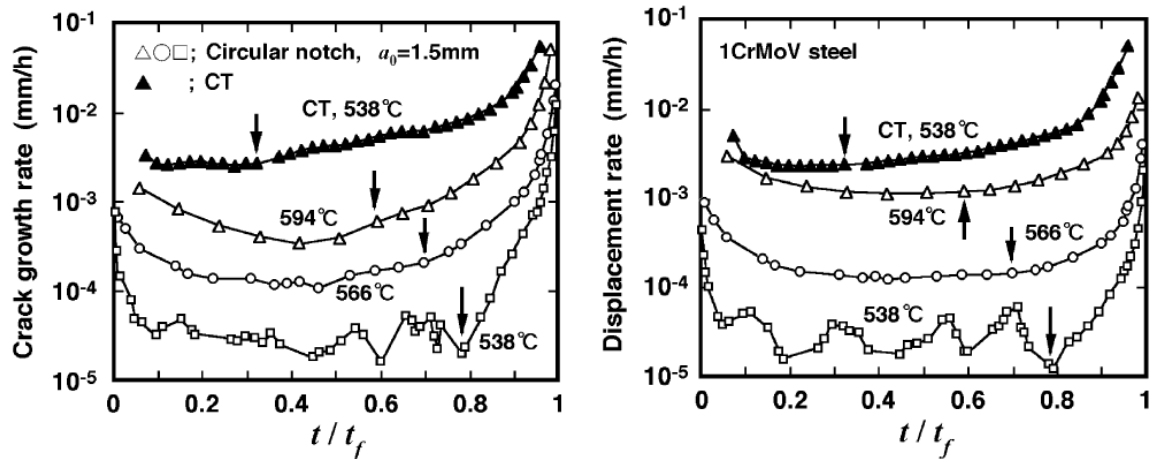


Figure 2.22 Relations between creep crack growth rate and displacement rate versus time of different geometries specimens. [52]

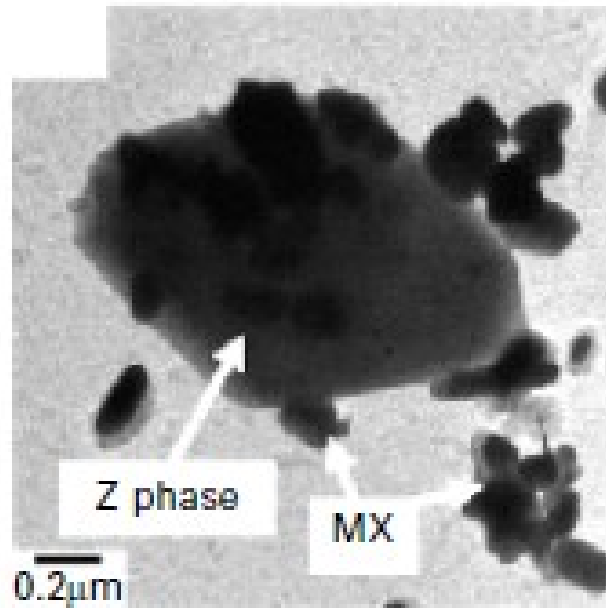


Figure 2.23 Z-phase precipitated in NF12 tempered martensite steel at 650°C. [54]

Chapter 3

Table 3.1 General chemical composition of P92 and BG12Cr.

	C	Si	Mn	Cr	Mo	W	Co	V	Nb
P92	0.07-0.13	0-0.50	0.30-0.60	8.50-9.50	0.30-0.60	1.50-2.00	-	0.15-0.25	0.04-0.09
BG12Cr	0.08-0.12	0.40-0.60	0.15-0.45	11.0-12.0	0.20-0.40	1.00-2.00	1.60-3.00	0.20-0.30	0.03-0.08
	B	N	Ni	Al	Ti	Zr	Cu	Fe	
P92	0.001-0.006	0.030-0.070	0-0.40	0-0.02	0-0.01	0-0.01	-	Balance	
BG12Cr	0.003-0.006	0.030-0.070	-	-	-	-	-	Balance	

Table 3.2 Summary of stress-ruptured specimens.

Material	Test temperature (° C)	Stress (MPa)	Ruptured specimens' status	Rupture life (hour)	d ₀ /d _u (mm)	L ₀ /L _u (mm)
P92-E	649	90	Half	12234	4.98/4.94	/
P92-E	649	100	Half	7756	/	/
BG12Cr-E	650	65	Half	10380	5.03/2.25	/
BG12Cr-E	625	110	Half	7286	5.00/1.94	25/31.23
BG12Cr-E	625	100	Half	8597	5.01/1.84	25/30.83
BG12Cr-E	650	75	Half	5723	5.02/1.76	25/30.85
BG12Cr-E	650	90	Complete	3048	10.02/3.64	50/61.67
“complete” means: the two pieces of ruptured specimen received. “half” means only half of ruptured specimen received. “/” means: not recorded. All the data of these ruptured specimens were supplied by Baowu Steel.						

Table 3.3 Summary of creep crack growth tests in ring-notched geometry.

Material	Sample ID	Diameter	Net section	Initial notch depth	Load	Net section Stress	Duration	Final crack depth	Displacement	L
		(mm)	diameter	(mm)	(kN)	(MPa)	(hour)	(mm)	(mm)	
P92	PL-1	9.58	7.28	1.15	4.00	96	4131.0	1.32	0.09	
P92	PS-2-1	7.98	5.66	1.16	3.60	145	307.0	0.41	Extensometer not attached (trail test)	
	PS-2-2			/	4.30	170	166.4	/		
	PS-2-3			/	6.30	250	0.2	/		
P92	PS-3	7.98	5.66	1.16	5.03	200	98.0	0.17	Interrupted by reaching machine soft and violated limitation/0.40	
P92	PL-4	9.53	7.17	1.18	6.00	149	536.2	0.83	1.00	
BG12Cr	BL-1	9.53	7.17	1.18	6.04	150	3234.0	0.80	0.24	
BG12Cr	BS-2	8.09	5.67	1.21	5.07	201	1346.6	0.58	0.34	
BG12Cr	BS-3	7.97	5.66	1.15	4.30	170	1450.3	0.42	0.24	
*Crack was not straight; crack extension is an averaged value.										

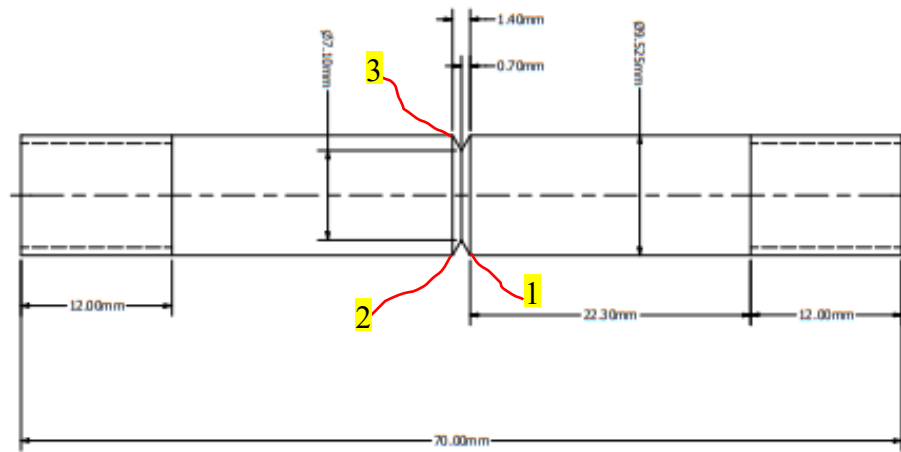


Figure 3.1 Schematic drawing of ring-notched specimen for creep crack growth and spot weld positions for d.c.p.d crack length monitoring method.

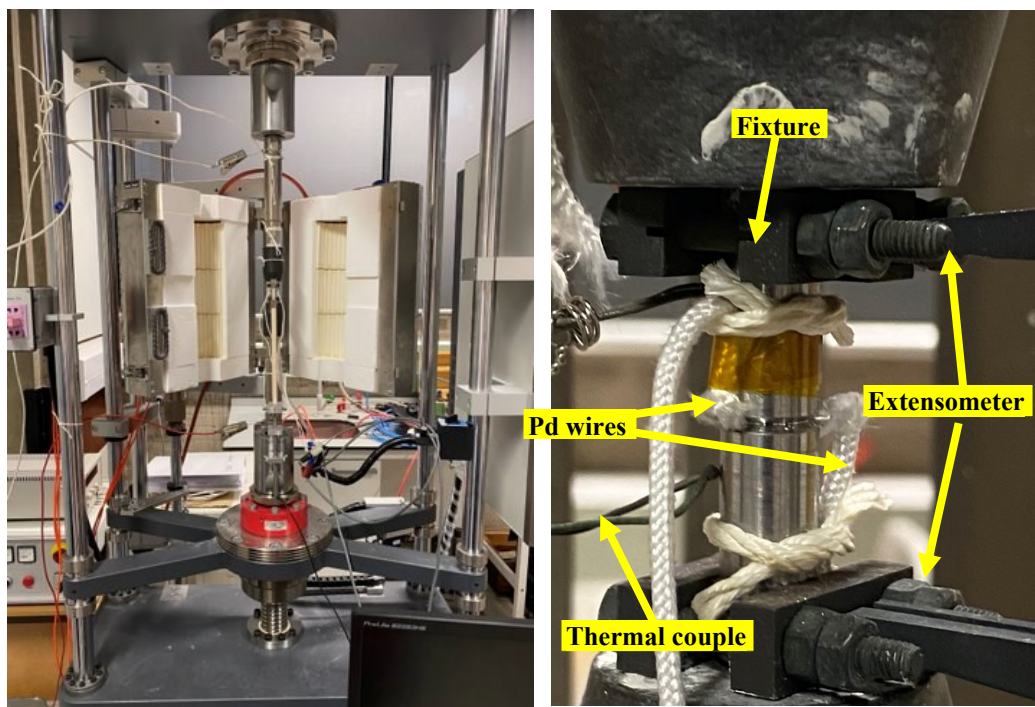


Figure 3.2 The setting-up for a circular notched creep crack growth test on a screw driving Zwick Kappa machine.

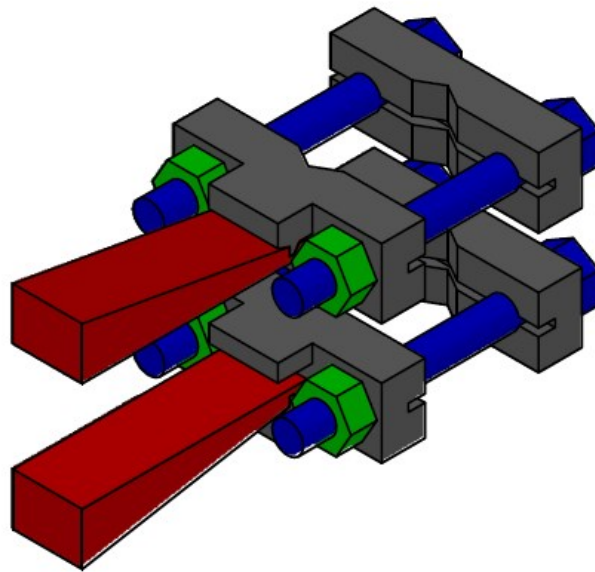
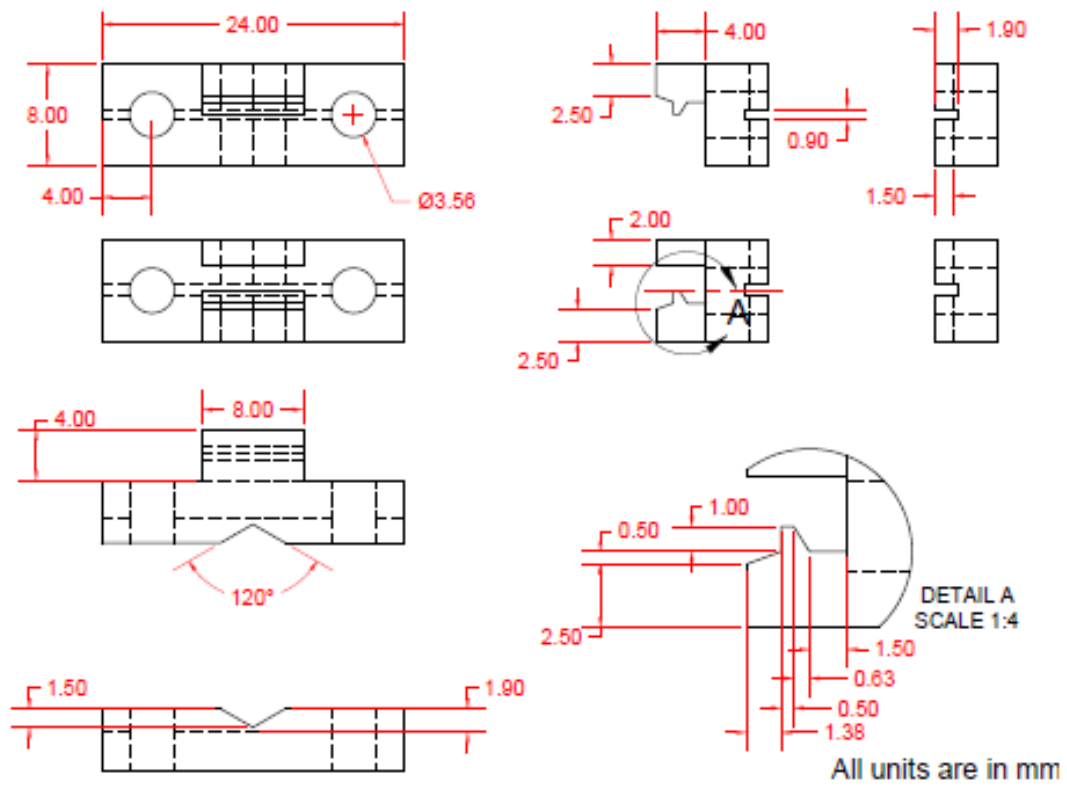


Figure 3.3 Schematic drawing of extensometer fixtures for ring-notch creep crack growth loadline displacement recording.

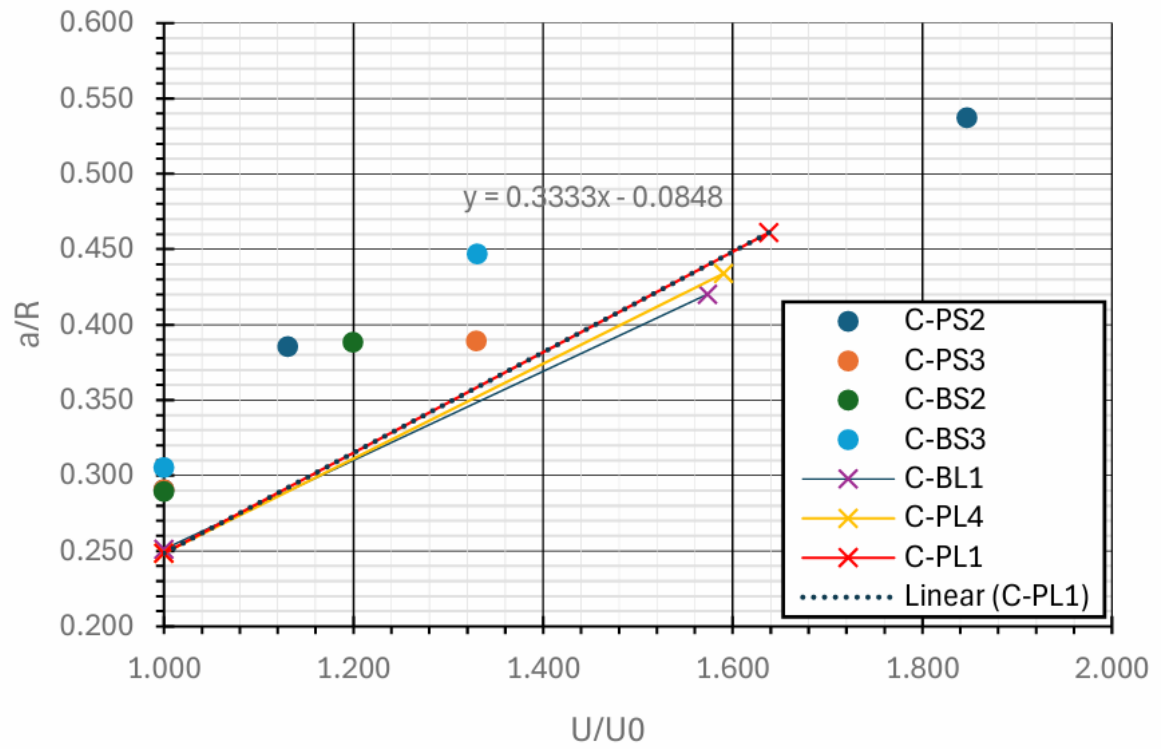


Figure 3.4 Crack size calibration plot (a/R versus U/U_0)

Chpater 4

Table 4.1 Summary of creep crack growth tests in CT geometry.

Material	Test temperature (° C)	Load (kN)	Number of creep crack growth regime	Initial K (MPa√m)	Duration (hour)	Crack growth (mm)
P92	600	6	2	21.6/25.8	729/569	0.31/1.17
P92	600	10	2	37.9/45.2	65/29	0.59/0.94
P92	600	10	2	39.1/55.7	193/1	2.29/1.44
P92	650	3	2	11.3/14.4	1863/235	0.64/0.80
P92	650	6	1	21.1	188	6.73
P92	700	3	1	10.8	158	2.8
BG12Cr-E TWT	600	6	2	23.7/26.6	759/479	0.17/0.41*
BG12Cr-E TWT	600	6	1	20.5	886	4.95
BG12Cr-E plate	600	10	2	35.7/50.2	114/40	0.31/2.48
BG12Cr-E TWT	600	10	1	36.4	287	4.66
BG12Cr-E TWT	650	3	2	10.9/14.1	557/1968	0.92/1.38
BG12Cr-E TWT	650	6	2	20.5	886	4.92
BG12Cr-E TWT	700	3	1	11.1	836	4.32
*Crack was not straight; crack length is an averaged value.						

Table 4.2 Summary of creep crack growth tests in ring-notched geometry.

Material	Sample ID	Diameter	Net section	Initial notch depth	Load	Net section Stress	Duration	Final crack depth	Displacement	L
		(mm)	diameter	(mm)	(kN)	(MPa)	(hour)	(mm)	(mm)	
P92	PL1	9.58	7.28	1.15	4.00	96	4131.0	1.32	0.09	
P92	PS2-1	7.98	5.66	1.16	3.60	145	307.0	0.41	Extensometer not attached (trail test)	
	PS2-2			/	4.30	170	166.4	/		
	PS2-3			/	6.30	250	0.2	/		
P92	PS3	7.98	5.66	1.16	5.03	200	98.0	0.17	Interrupted by reaching machine soft and violated limitation/0.40	
P92	PL4	9.53	7.17	1.18	6.00	149	536.2	0.83	1.00	
BG12Cr	BL1	9.53	7.17	1.18	6.04	150	3234.0	0.80	0.24	
BG12Cr	BS2	8.09	5.67	1.21	5.07	201	1346.6	0.58	0.34	
BG12Cr	BS3	7.97	5.66	1.15	4.30	170	1450.3	0.42	0.24	
*Crack was not straight; crack extension is an averaged value.										

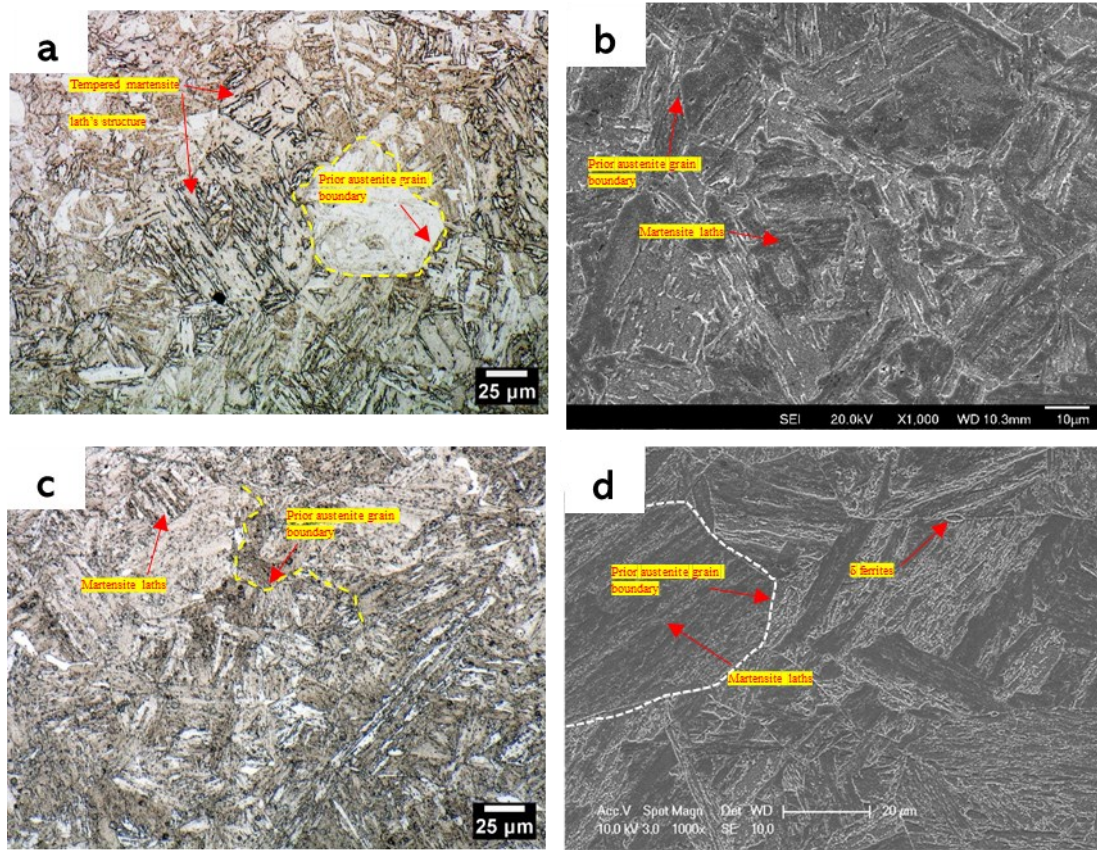
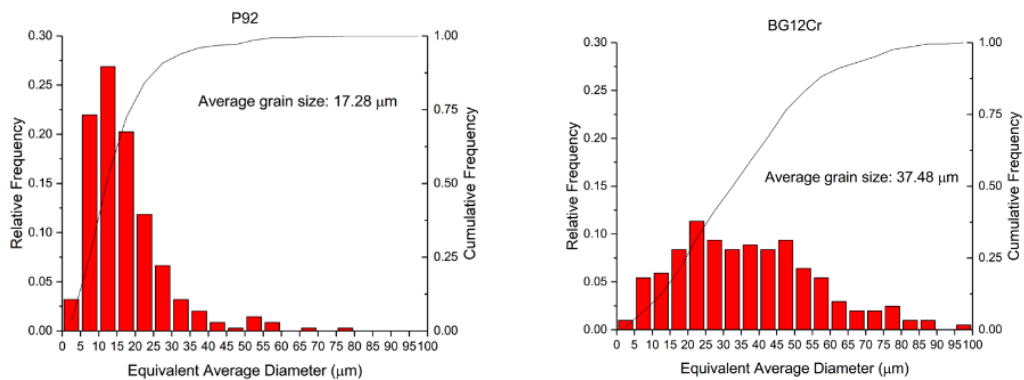


Figure 4.1 (a) optical image of as-received P92, (b) high magnification SE micrograph of as-received P92, (c) optical image of as-received BG12Cr, (d) high magnification SE micrograph of as-received BG12Cr.



(a) Prior austenite grain size distribution histogram of P92 (b) Prior austenite grain size distribution histogram of BG12Cr

Figure 4.2 Prior austenite grain size distribution histogram of both alloys.

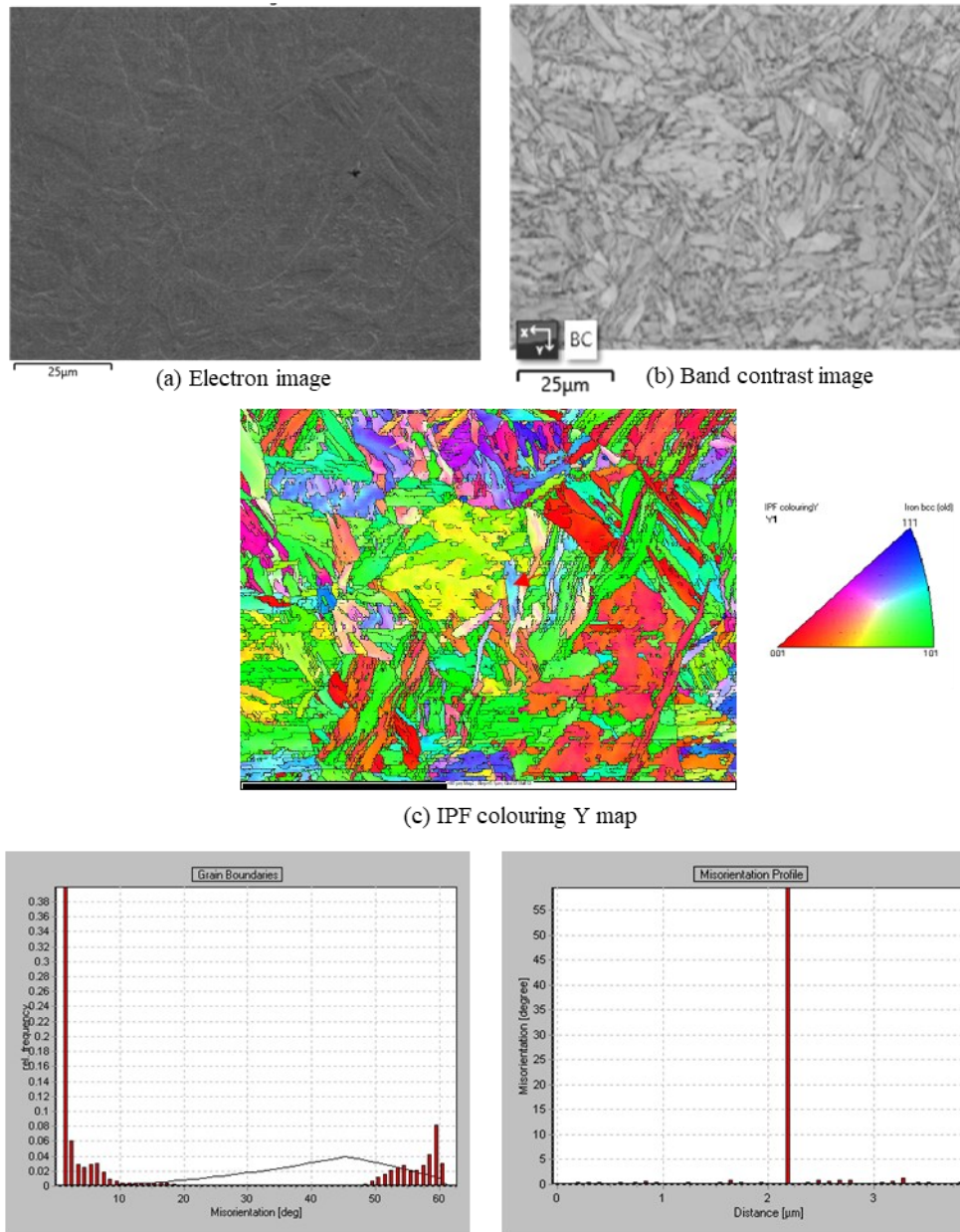


Figure 4.3 EBSD scanning image of P92 as-received material: (a) Secondary electron image; (b) Band contrast image; (c) IPF colouring Y image; (d) Grain boundary distribution histogram and misorientation profile across two adjacent laths.

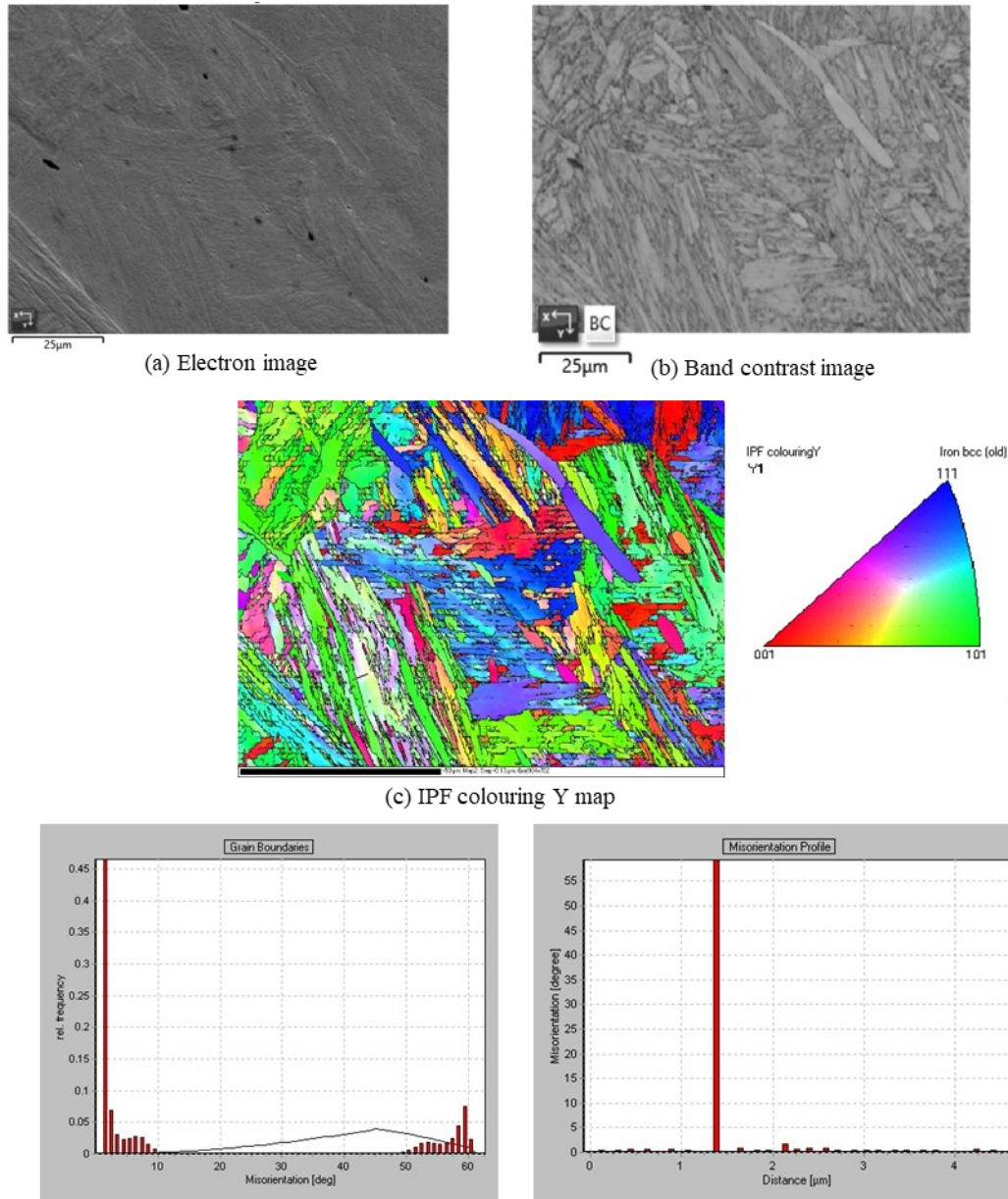


Figure 4.4 EBSD scanning image of BG12Cr as-received material: (a) Electron image; (b) Band contrast image; (c) IPF colouring Y image; (d) Grain boundary distribution histogram and misorientation profile across two adjacent laths.

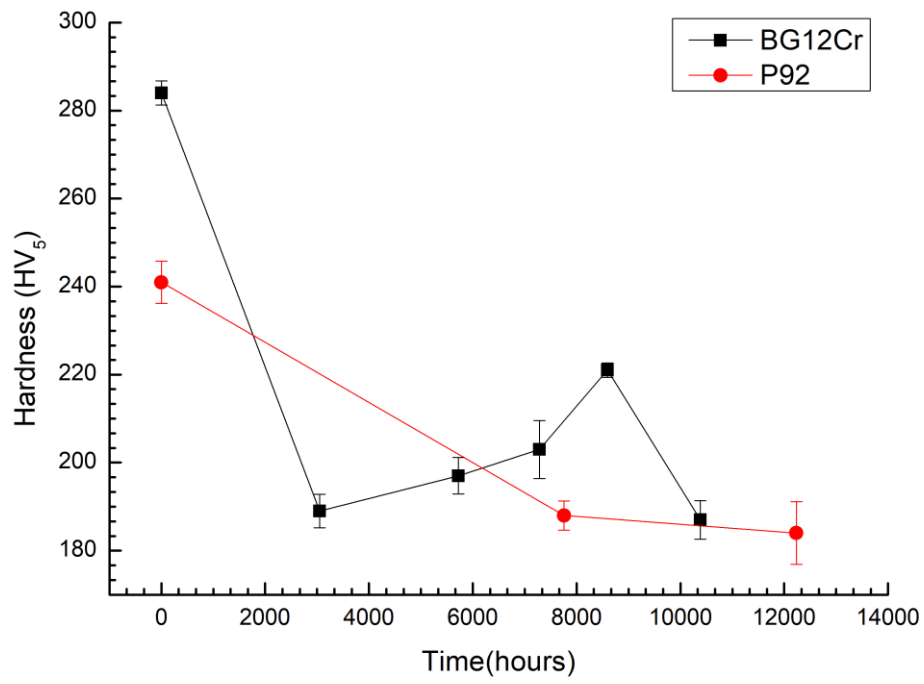


Figure 4.5 Hardness of both alloys at as-received condition (starting point) and after different periods of high temperature exposure at 650°C.

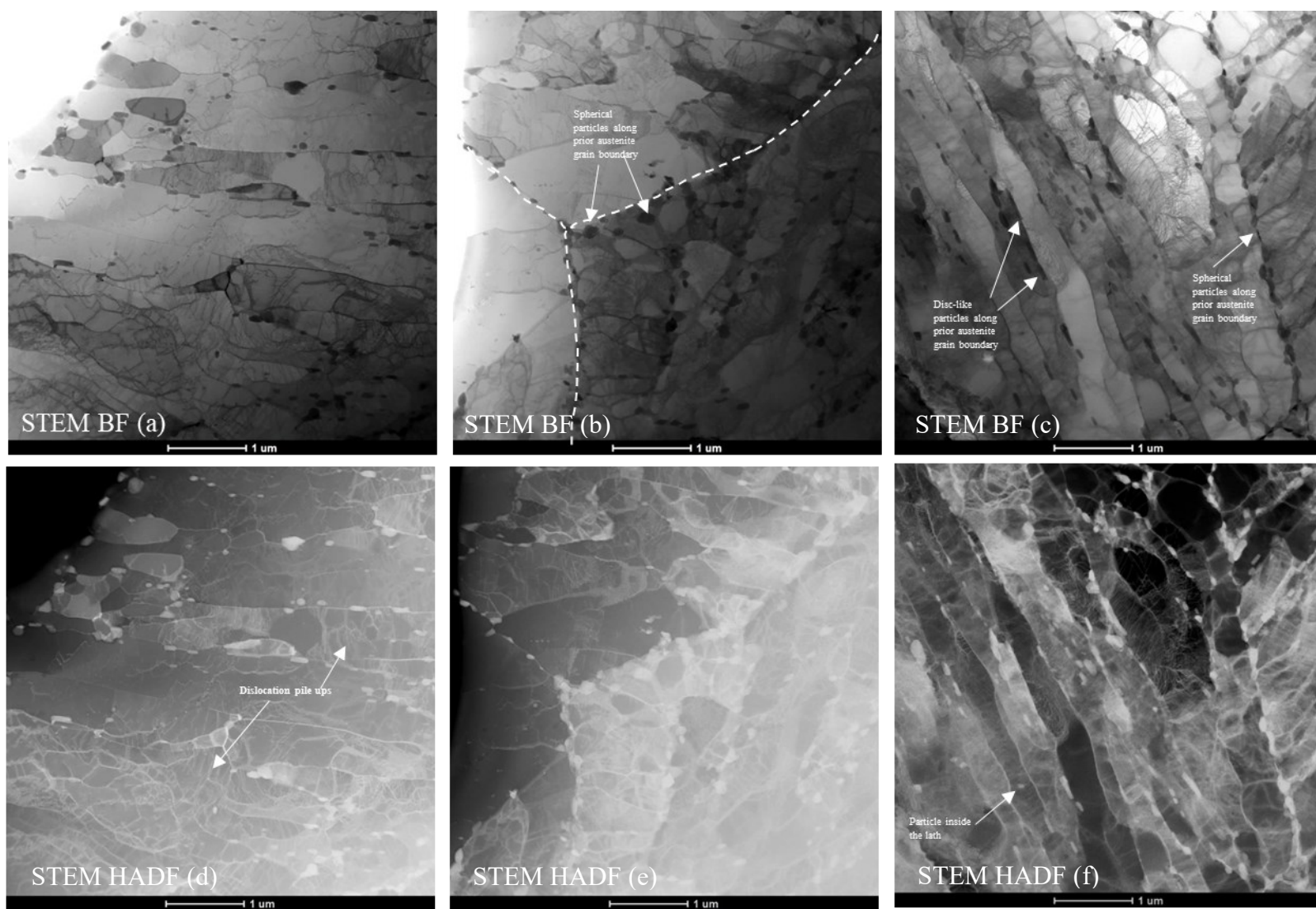


Figure 4.6 STEM images of P92 parent metal, (a)-(c) BF images, (d)-(f) HADF images (assisted by Rengeng Ding).

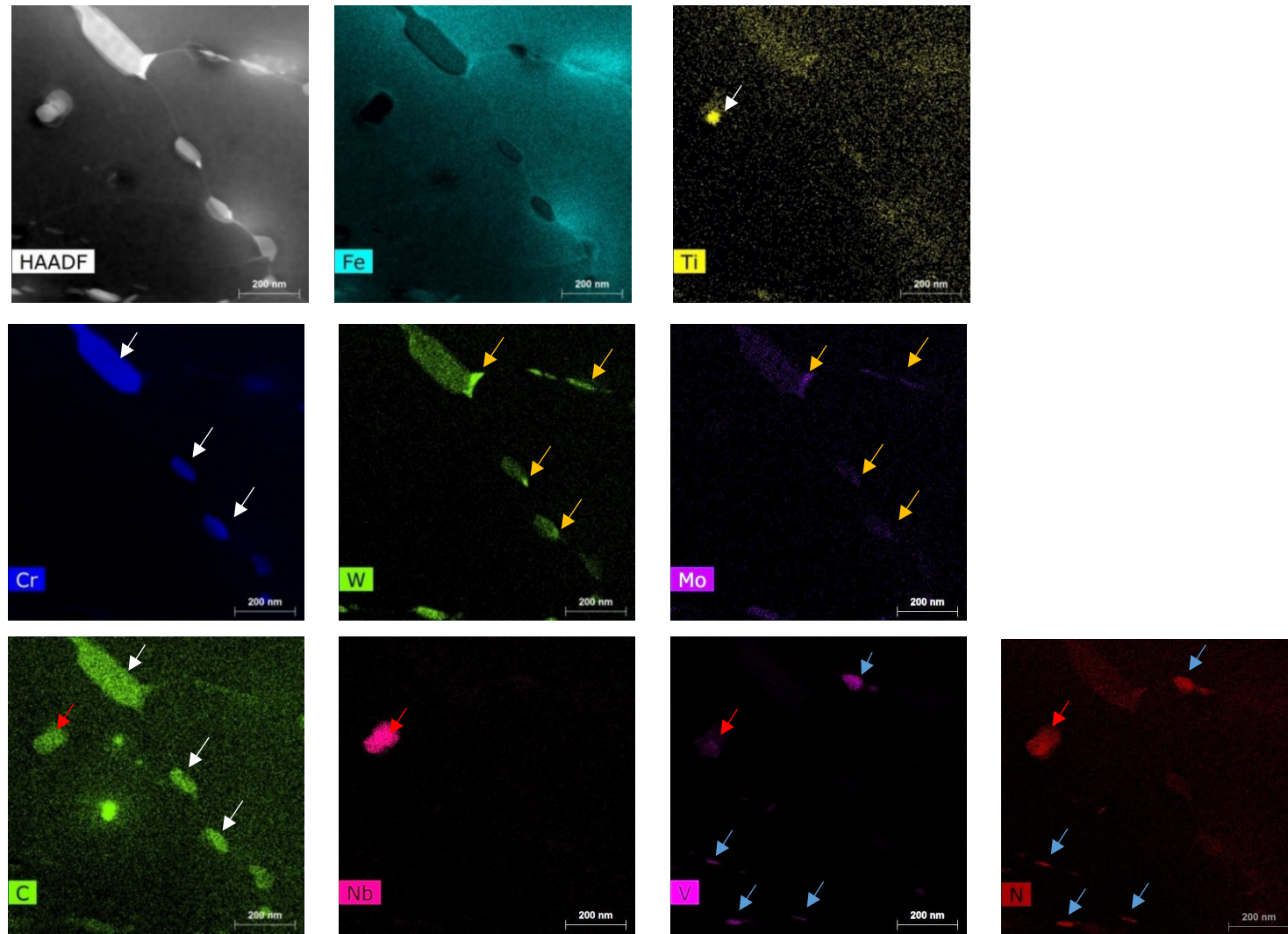


Figure 4.7 EDS maps for precipitates found in P92 as-received material showing (Cr,Mo,W)-rich carbides, (W,Mo)-rich intermetallics, Ti-rich carbonitrides, (Nb,V)-rich carbonitrides and V-rich carbonitrides (assisted by Rengeng Ding).

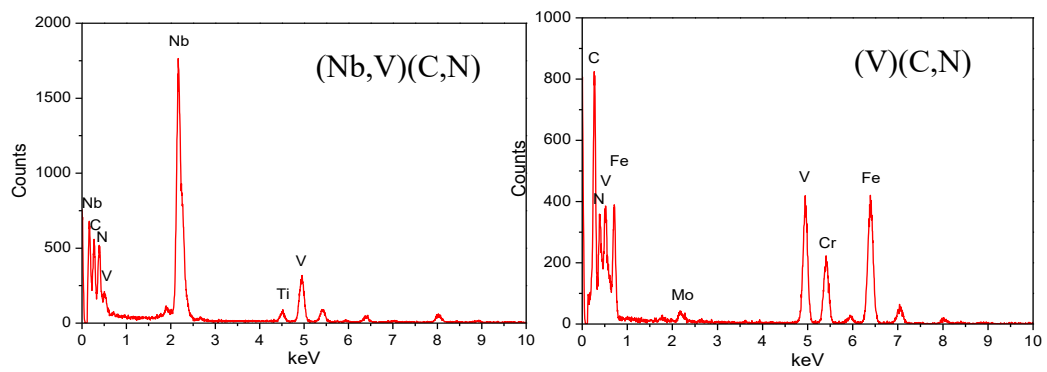


Figure 4.8 EDS spectra of typical precipitates in P92: $(\text{Nb,V})(\text{C,N})$ and $\text{V}(\text{C,N})$

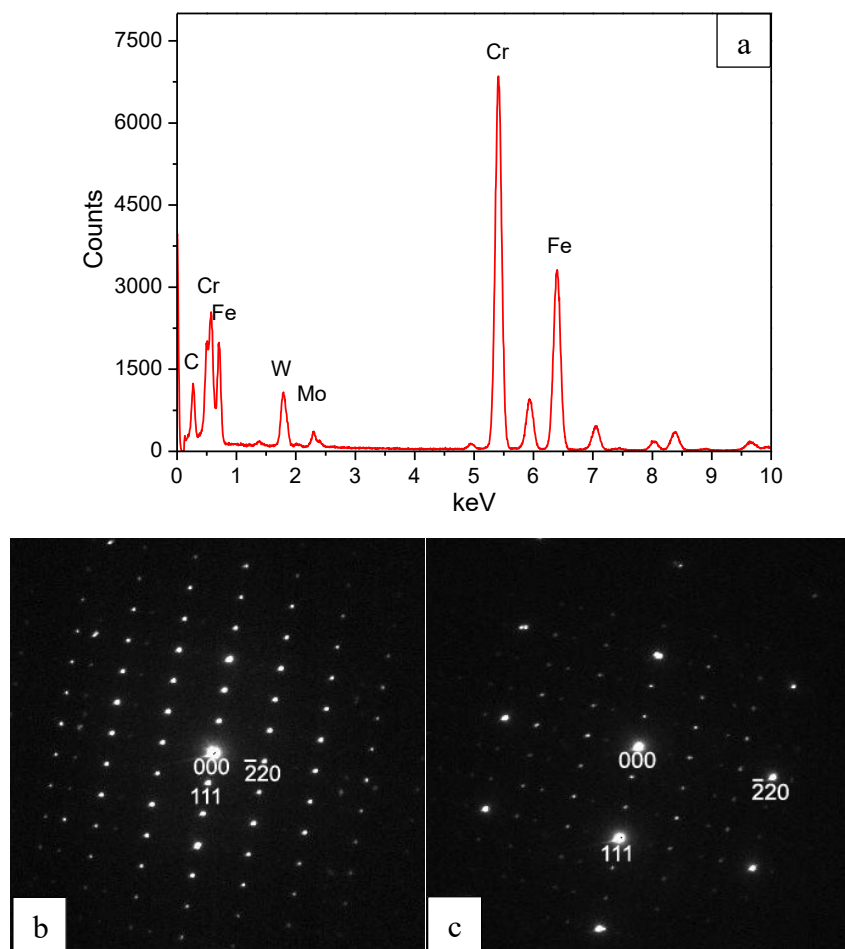


Figure 4.9 (a) EDS spectrum from M_{23}C_6 carbide in P92 as-received material, (b) SAD of $[11-2]$ from M_{23}C_6 , (c) Composite SADs of $[11-2]$ from M_{23}C_6 + matrix (assisted by Rengeng Ding).

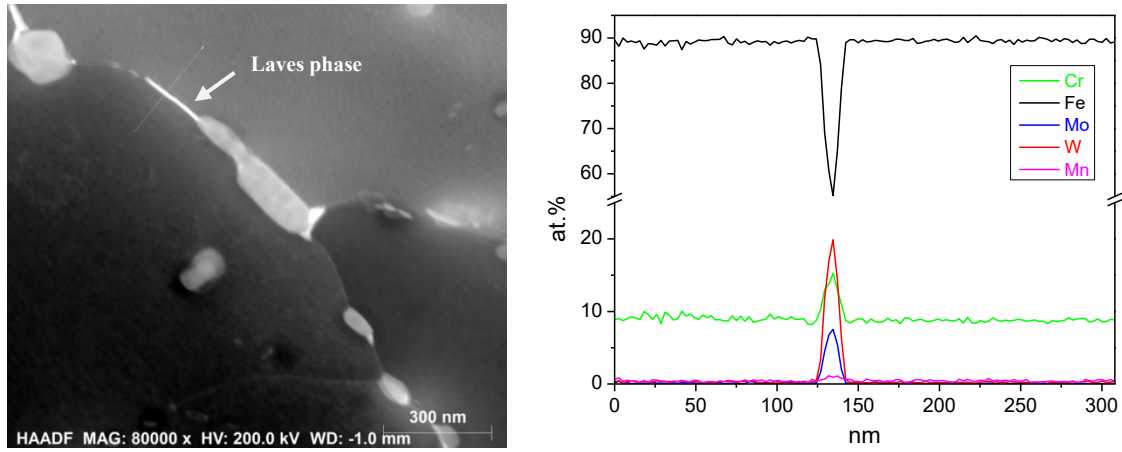


Figure 4.10 HAADF image of Laves phase along a GB and EDS linescan across the Laves phases (assisted by Rengeng Ding).

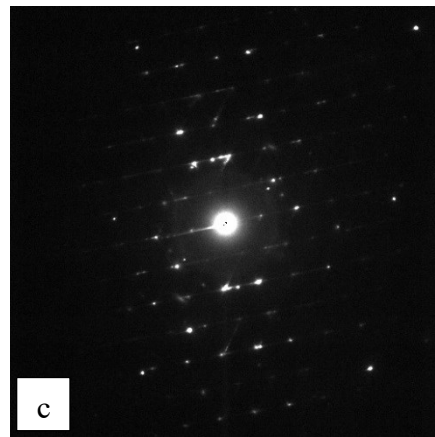
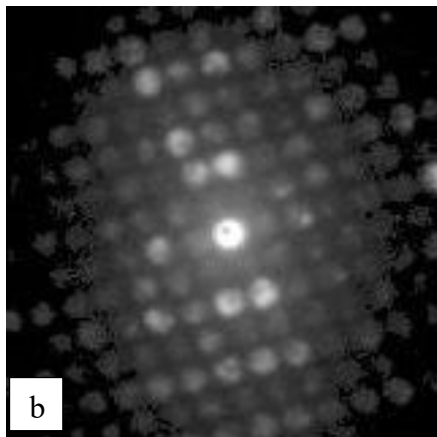
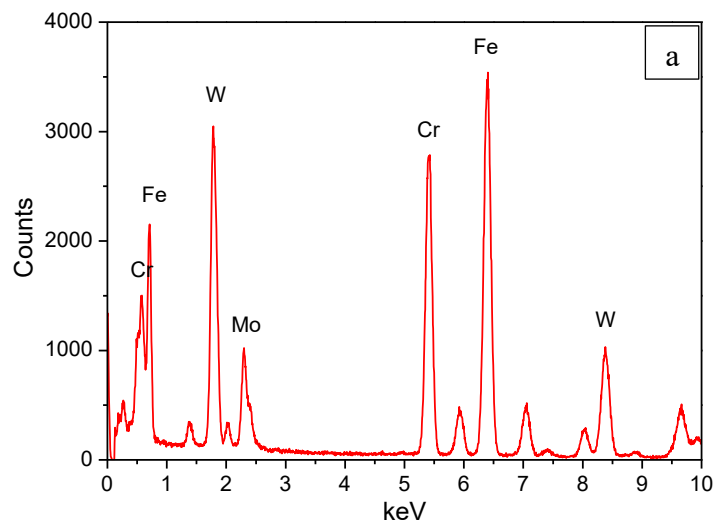


Figure 4.11 CBED and SAD of Laves phase and EDS spectrum from Laves phase (at. %:51.7Fe,13.4Cr, 20.2W, 13.1Mo, 0.8Mn,0.8Co) (assisted by Rengeng Ding).

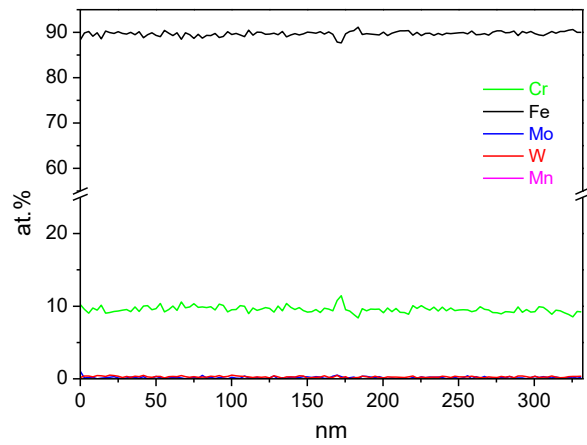
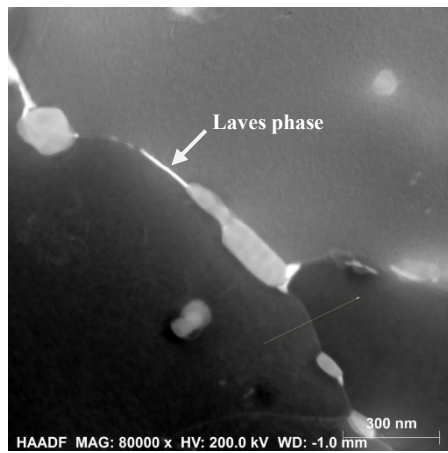


Figure 4.12 HAADF image of Laves phase along a GB and element profiles along the line shown in the image showing Cr enhancement at GB (assisted by Rengeng Ding).

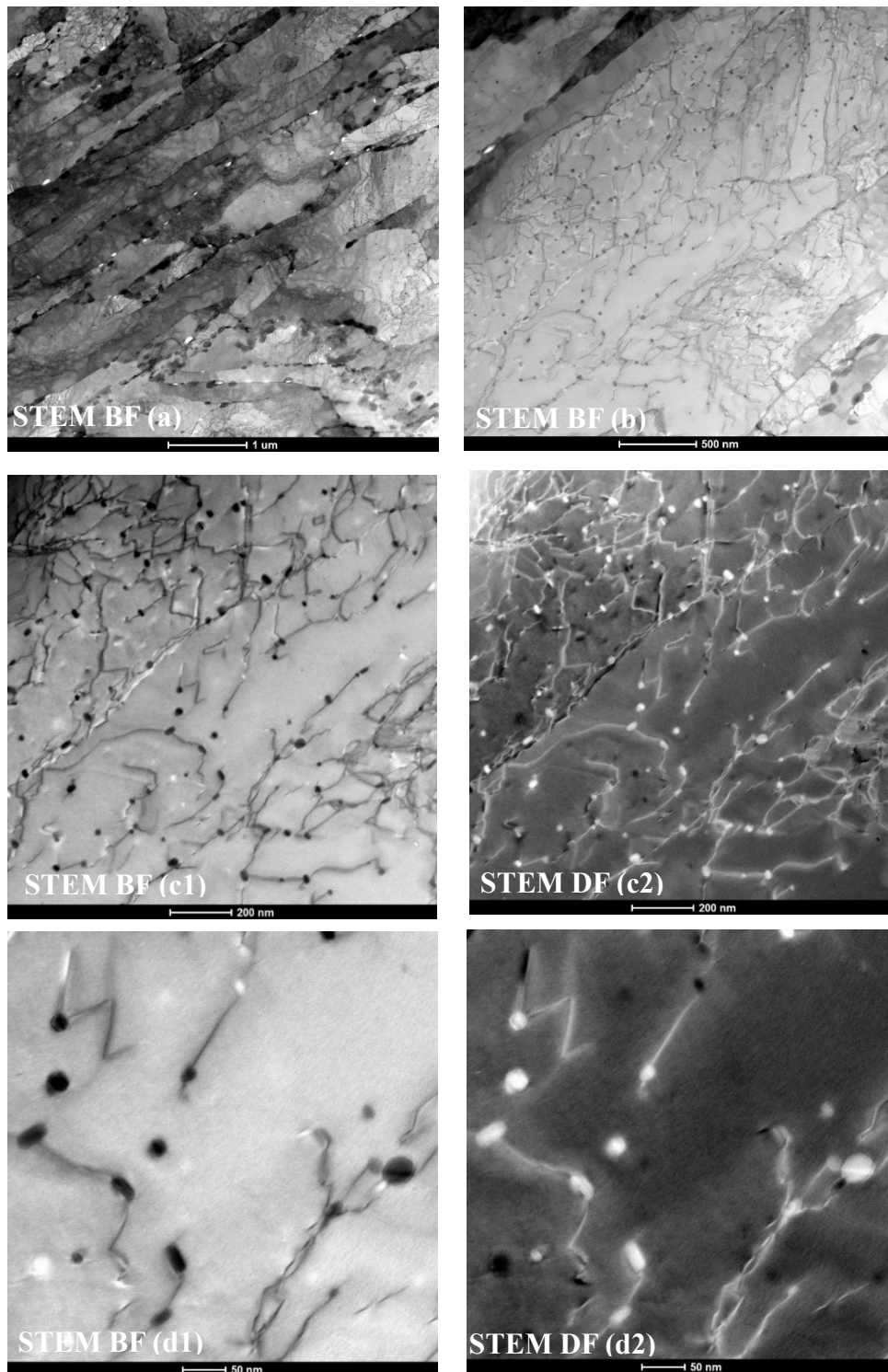


Figure 4.13 STEM images of tempered BG12Cr parent metal, showing precipitates mainly decorated on lath boundaries (a), a high density of dislocations pinned by nano-sized particles within lath (b), and a higher magnification image of (c1) -(d2) (assisted by Rengeng Ding).

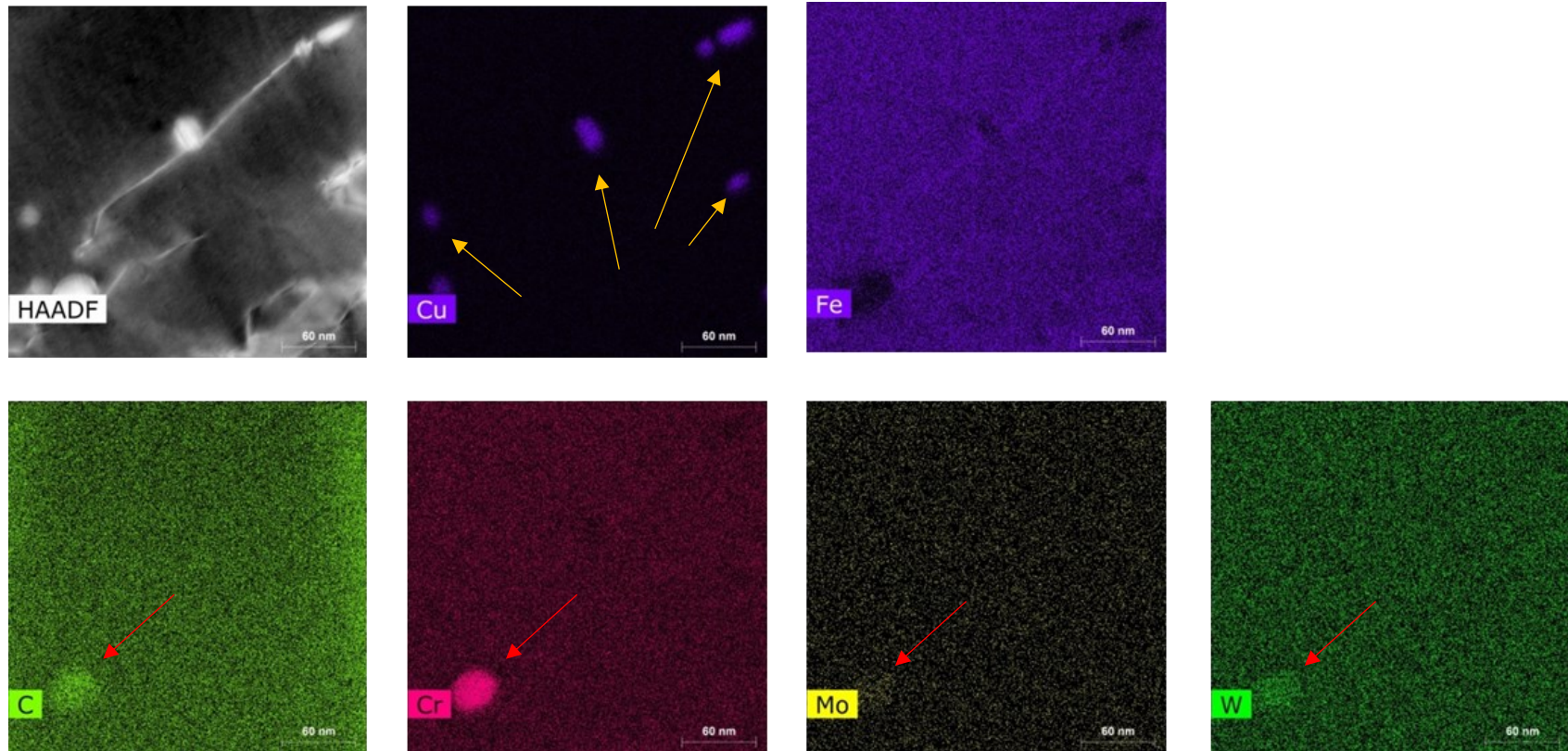


Figure 4.14 EDS of Cu-particles precipitated interior martensite lath showing pinning effect (assisted by Rengeng Ding).

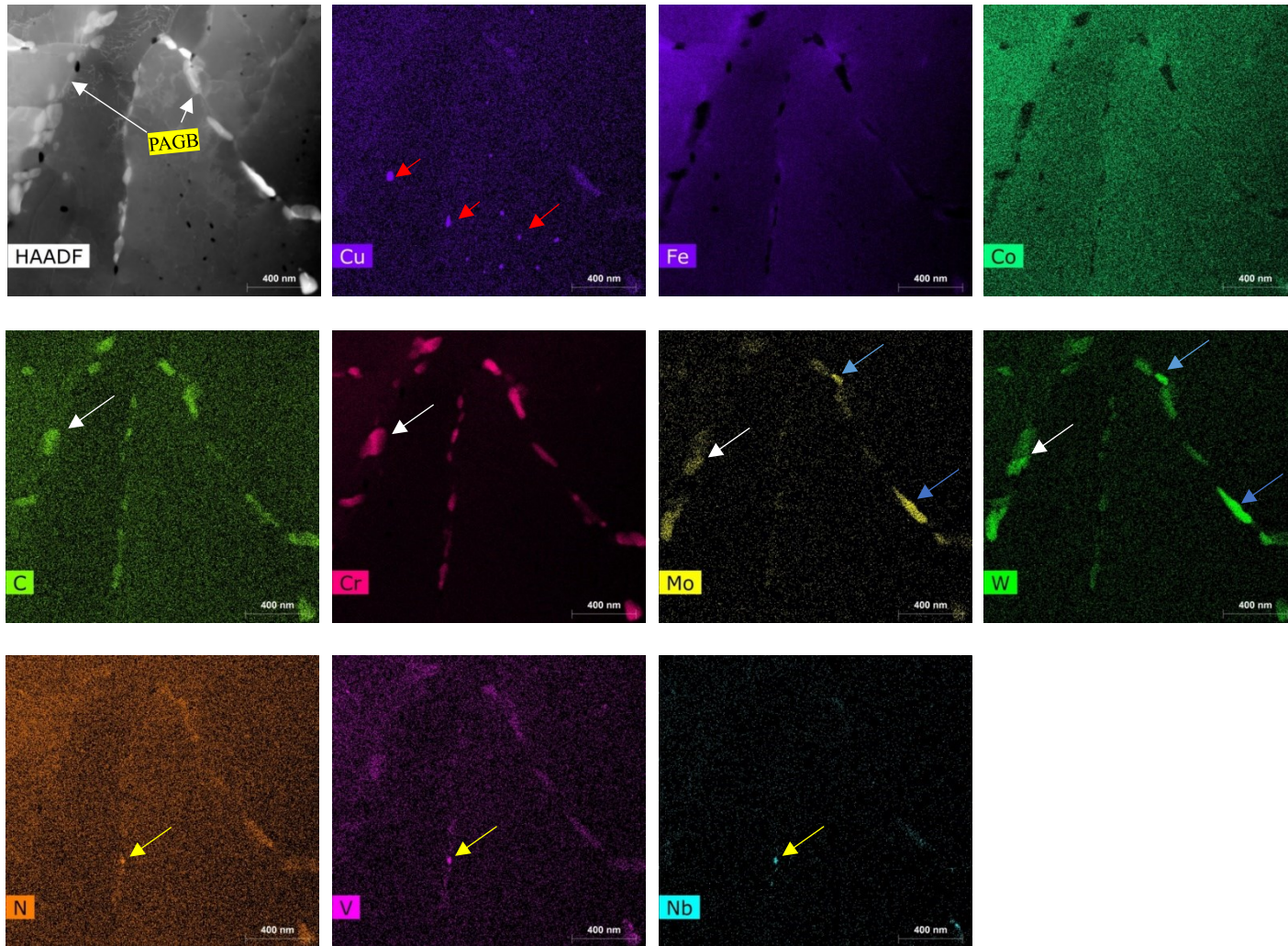


Figure 4.15 EDS mapping of precipitates in BG12Cr base material, showing Cu-rich precipitates in the interior of a grain. (Cr, Mo, W)-rich carbides at grain boundaries and (Nb, V)(C, N) precipitations at boundaries (assisted by Rengeng Ding).

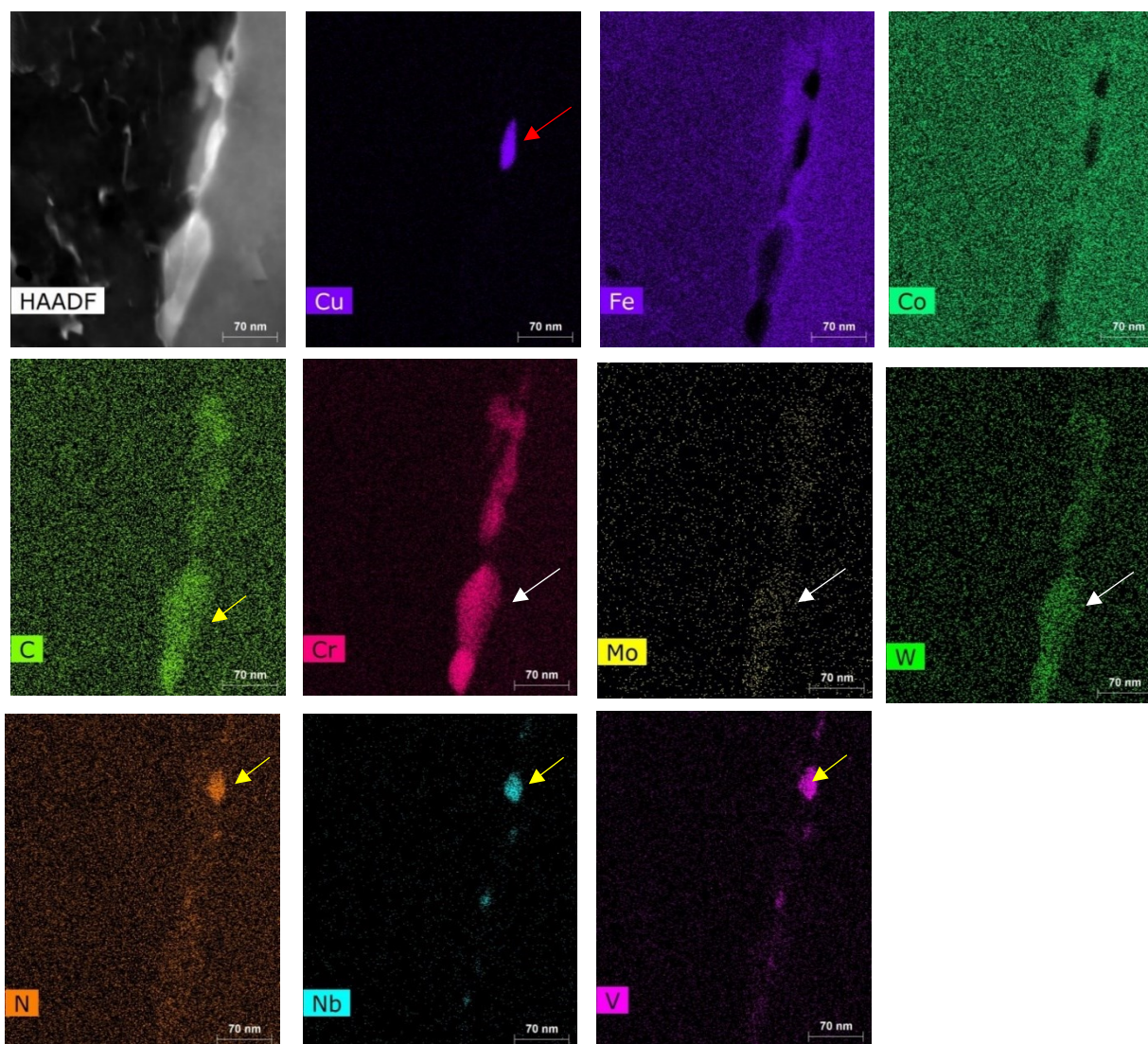


Figure 4.16 EDS maps of precipitates on a lath boundary indicating Cu-rich particle with ~50 nm and (Nb,V)-rich carbonitride with ~30 nm.

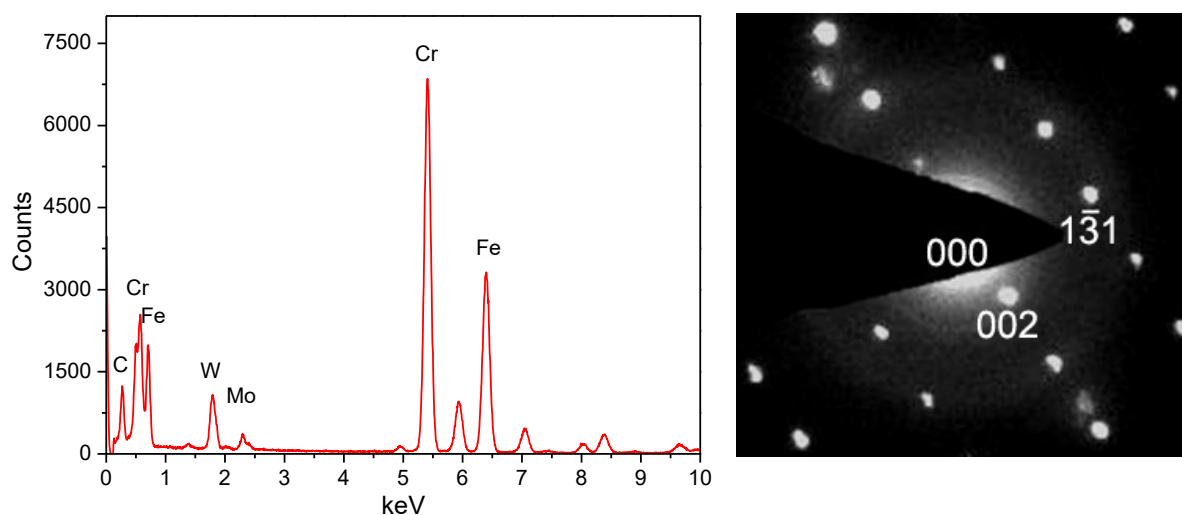


Figure 4.17 A typical EDS spectrum of a Cr-rich particle in BG12Cr and a SAD pattern from this particle of $[310]$ zone axis (assisted by Rengeng Ding).

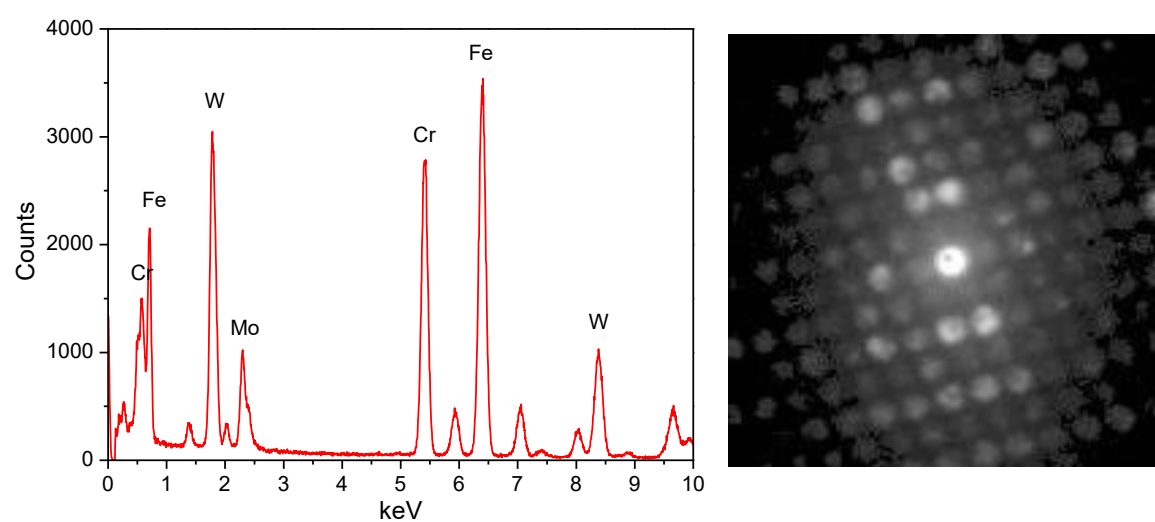


Figure 4.18 A typical EDS spectrum taken from a (W,Mo)-rich particle (a), a CBED pattern from the W-rich particle of $[\bar{1}2\bar{1}3]$ zone axis (b) (assisted by Rengeng Ding).

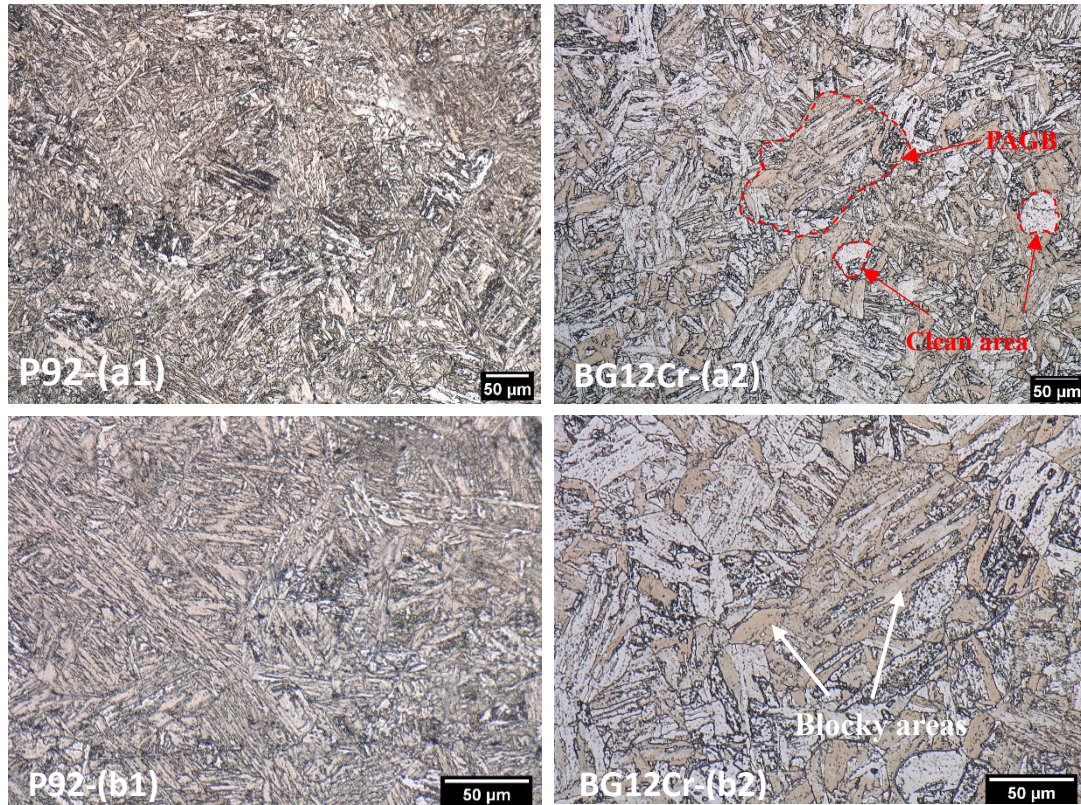


Figure 4.19 Optical micrographs taken from the undeformed regions of two long-term stress-ruptured P92-E and BG12Cr-E testpieces ($>10^4$ hours), (a1)-(a2): low magnified view, (b1)-(b2): high magnified view.

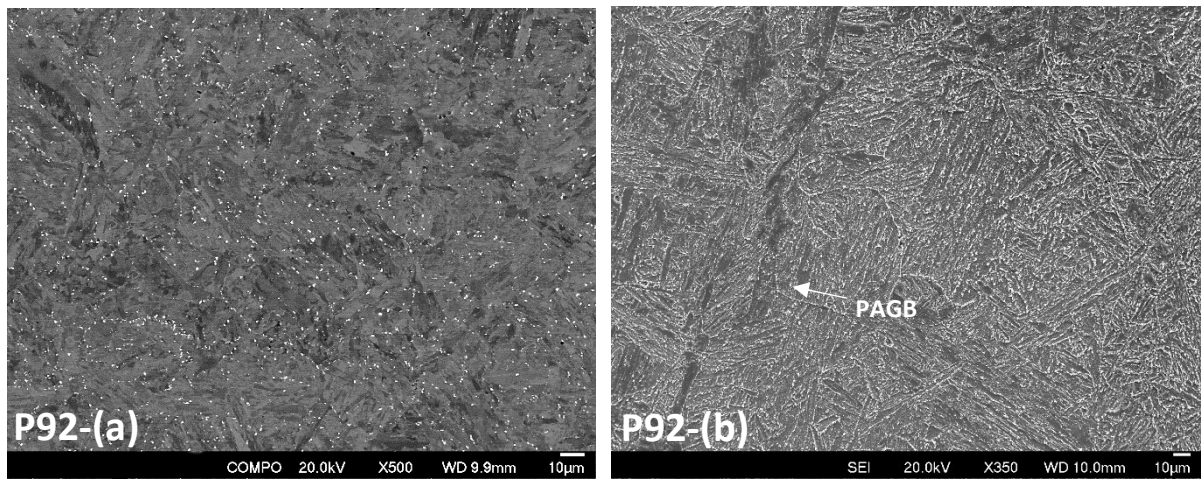


Figure 4.20 SEM micrographs from the undeformed regions of two long-term stress ruptured P92 testpieces (12234 hours).

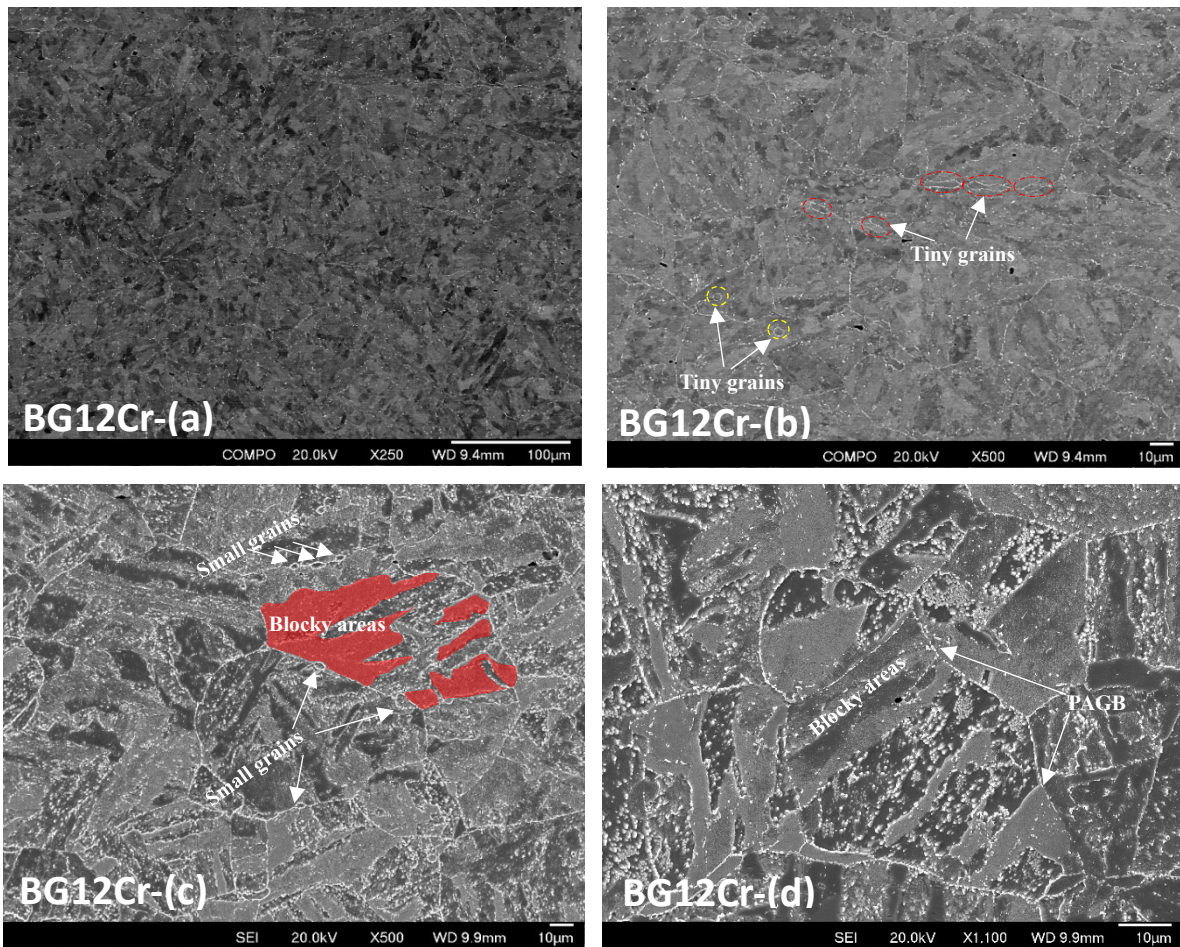


Figure 4.21 SEM micrographs from the undeformed regions of two long-term stress ruptured BG12Cr testpieces (10380 hours).

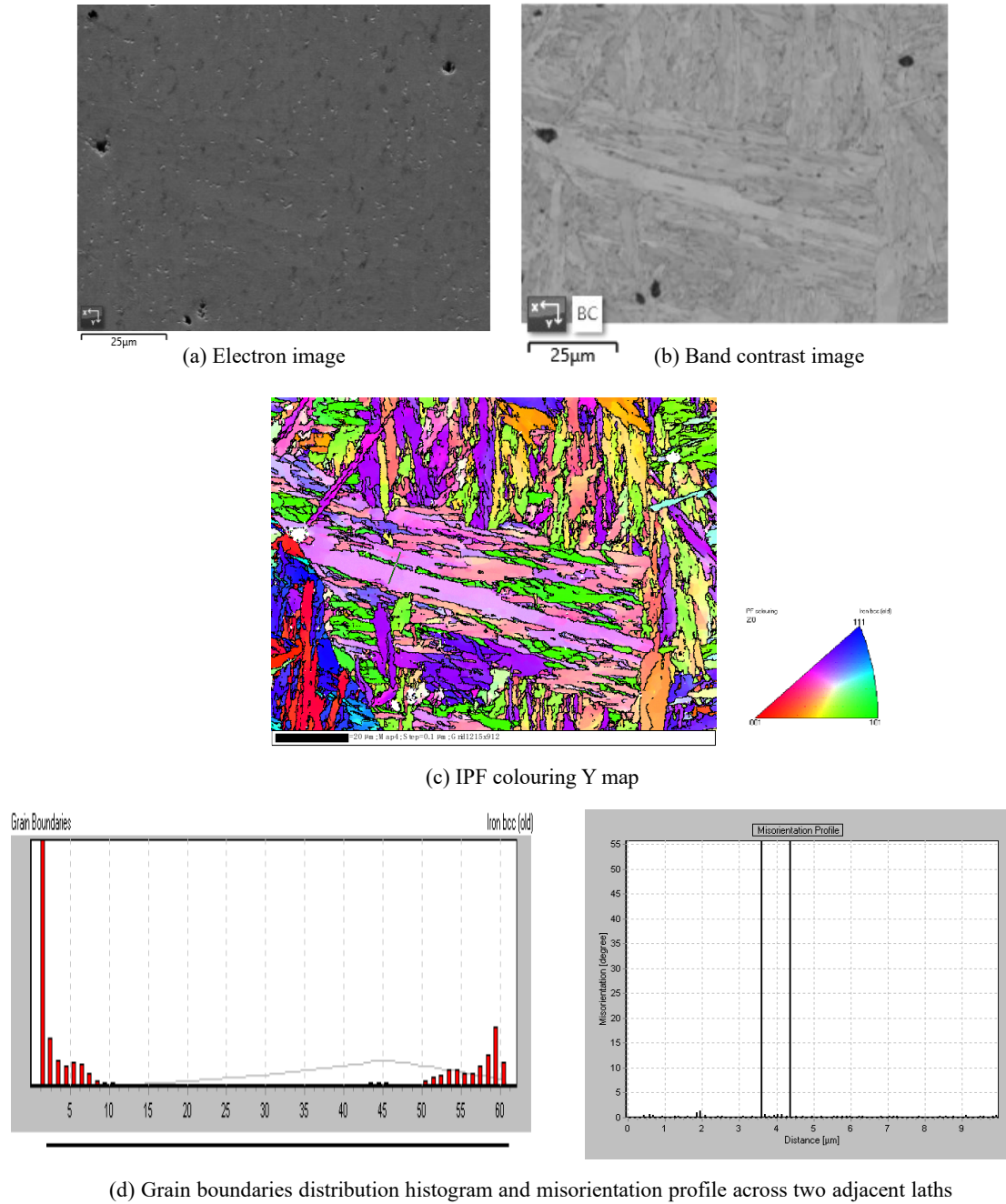


Figure 4.22 EBSD scanning image of P92 PT material: (a) Electron image; (b) Band contrast image; (c) IPF colouring Y image; (d) Grain boundary distribution histogram and misorientation profile across two adjacent laths.

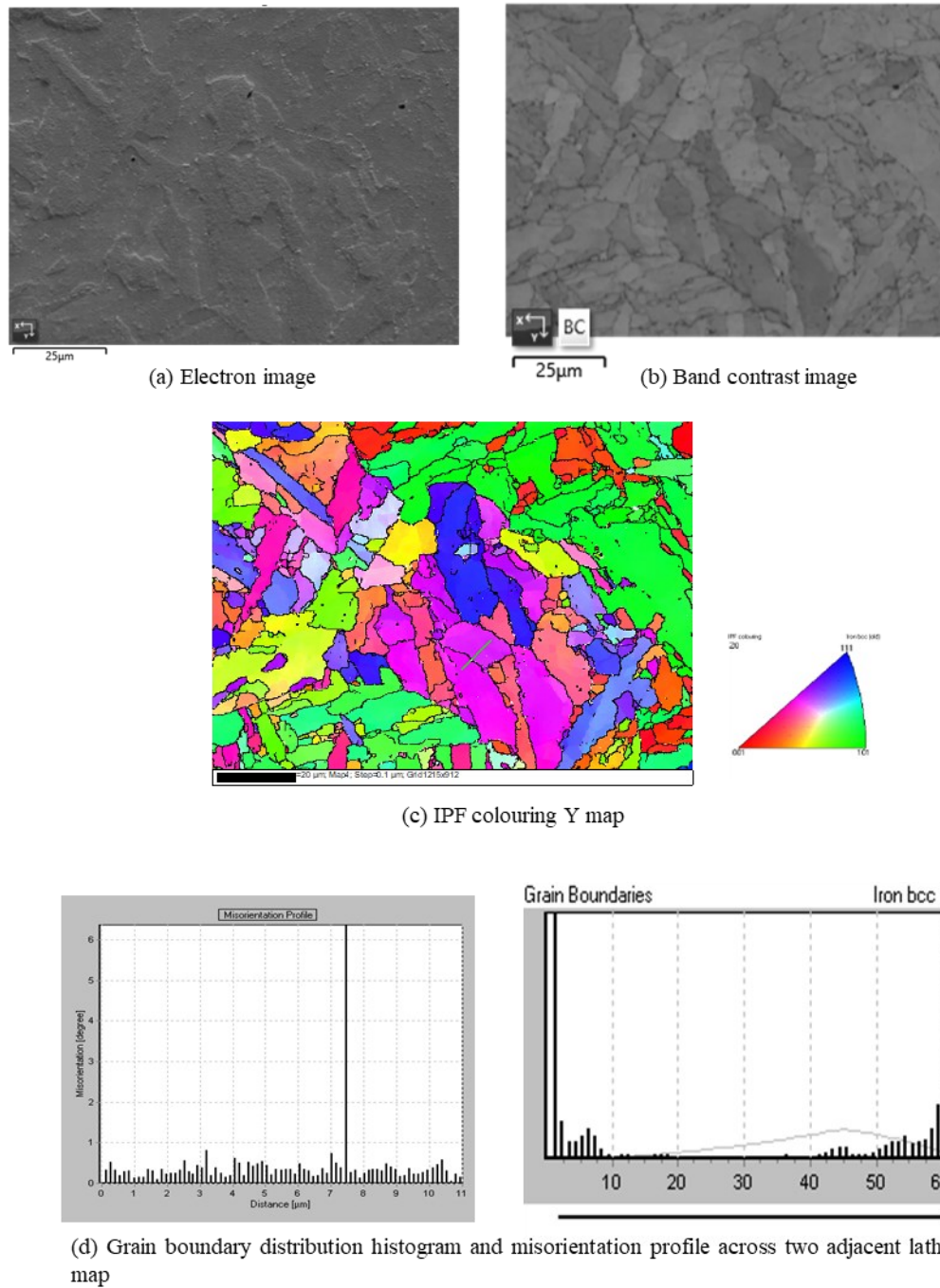


Figure 4.23 EBSD scanning image of BG12Cr PT material: (a) Electron image; (b) Band contrast image; (c) IPF colouring Y image; (d) Grain boundary distribution histogram and misorientation profile across two adjacent laths.

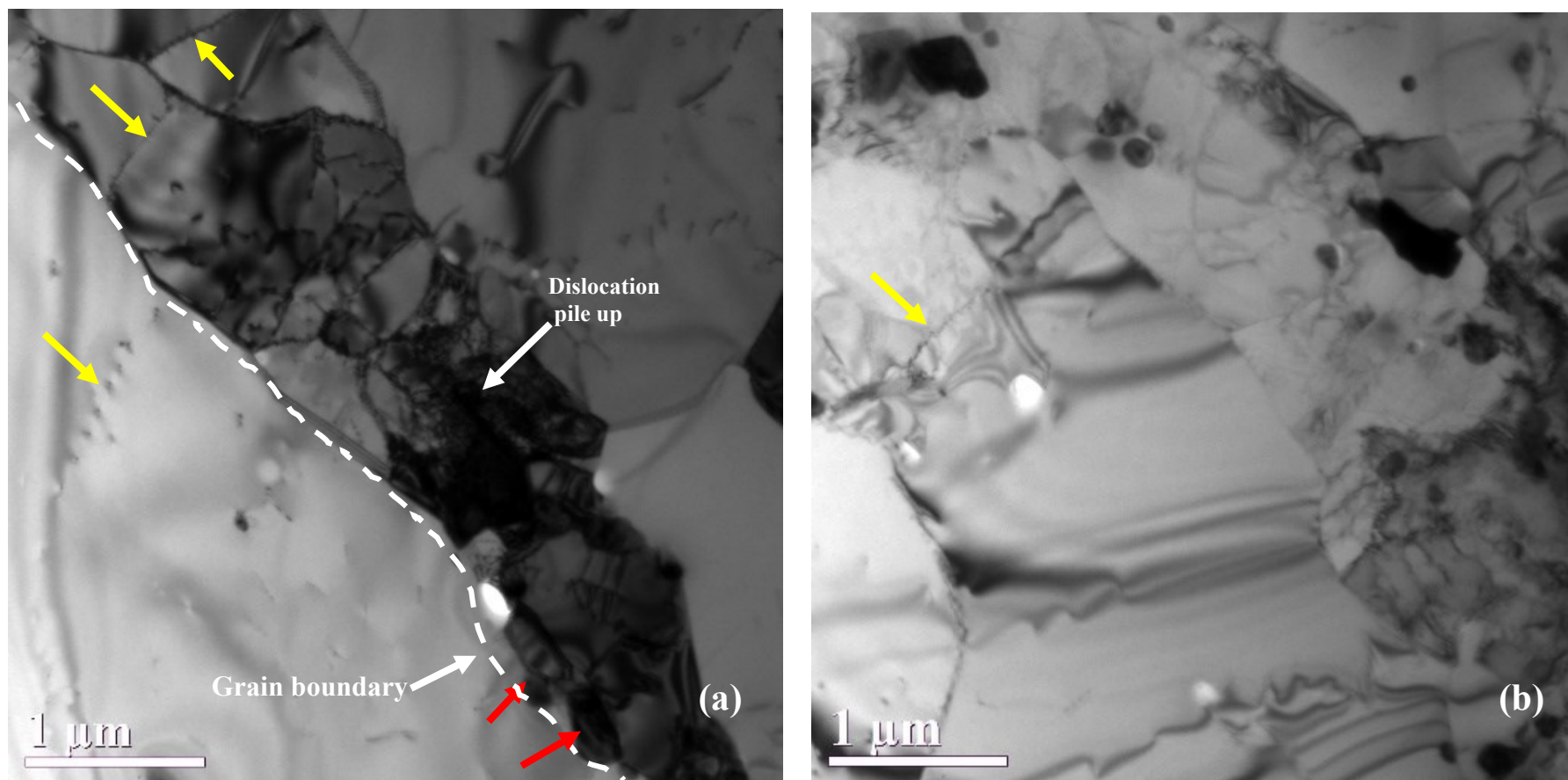


Figure 4.24 TEM bright field (BF) micrographs of PT BG12Cr (assissted by Dr. Hiroto).

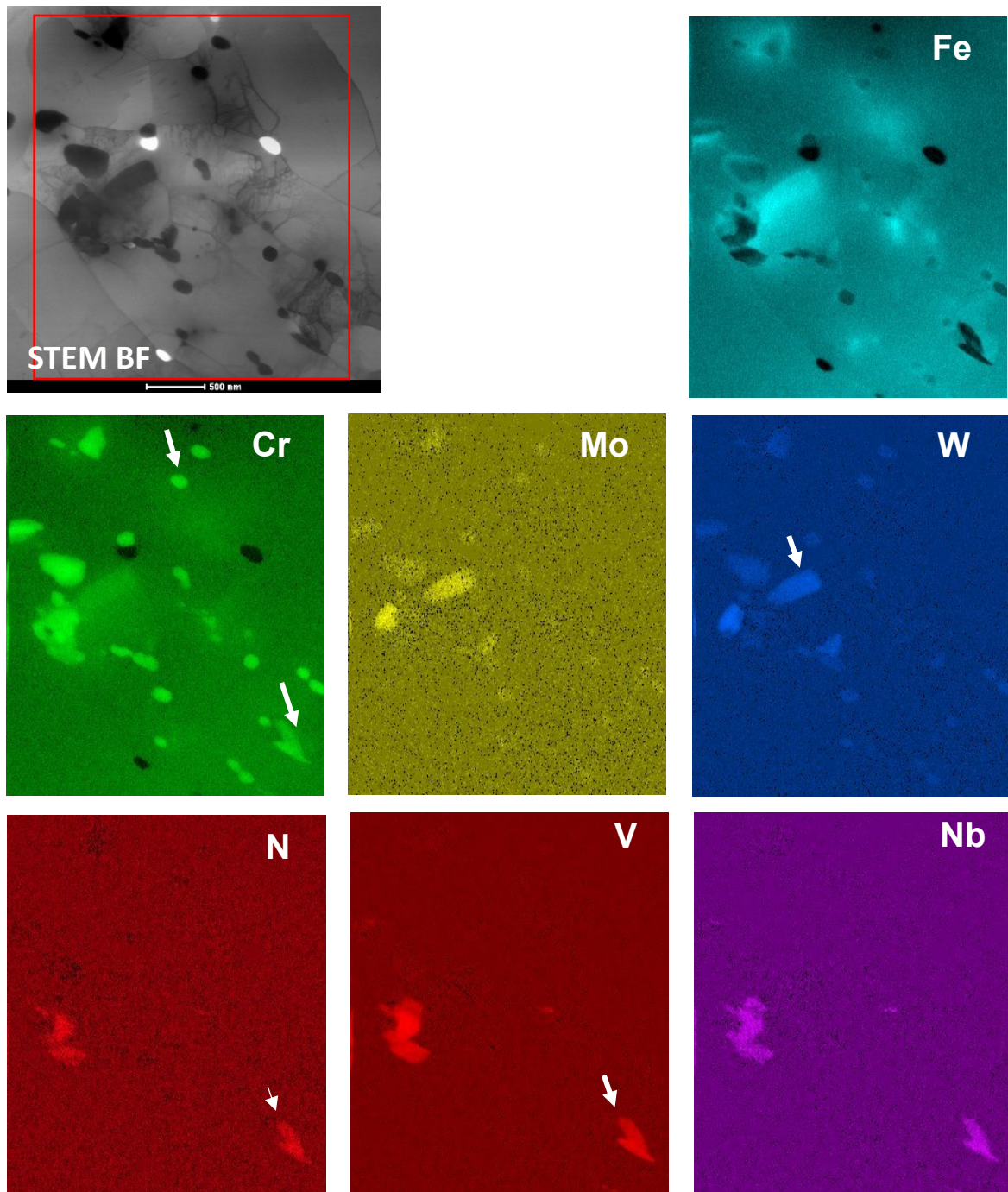


Figure 4.25 EDS mapping micrographs of PT BG12Cr general microstructure and chemistry including precipitates from particle populated area (boundaries included) (assisted by Dr. Hiroto).

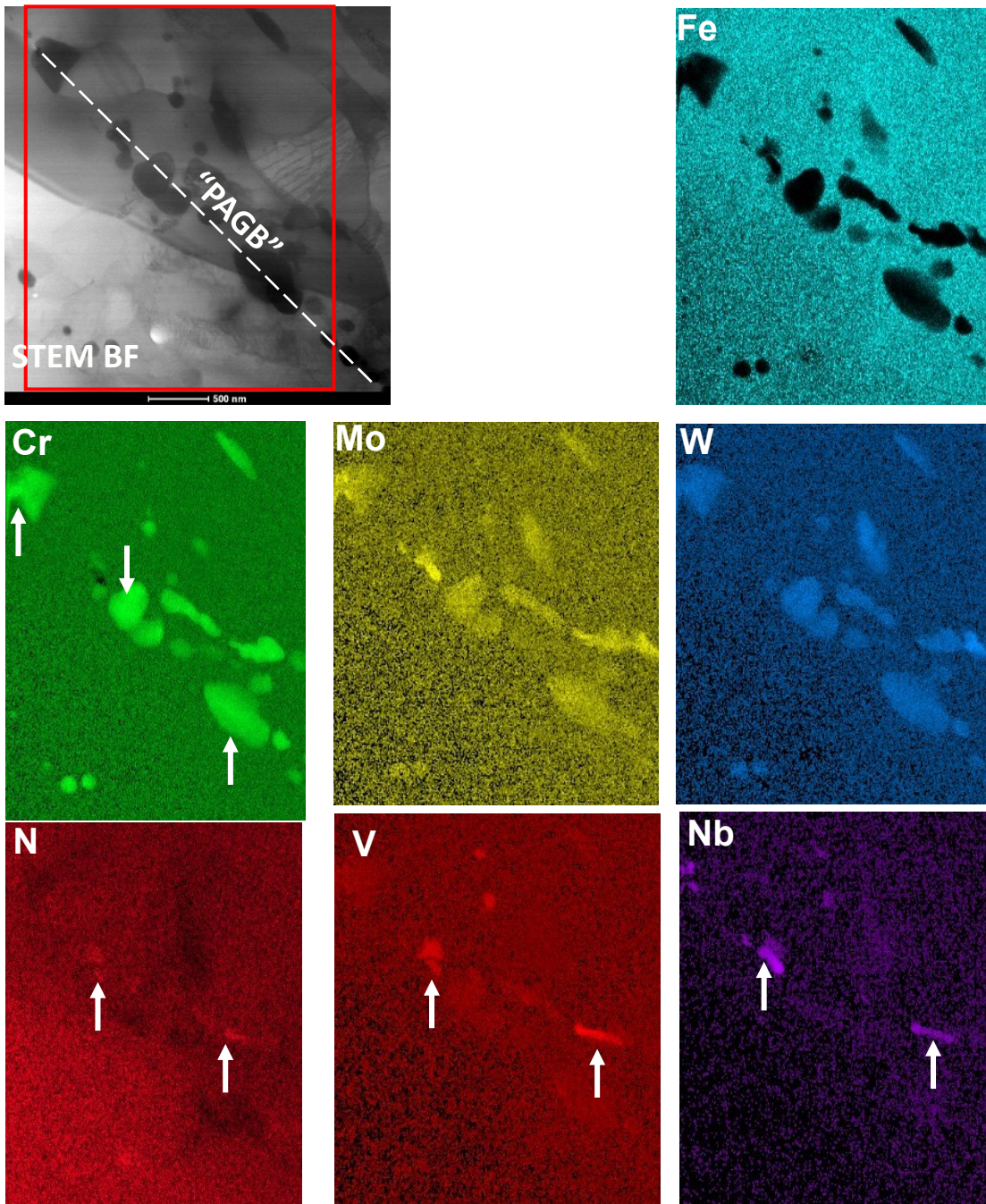


Figure 4.26 EDS mapping micrographs of PT BG12Cr general microstructure and chemistry including precipitates from a PAGB disappearing area (assisted by Dr. Hiroto).

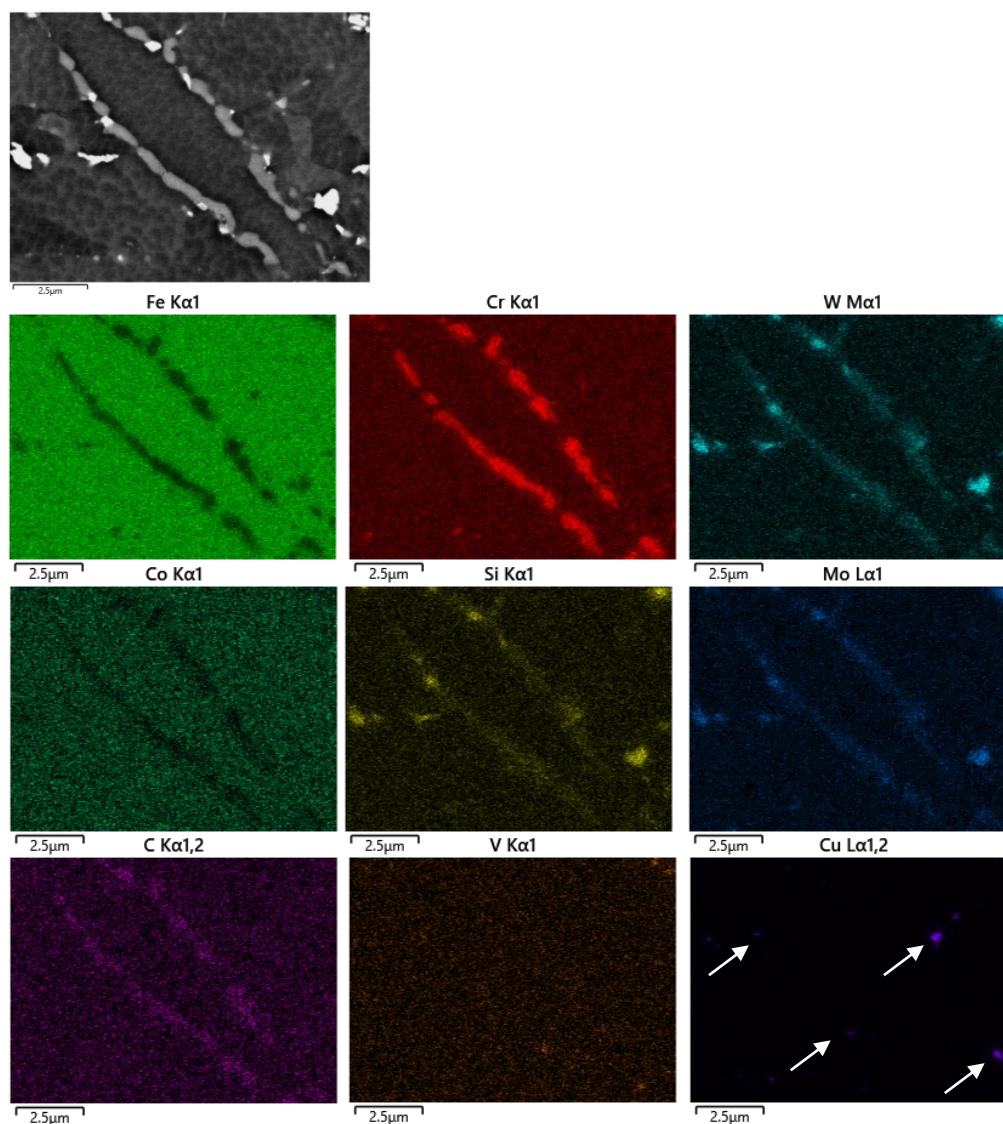


Figure 4.27 EDS mapping micrographs of tiny grains along PAGBs in PT BG12Cr showing precipitates along the grain boundary, indicating Cu-rich particles interior the grain (assisted by Dr. Hiroto).

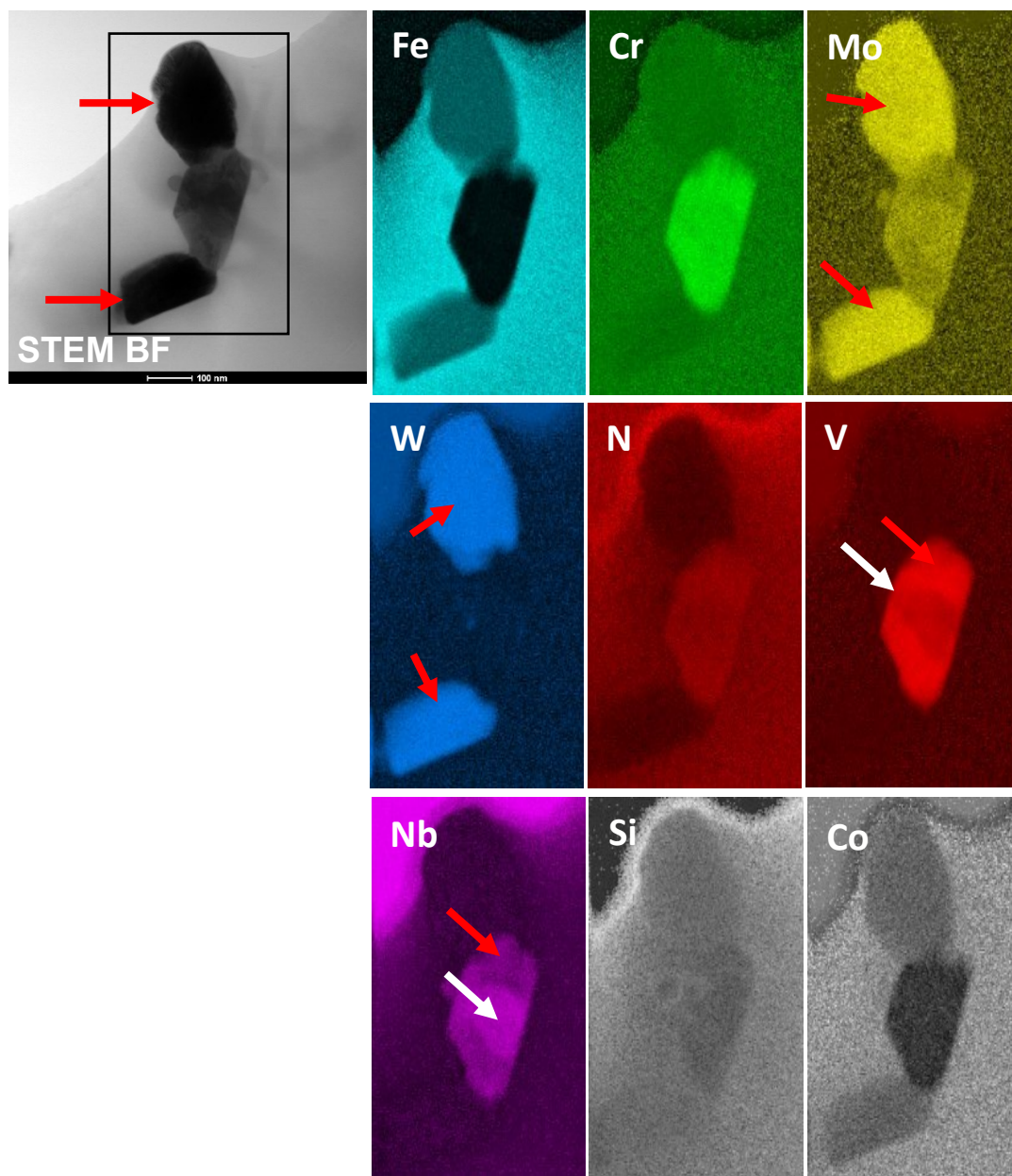


Figure 4.28 STEM EDS micrographs of (W,Mo)-rich particles (Laves phase) and (V,Nb)-rich particles (Z-phase) (assisted by Dr. Hiroto).

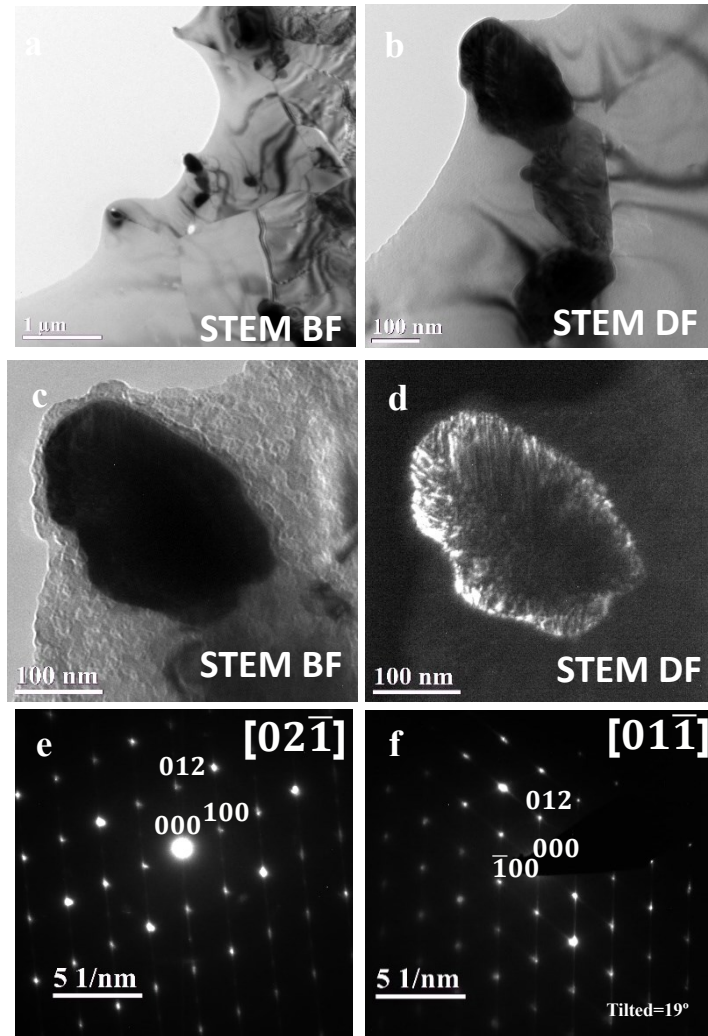


Figure 4.29 TEM bright field (BF) and dark field (DF) micrographs of Laves phase in PT BG12Cr: a: low magnification, b-d: high magnification, e-f: SAD profiles and EDS spectra of Laves phase with matrix (assisted by Dr. Hiroto).

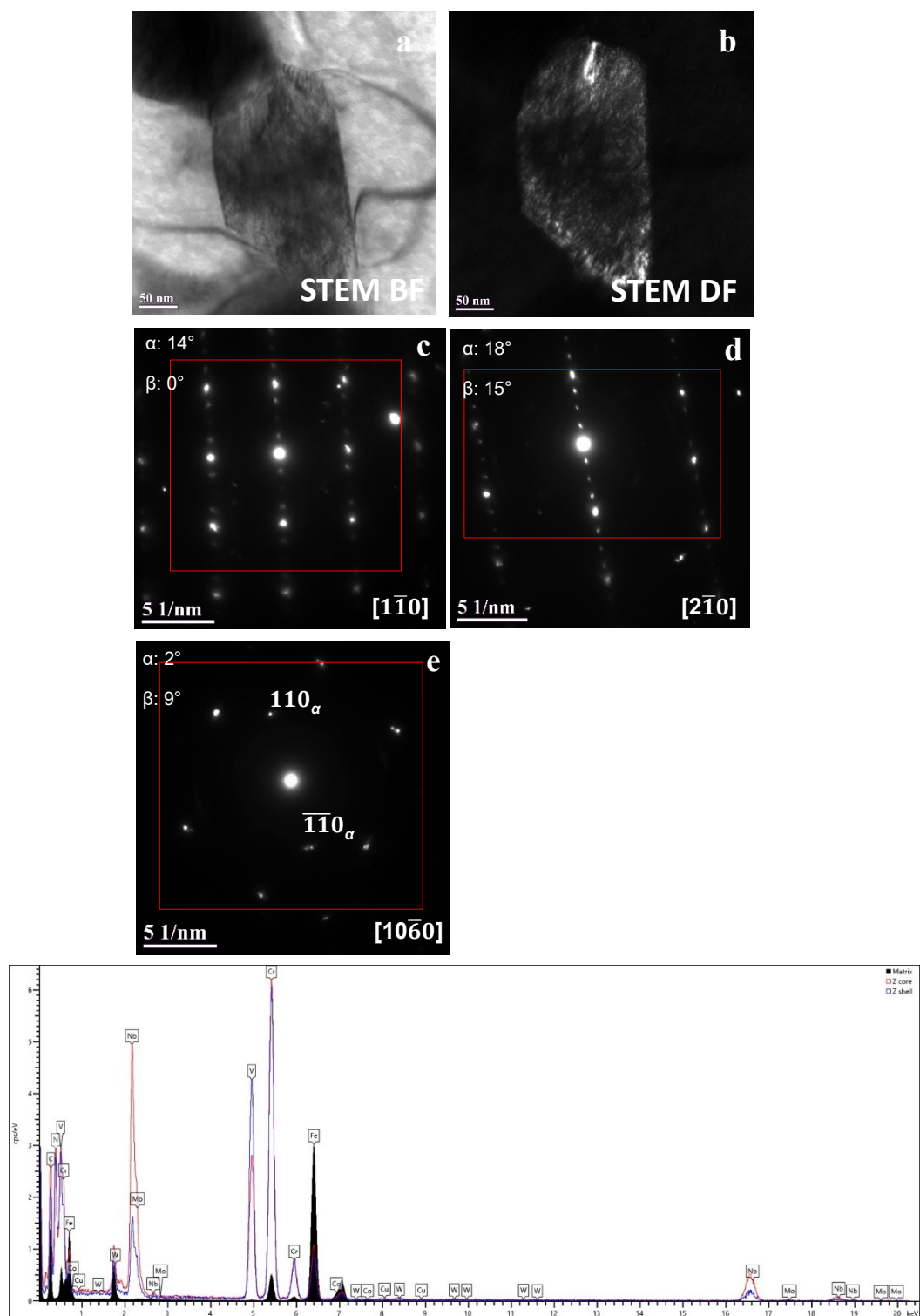


Figure 4.30 STEM bright field (BF) and dark field (DF) micrographs of Z-phase in PT BG12Cr (a-b), its SAD profiles (c-e) and EDS spectrum (assisted by Dr. Hiroto).

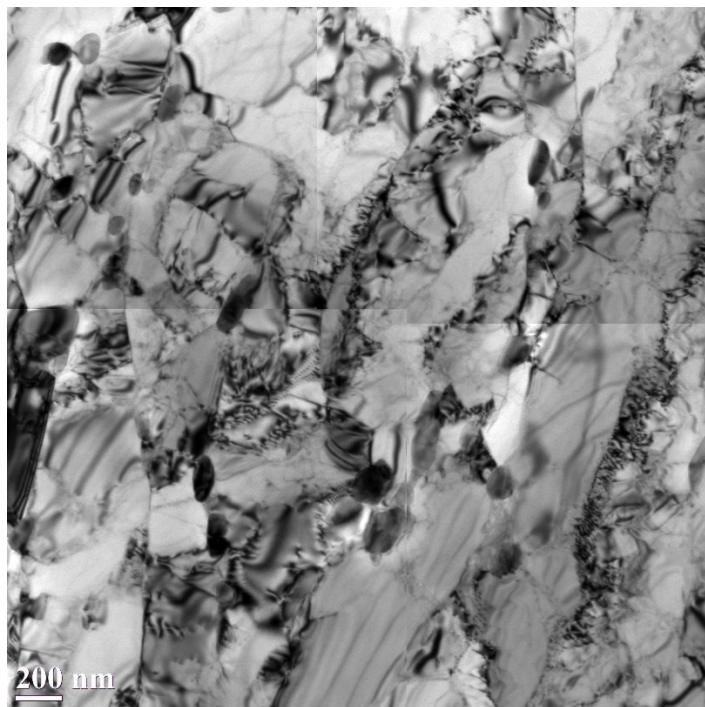


Figure 4.31 TEM bright field (BF) micrograph of PT P92 (assisted by Dr. Hiroto).

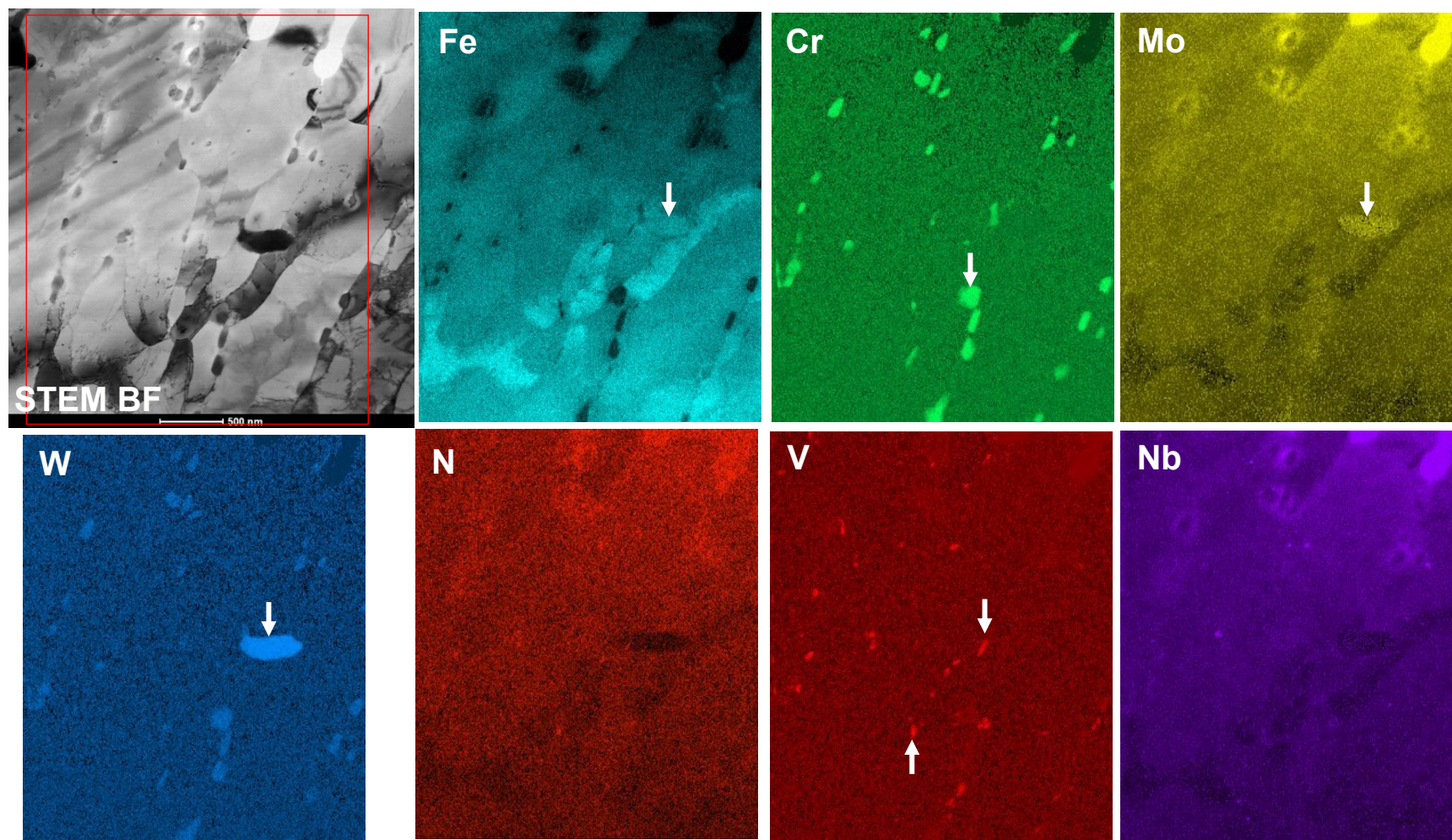


Figure 4.32 EDS mapping micrographs of a particles populated area in PT P92 indicating finer Cr-rich carbides and V-rich particles, coarsened (W,Mo)-rich precipitates (assisted by Dr. Hiroto).

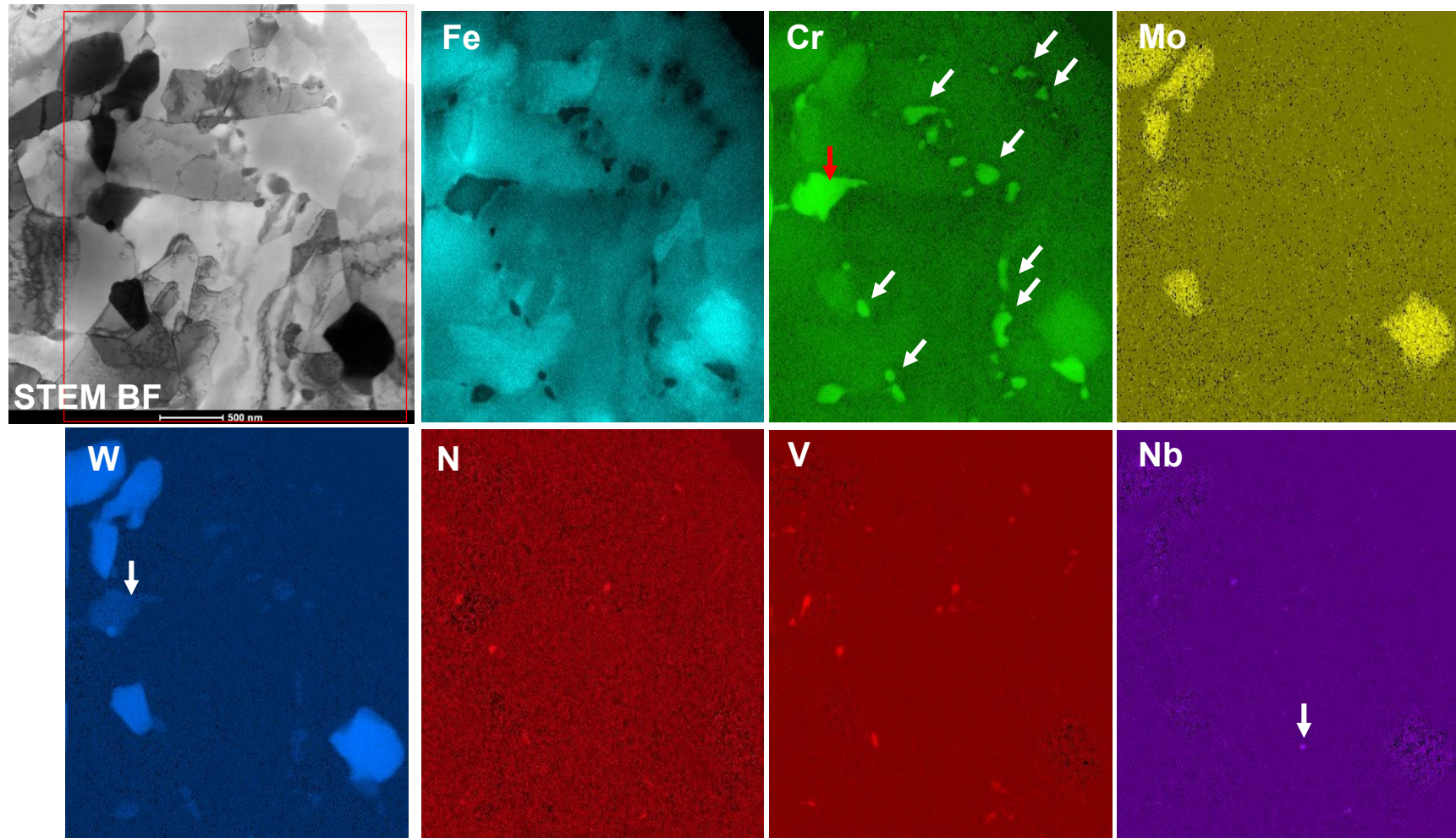


Figure 4.33 EDS mapping micrographs of a particles populated area in PT P92 indicating Cr-rich carbides along boundaries, fine V-rich particles, coarsened (W,Mo)-rich precipitates on the boundaries, and Nb-rich particles interior martensite matrix (assisted by Dr. Hiroto).

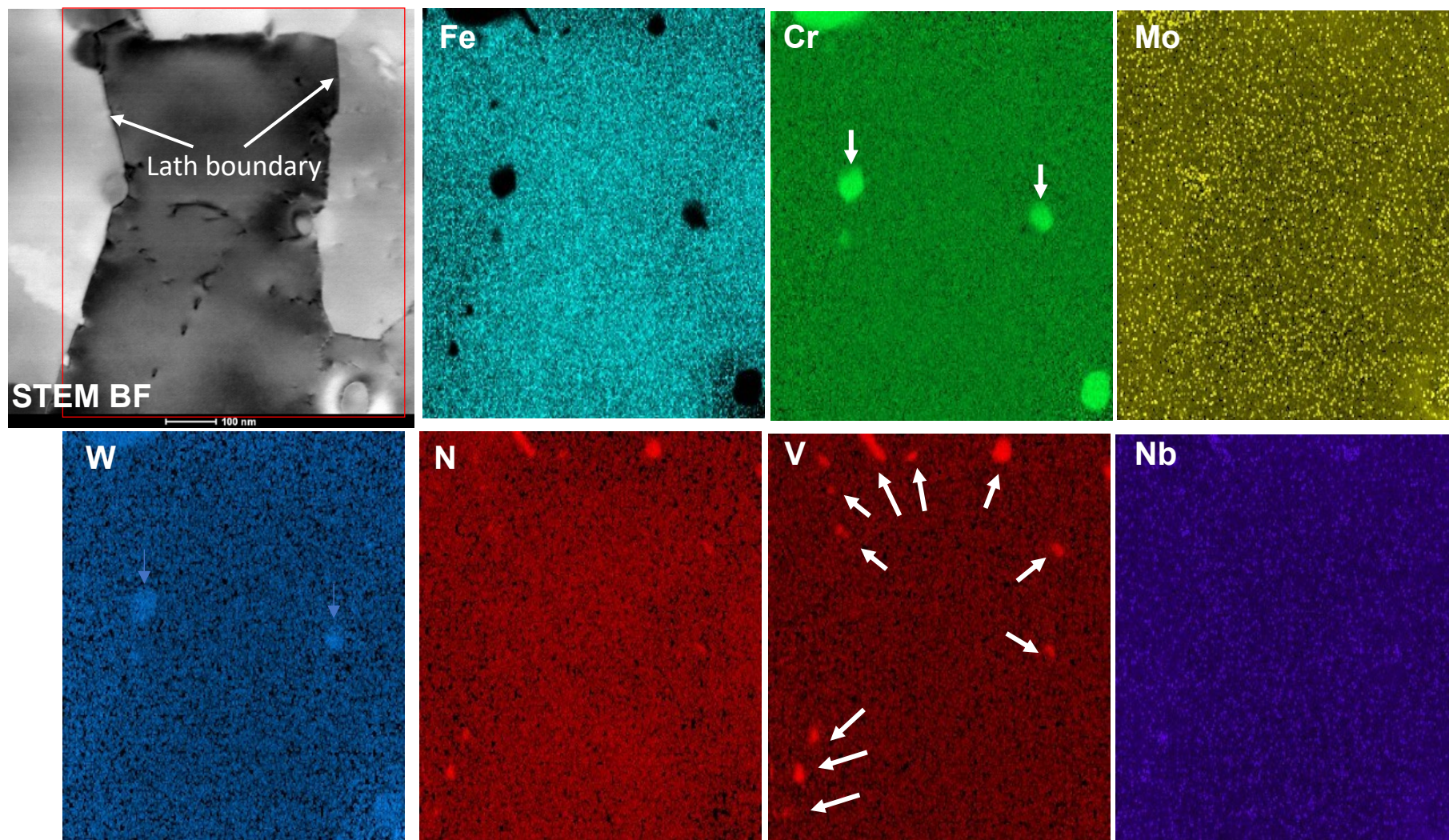


Figure 4.34 EDS mapping micrographs of pinning precipitates along lath boundary in PT P92 indicating fine distribution of V-rich particles pinning the lath boundary (assisted by Dr. Hiroto).

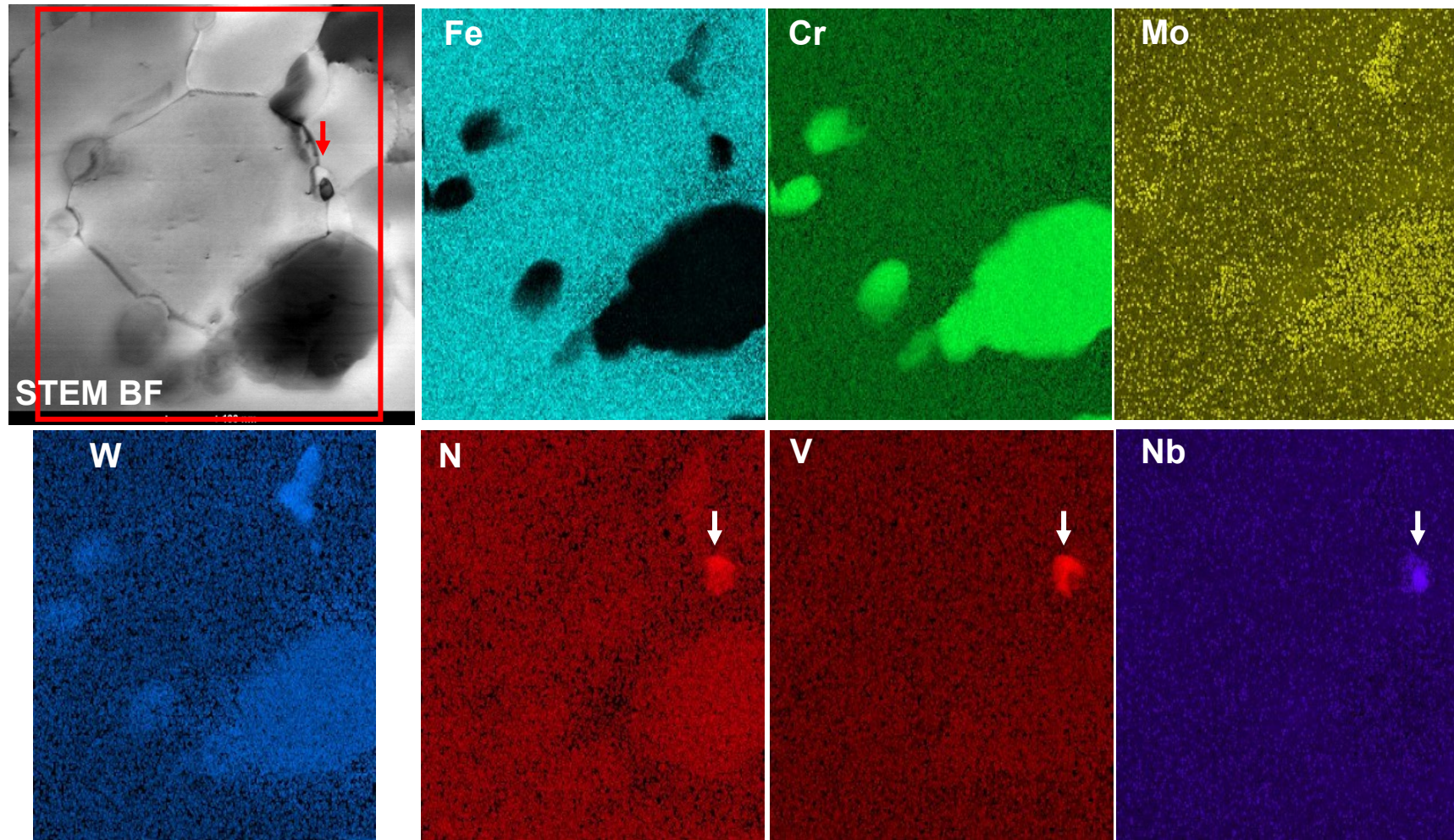


Figure 4.35 STEM bright field (BF) and EDS mapping micrographs of pinning precipitates in PT P92 indicating a V-rich particle covering a Nb-rich particle (assisted by Dr. Hiroto).

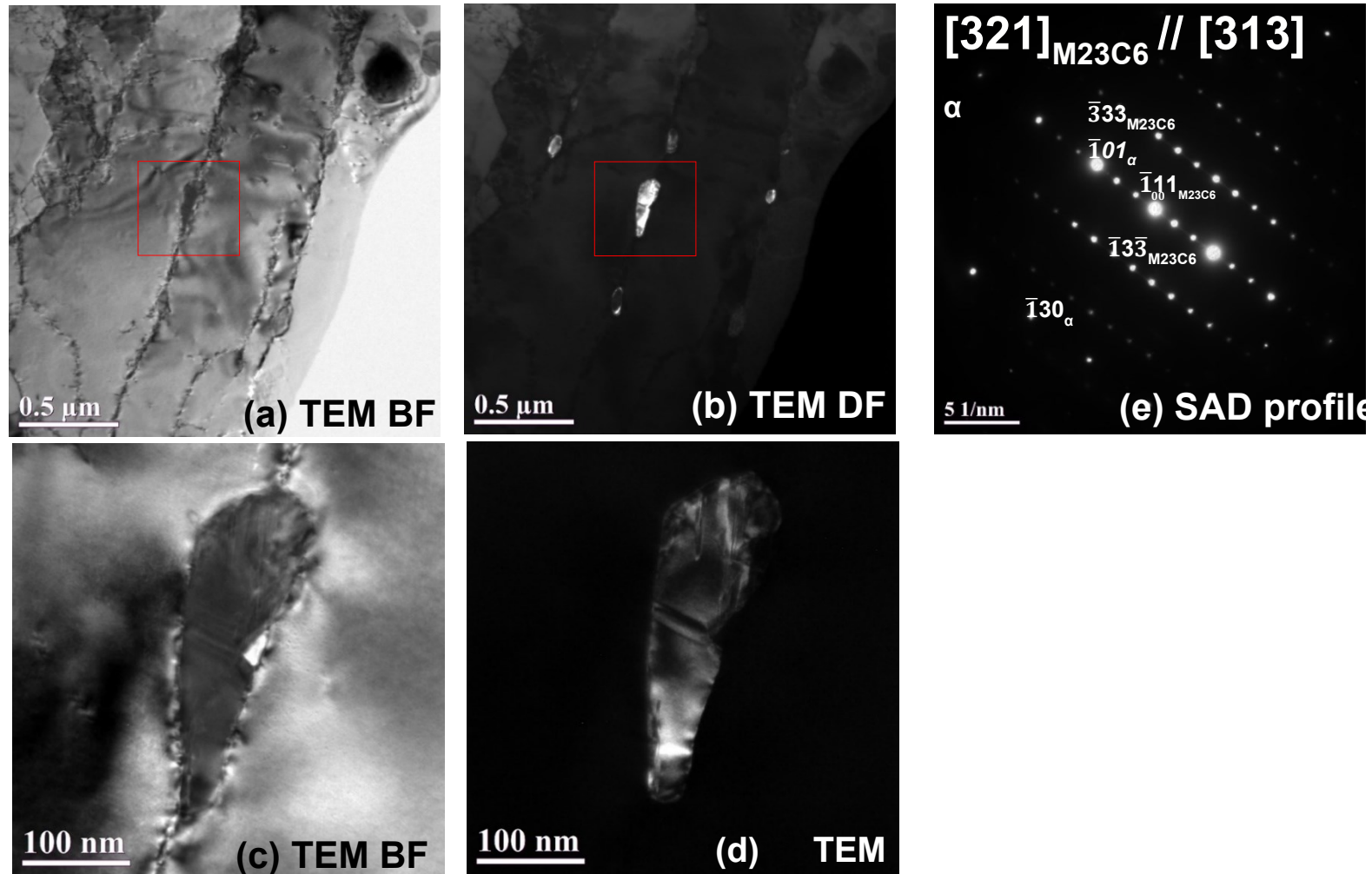


Figure 4.36 TEM bright field (BF), dark filed (DF) micrographs and SAD pattern profile of $M_{23}C_6$ precipitate in PT P92 (assisted by Dr. Hiroto).

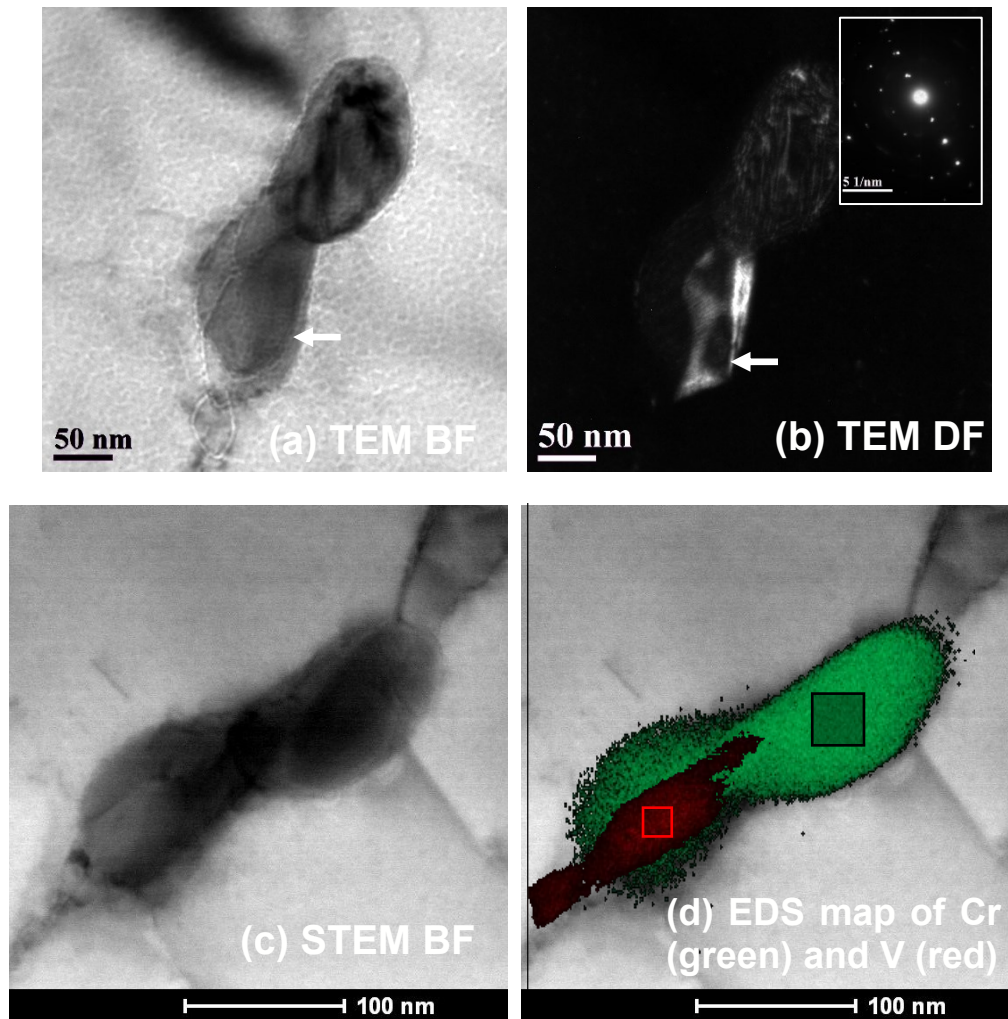


Figure 4.37 $M_{23}C_6$ carbide potential phase transformation in PT P92, (a)-(b): TEM bright field (BF), TEM dark filed (DF) micrographs of transformed phase and its SAD pattern profile, (c): STEM bright field (BF) micrograph, (d) STEM EDS mapping of chemistry difference in the carbide (assisted by Dr. Hiroto).

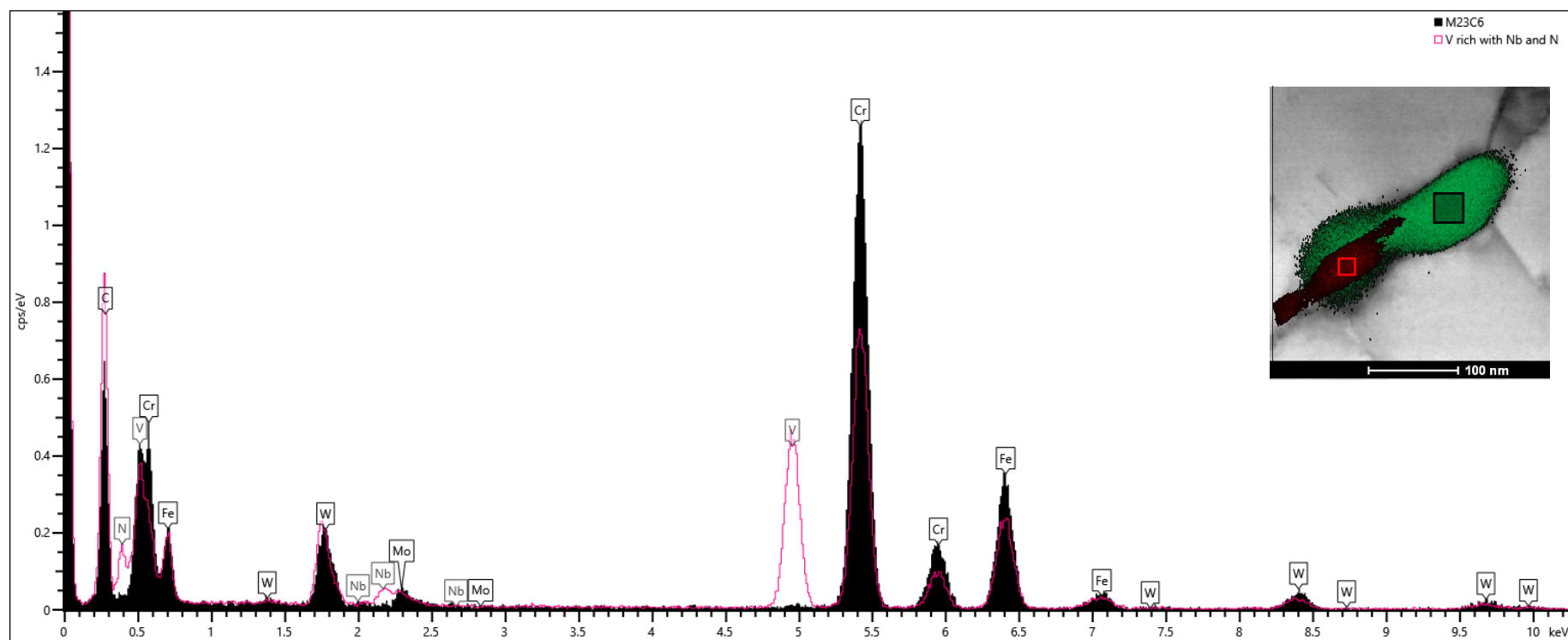


Figure 4.38 EDS spectrum of the selected areas from $M_{23}C_6$ carbide indicating N, Nb and V increasement in PT P92 (assissted by Dr. Hiroto).

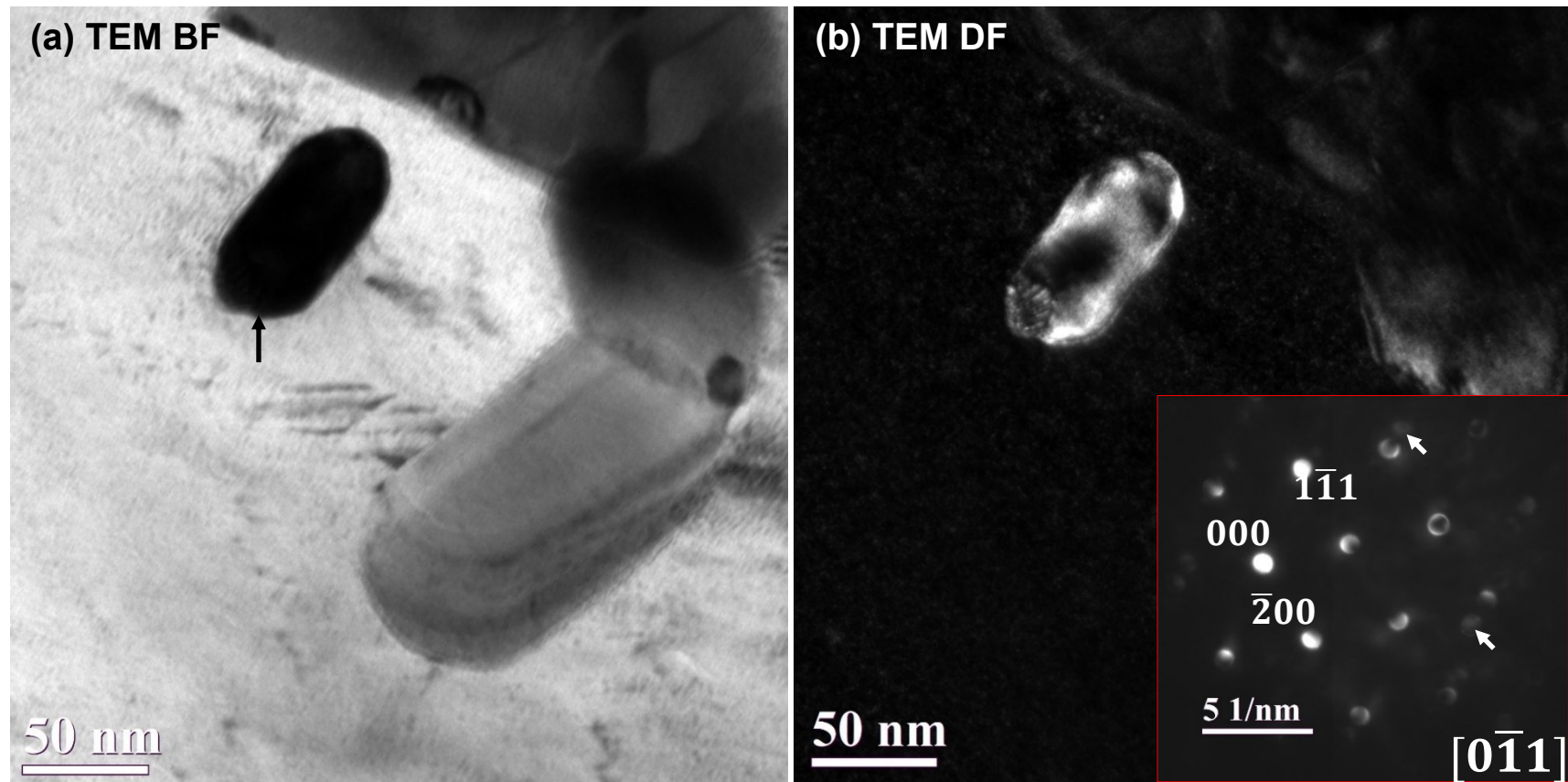


Figure 4.39 TEM bright field (BF), dark filed (DF) micrographs and SAD pattern profile of MX precipitate in PT P92 (assissted by Dr. Hiroto).

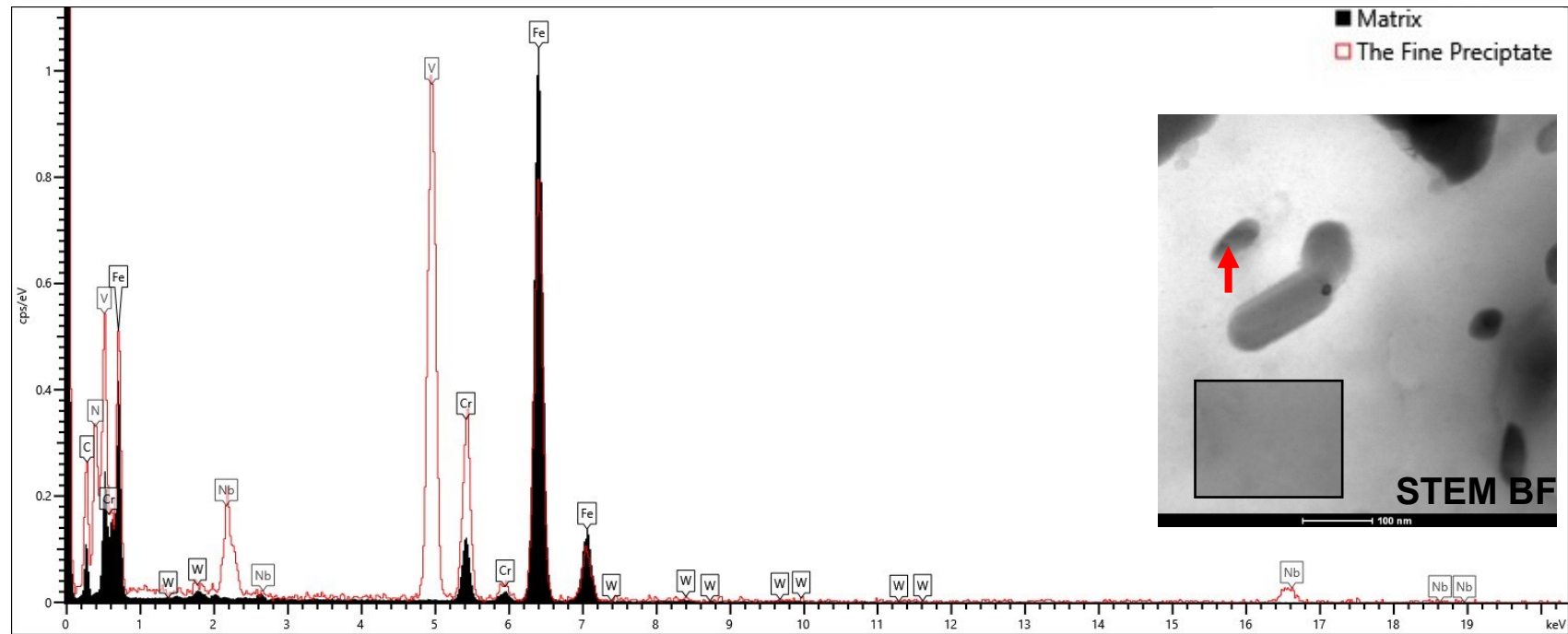


Figure 4.40 EDS spectra of matrix and MX particle in PT P92 indicating clear V , Nb and Cr-rich in MX particle (assissted by Dr. Hiroto).

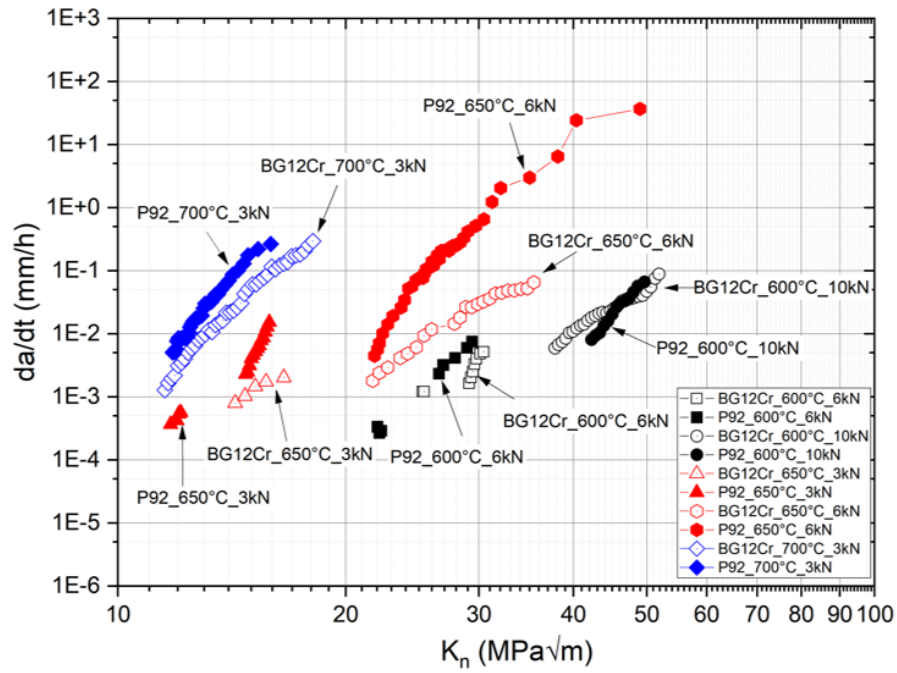


Figure 4.41 Creep crack growth resistance curves using K parameter as driving force in CT geometry.

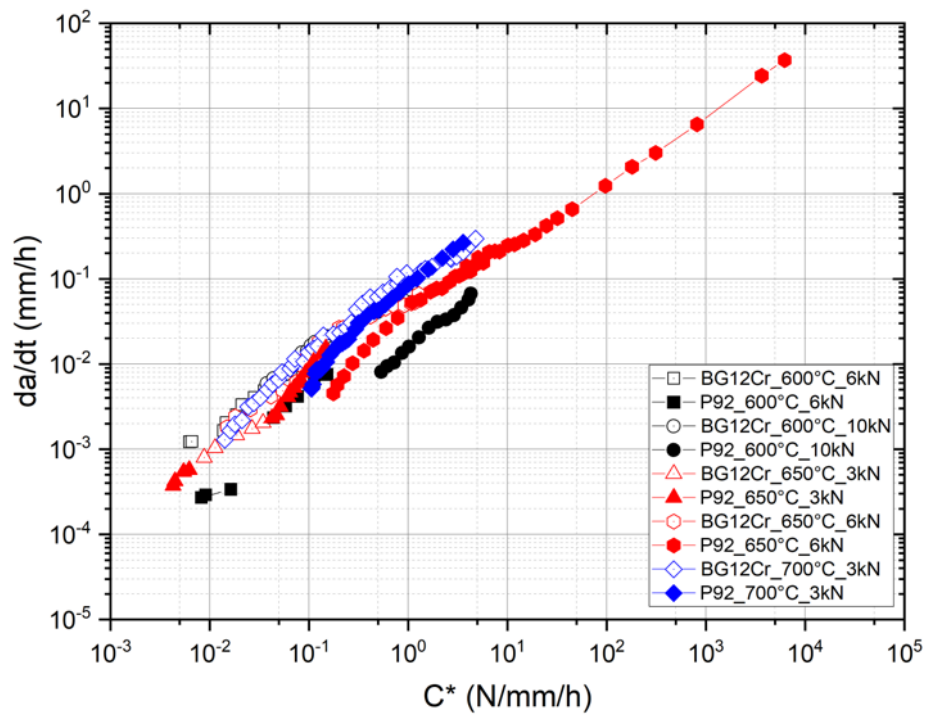


Figure 4.42 Creep crack growth resistance curves using C* parameter as driving force in CT geometry.

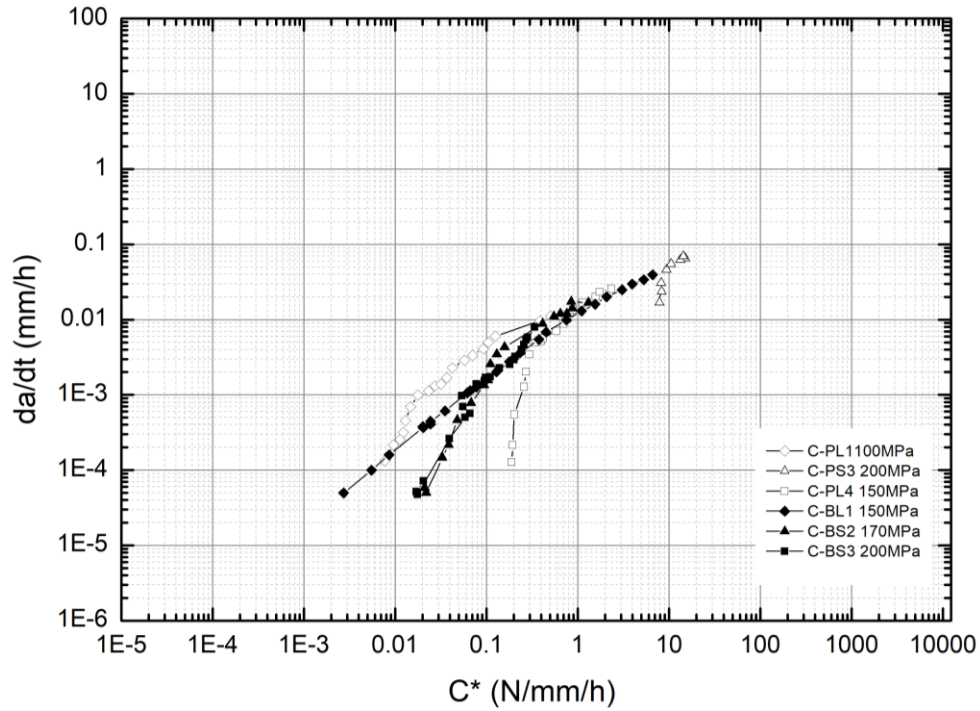


Figure 4.43 Creep crack growth resistance curves using the elastic-plastic parameter C^* as the crack driving force in ring-notched geometry at 650°C.

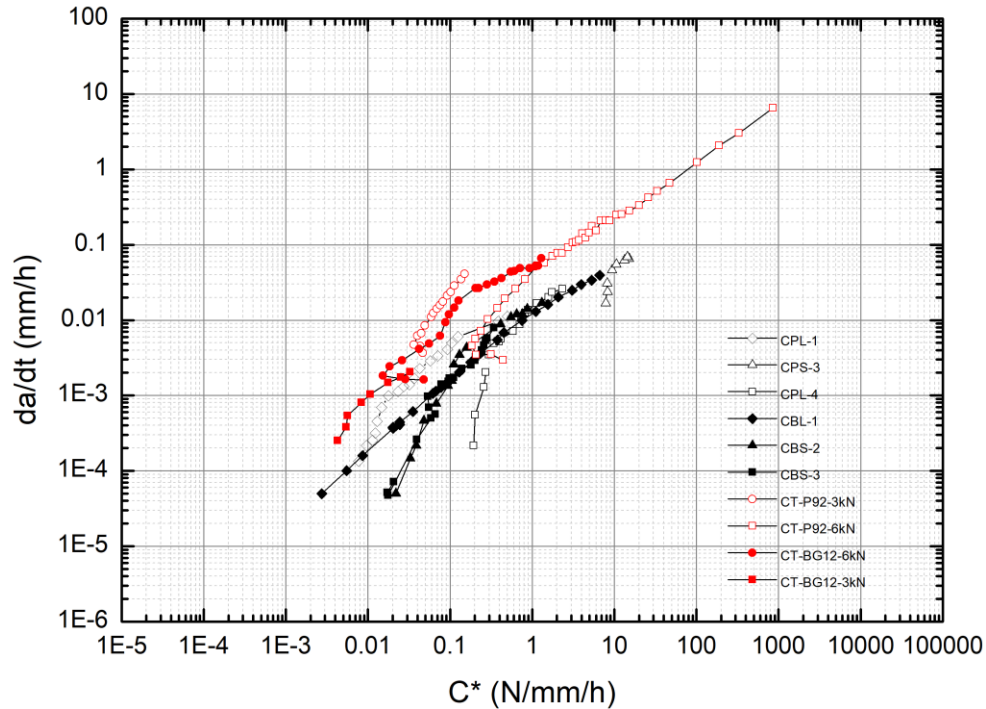


Figure 4.44 Creep crack growth resistance curves using C^* parameter at 650°C with two different geometries ("C-" is circular notched testpiece, "CT" is compact tension testpiece).

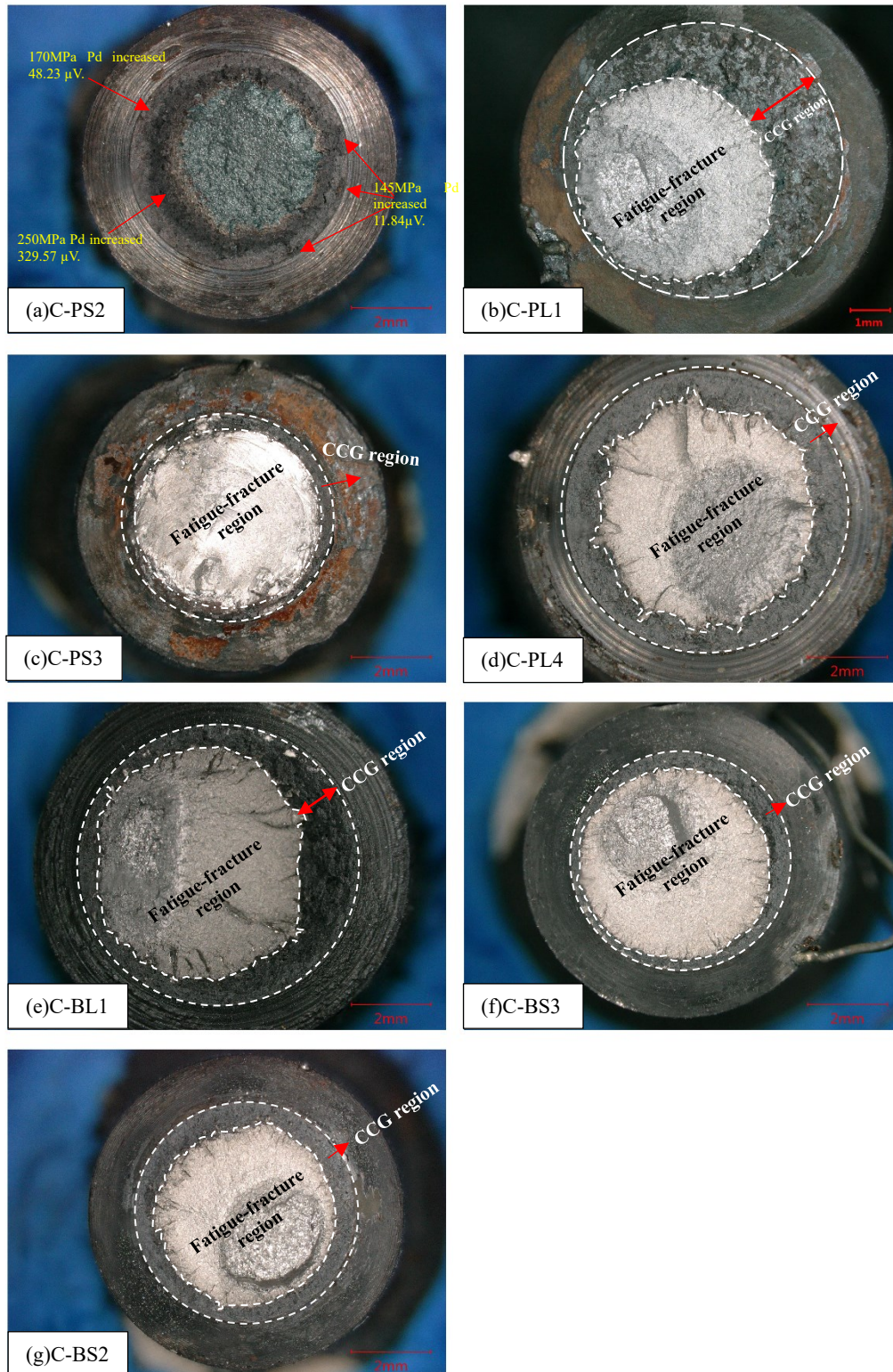


Figure 4.45 Fractography of fracture surfaces from all the ring-notched testpieces.

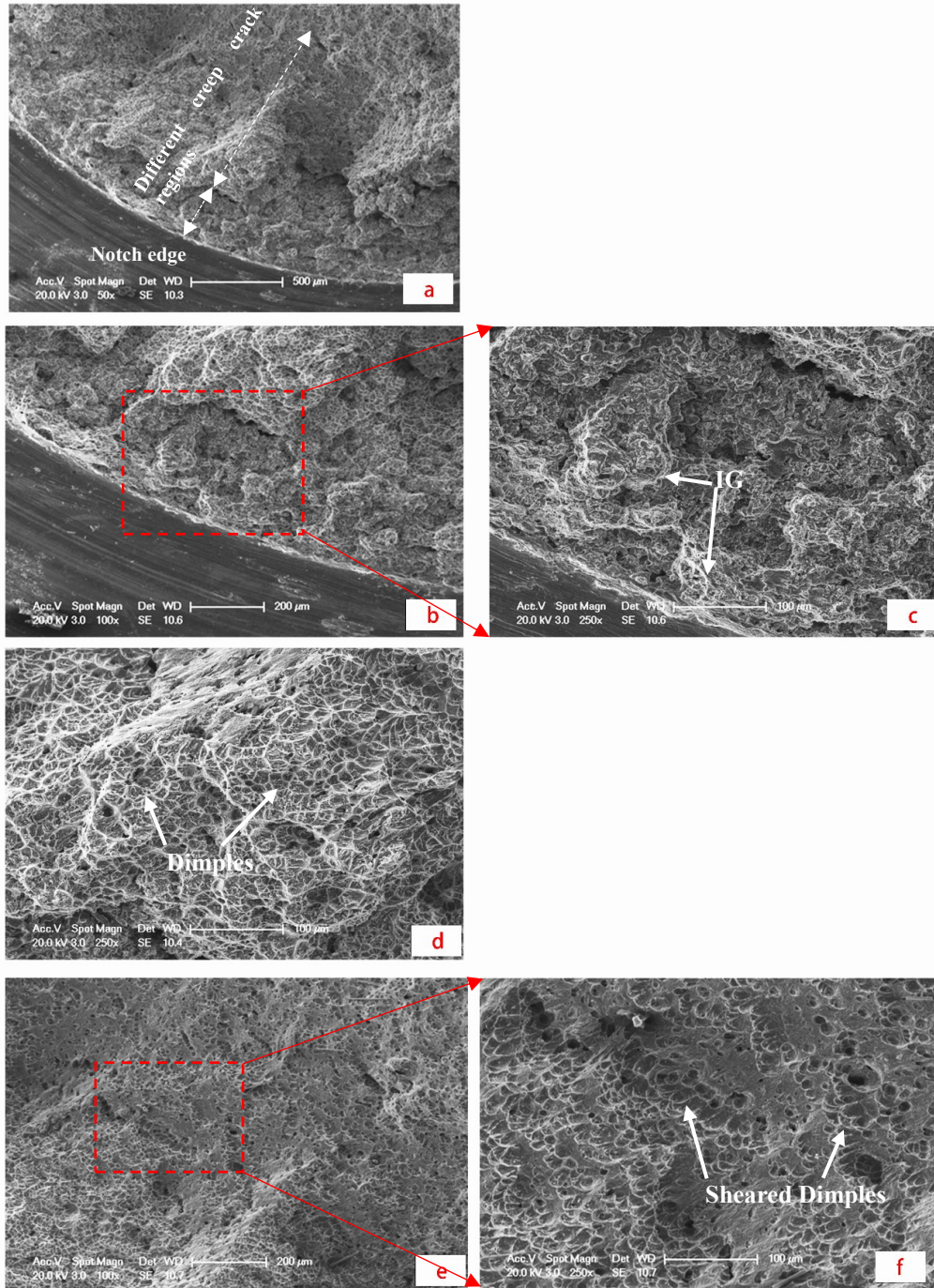


Figure 4.46 SEM fractography of specimen C-PS2, (a) low magnified view of whole CCG region; (b)-(c) CCG start region showing intergranular feature; (d) high magnified view of CCG middle zone showing dimples; (e)-(f) different magnifications of CCG final stage showing sheared dimples.

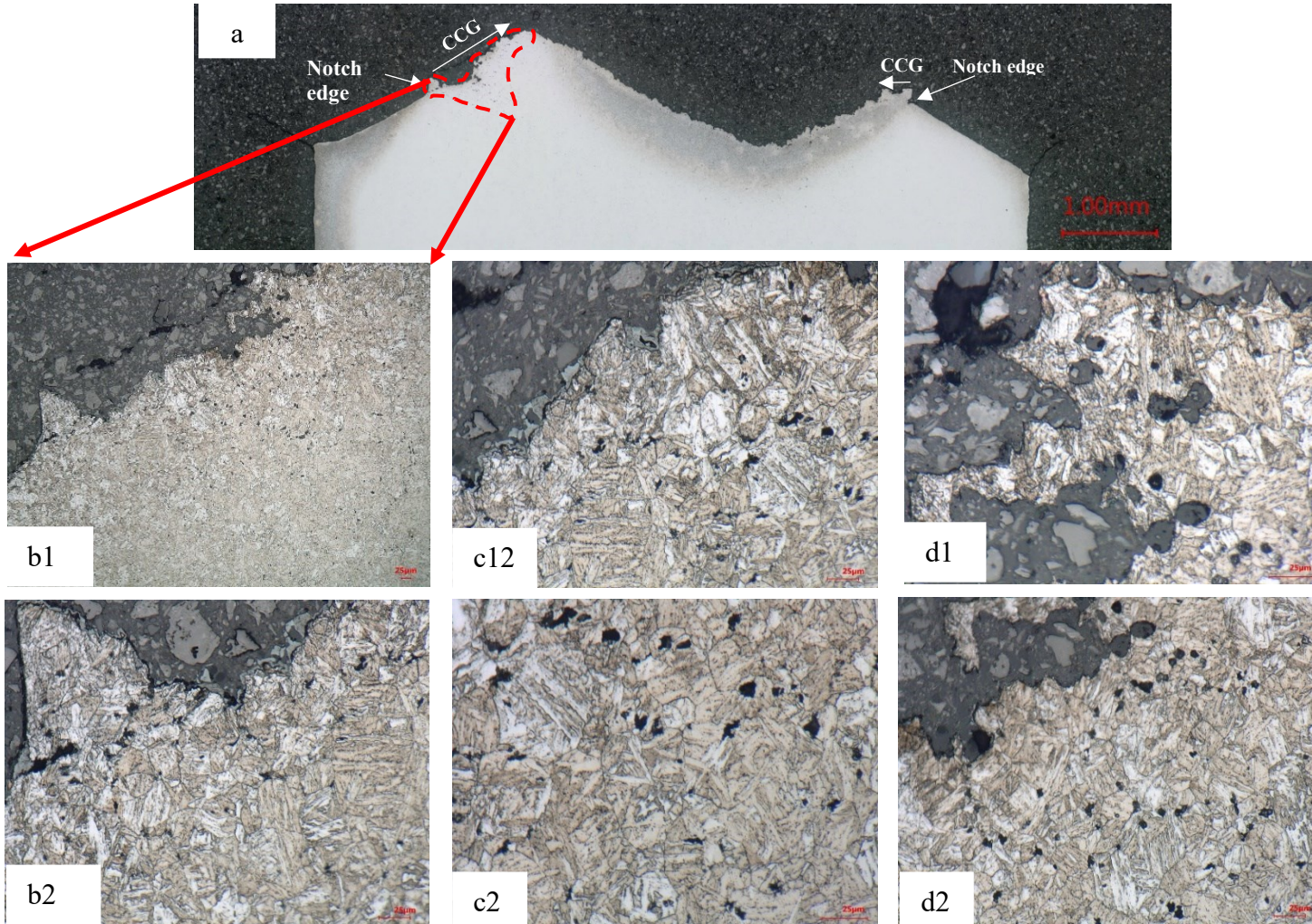


Figure 4.47 Optical micrographs of C-PS2, (a): optical low magnified view of whole cross-sectioned specimen close to the fracture surface, (b1): low magnified view of creep crack growth (CCG) zone; (b2): crack growth region close to the notch edge; (c1), (d1)-(d2): the middle and final CCG regions; (c2) regions below (c1).

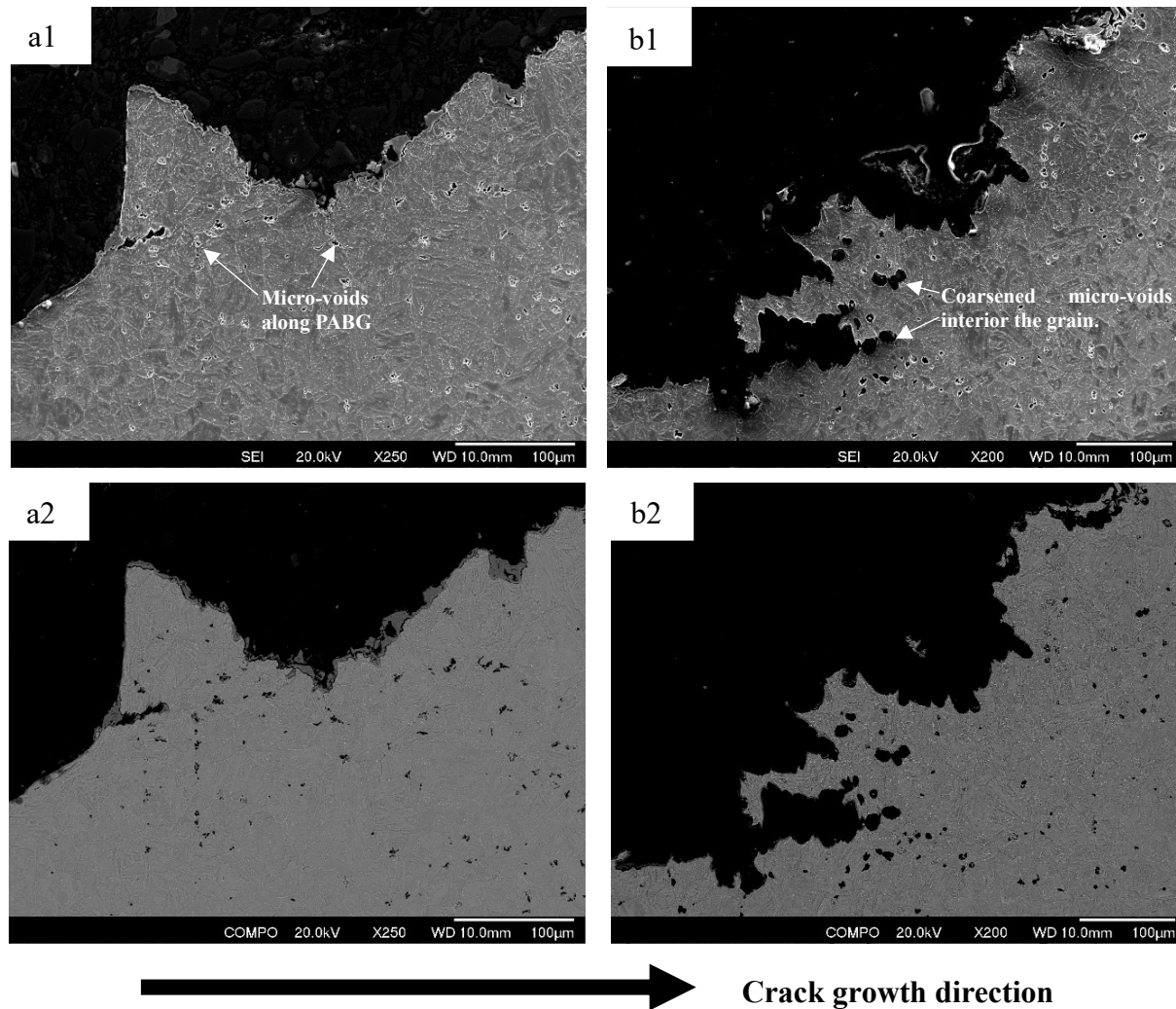


Figure 4.48 SEM images of a longitudinal cross-section through C-PS2 testpiece: (a1) -(a2) beginning stage of CCG showing micro-voids formed along PABGs; (b1) -(b2) final stage of CCG showing coarsened micro-voids across two adjacent PAGs.

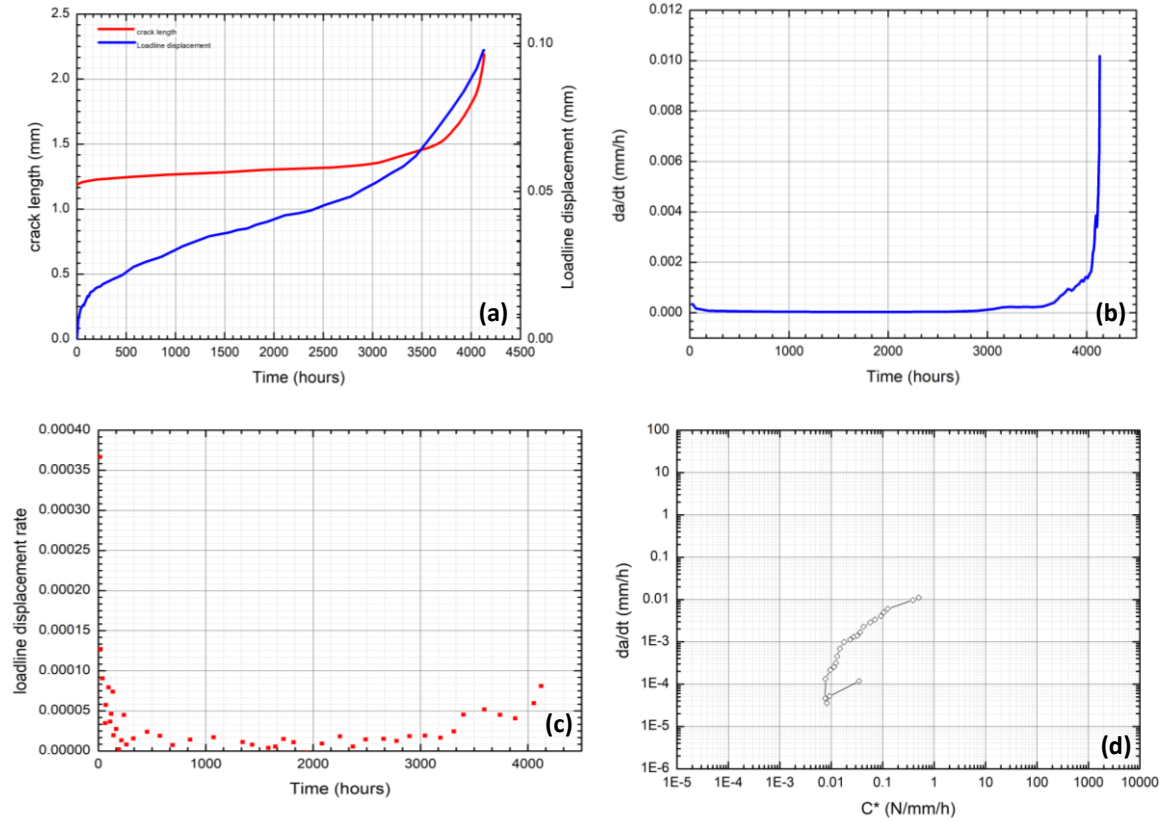


Figure 4.49 creep crack growth plots of C-PL1: (a) creep crack length (mm) and loadline displacement (mm) versus time (hours), (b) creep crack growth rate (mm/h) versus time (hours), (c) loadline displacement rate (mm/h) versus time (hours), (d) creep crack growth resistance curve with C^* as a driving force (tail part was kept).

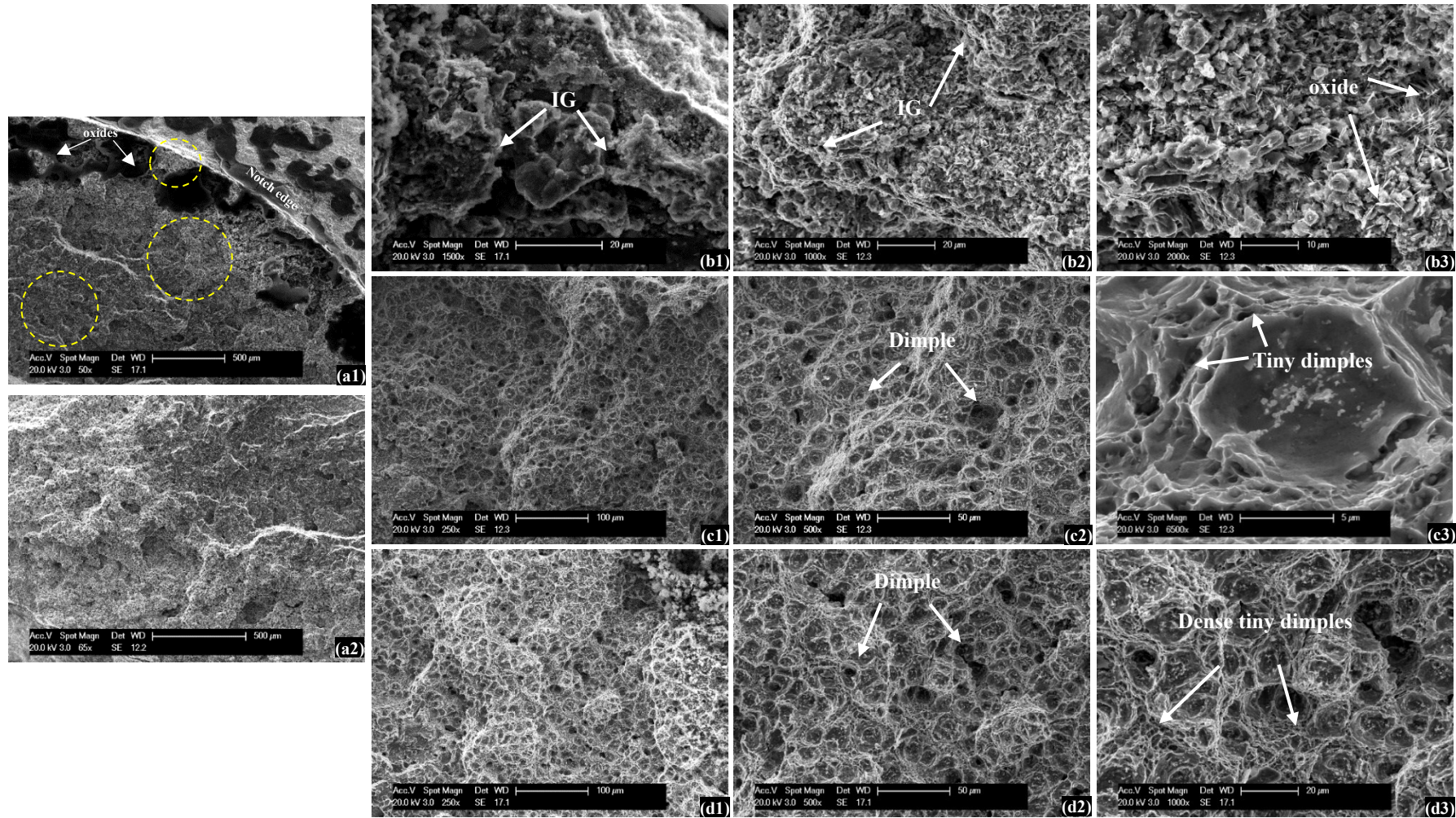


Figure 4.50 SEM fractography of specimen C-PL1, (a1) -(a2) low magnified view of whole CCG region; (b1) -(b3) high magnification image of the beginning stage of CCG region; (c1) -(c3) different magnification images of transition stage; (d1) -(d3) different magnification images of CCG final stage before interrupted.

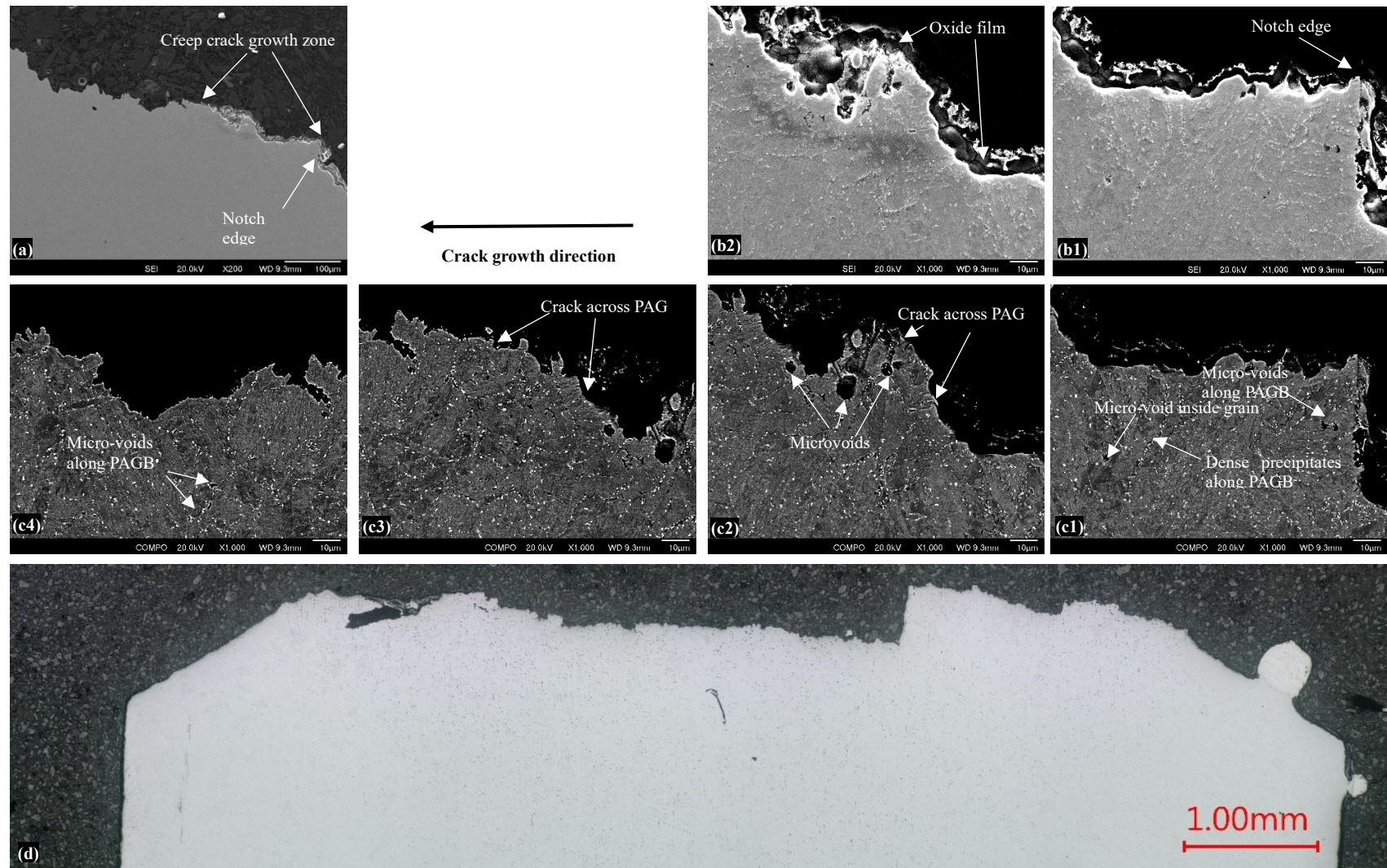
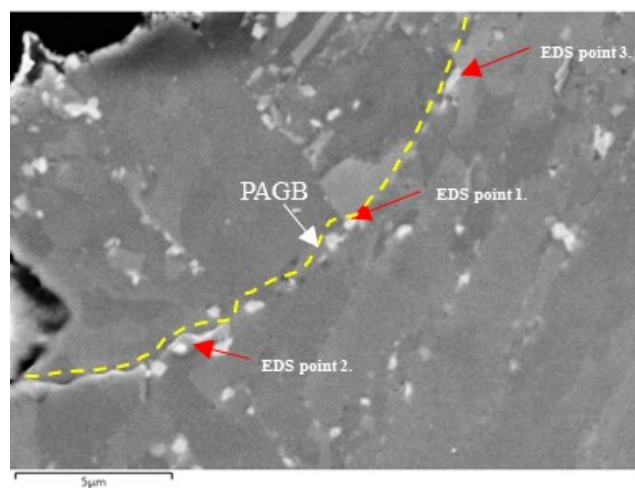


Figure 4.51 OM and SEM micrographs of cross section through C-PL1 (96MPa, 650°C), (a): low magnification image of whole CCG zone; (b2) -(c4) high magnified view of cross-sectioned specimen close to fracture surface; (d): optical micrograph indicating micro-voids distribution.



Spectrum	1	2	3
Element	weight(%)		
C	49.25	10.73	44.85
N	/	5.98	/
Cr	11.12	53.6	10.59
Fe	32.51	24.22	40.7
Co	0.25	/	/
Mo	1.69	1.04	1.12
W	5.17	4.43	2.75

Figure 4.52 SEM image close to the fracture surface and EDS spectra of C-PL1.

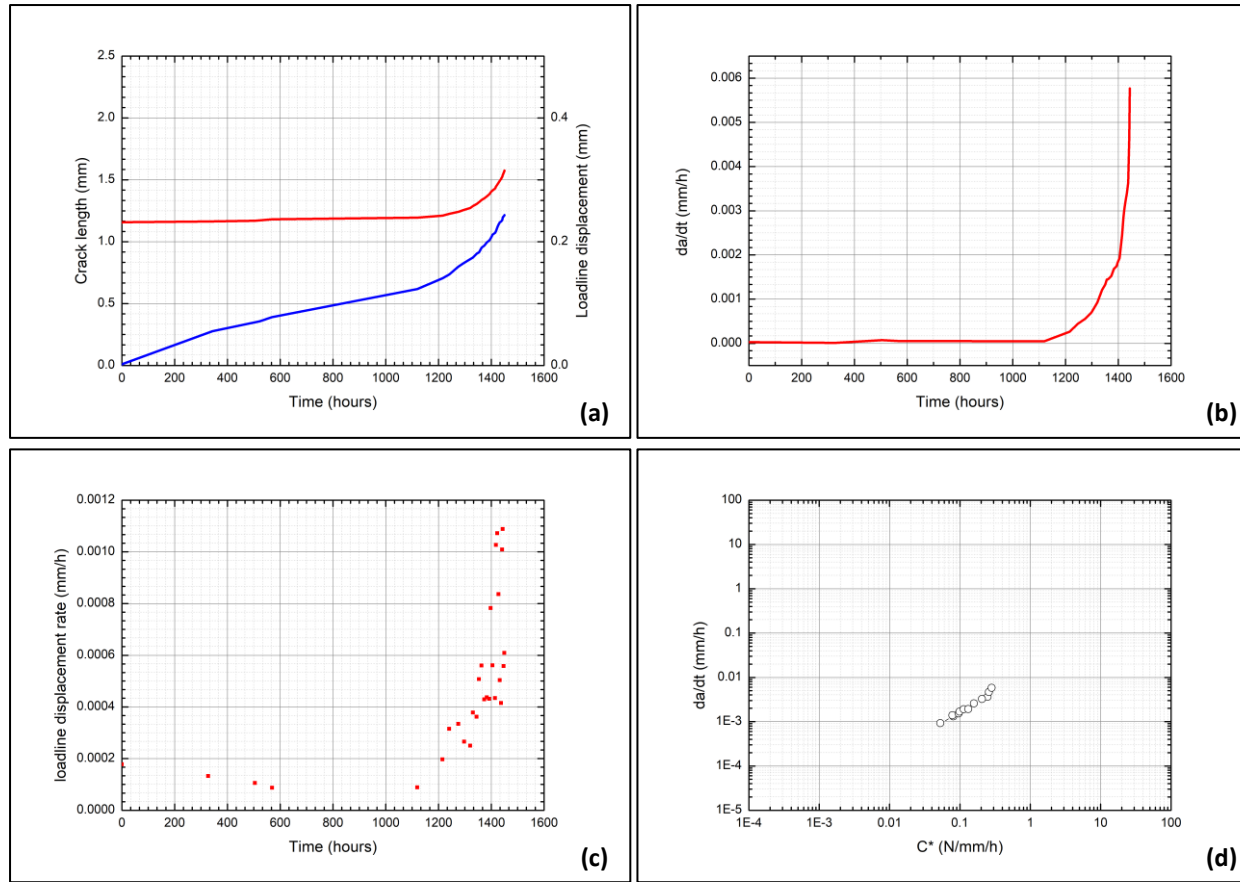


Figure 4.53 creep crack growth plots of C-BS3 (170MPa, 650°C): (a) creep crack length (mm) and loadline displacement (mm) versus time (hours), (b) creep crack growth rate (mm/h) versus time (hours), (c) loadline displacement rate (mm/h) versus time (hours), (d) creep crack growth resistance curve with C^* as a driving force.

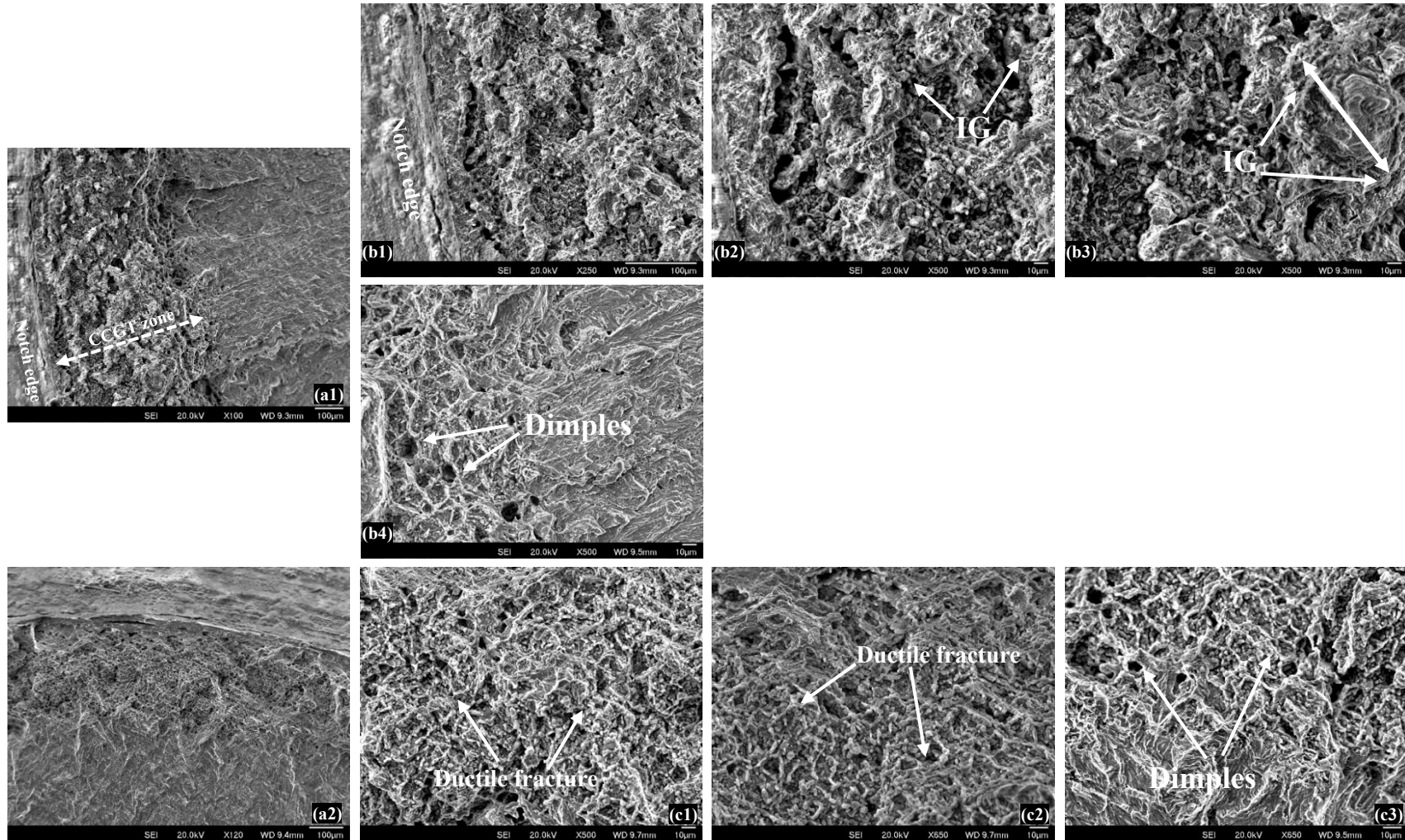


Figure 4.54 SEM fractography of specimen C-BS3 (170MPa,650°C), (a1)-(a2): low magnification image of two CCG zones; (b1)-(b2):different magnification images of the beginning CCG stage; (b3) the transition CCG stage; (b4) the end of CCG stage; (c1)-(c3) high magnification images of (a2) CCG zone.

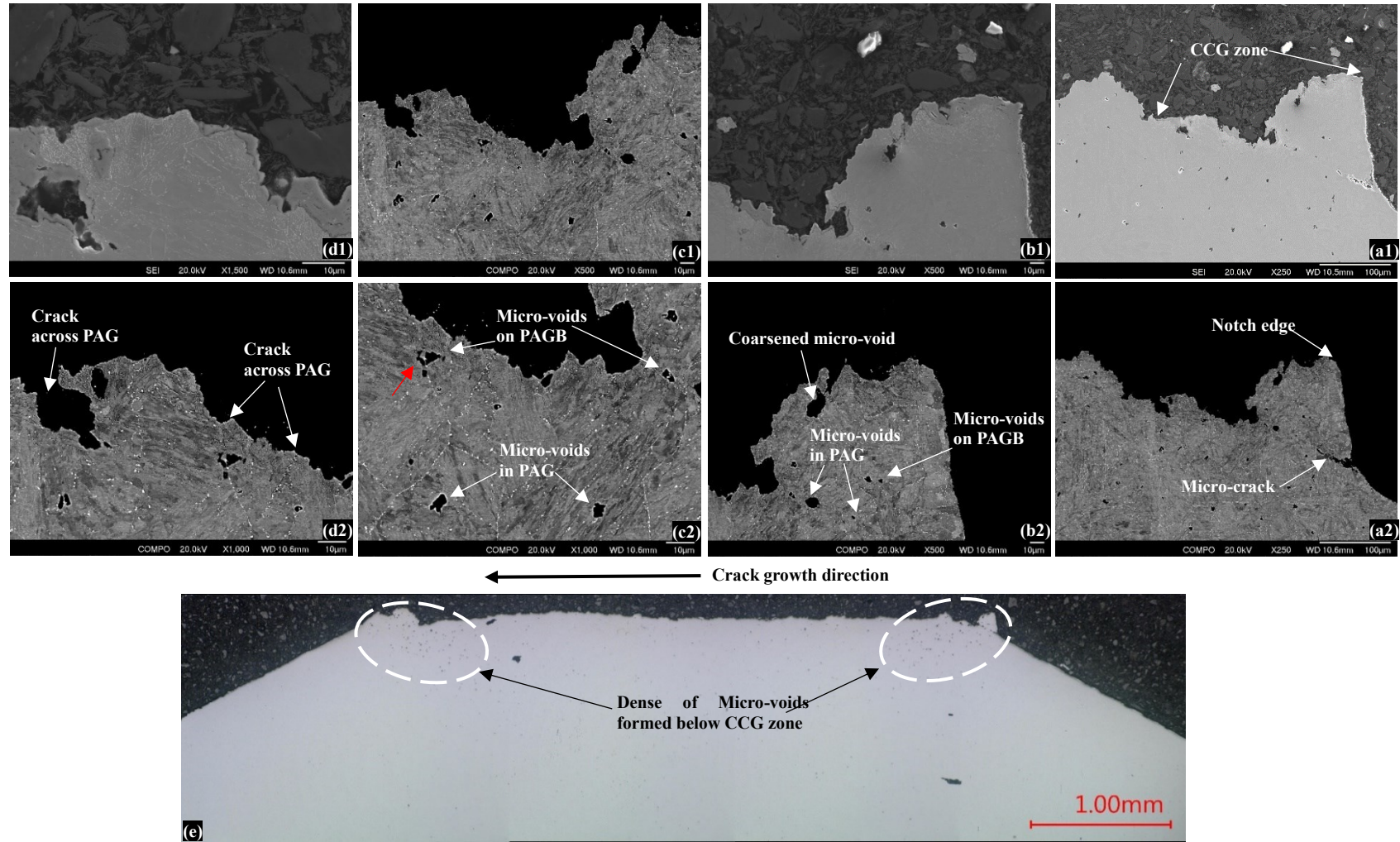


Figure 4.55 OM and SEM micrographs of cross section through C-BS3 (170MPa,650°C), (a1) -(a2) low magnification images of whole CCG zone, (b1) -(b2) high magnified view of the beginning CCG stage, (c1) -(c2) high magnified view of the middle CCG stage, (d1)-(d2) high magnified view of the final CCG stage, (e) optical micrograph indicating micro-voids distribution.

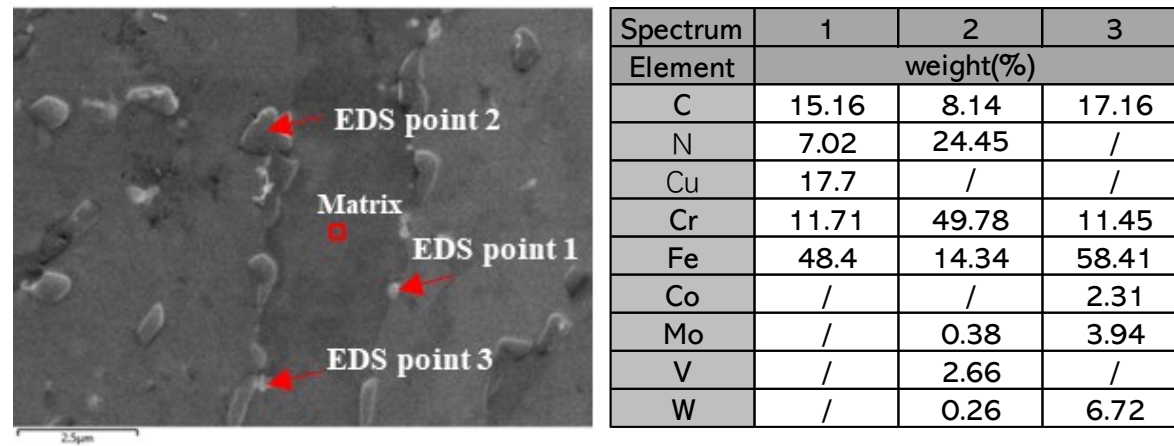


Figure 4.56 SEM image showing precipitates along prior austenite grain boundaries and EDS spectra of these precipitates in C-BS3.

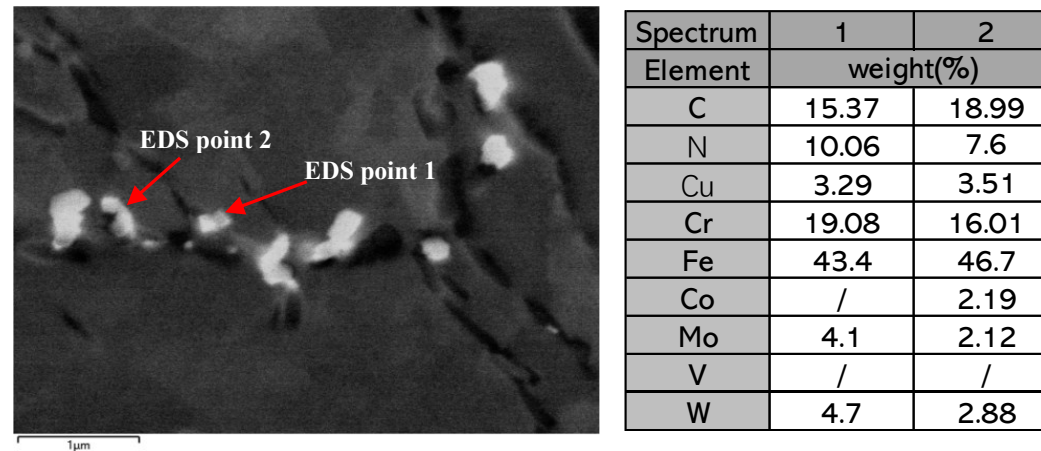


Figure 4.57 SEM image showing precipitates along prior austenite grain boundaries and EDS spectra of these precipitates in C-BS3 indicating (W,Mo)-rich particles with Cu content.

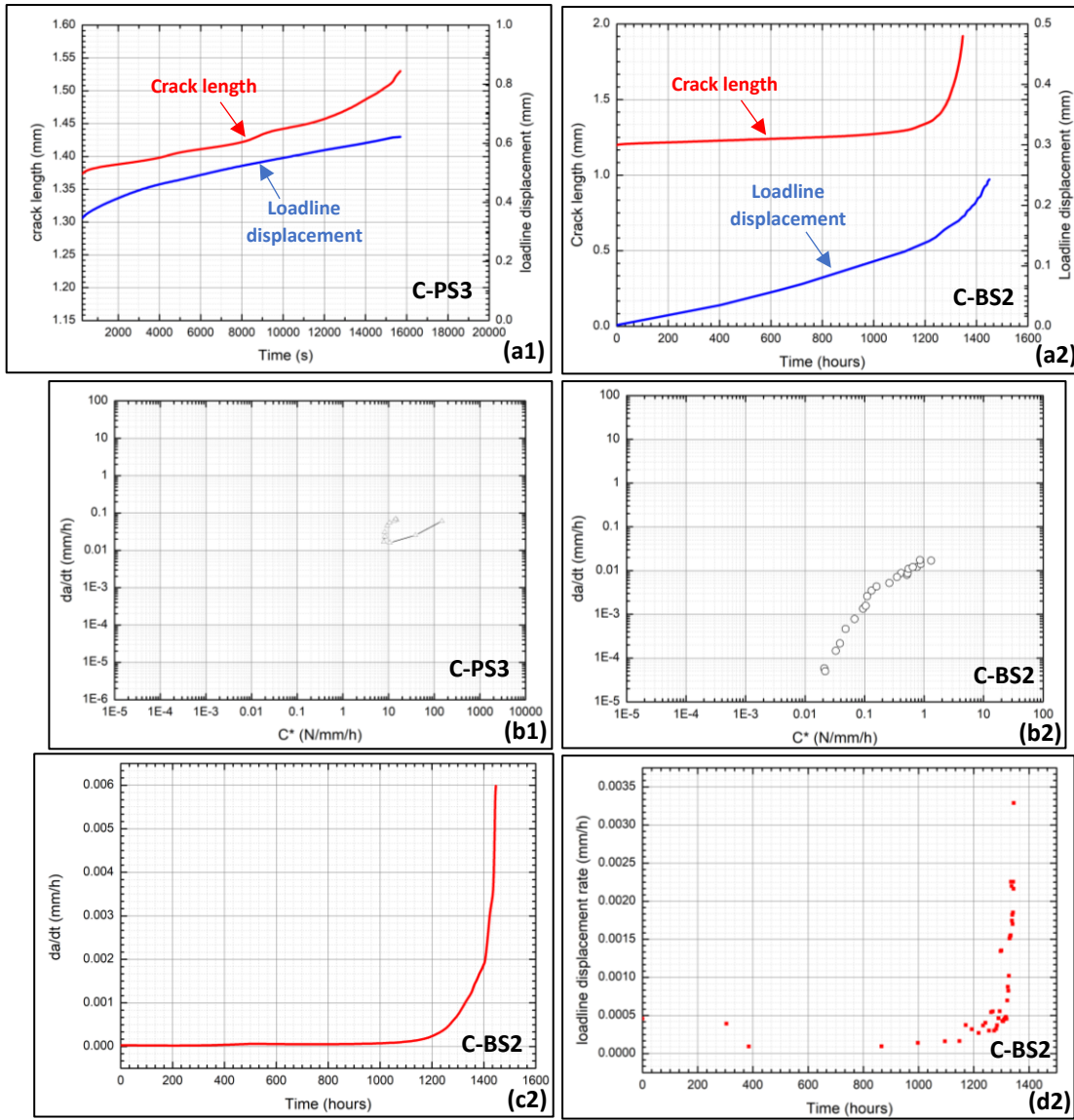


Figure 4.58 46 creep crack growth plots of testpieces (C-PS3&C-BS2) test at 200 MPa at 650°C, (a1)-(a2): creep crack length (mm) and loadline displacement (mm) versus time;(b1)-(b2): creep crack growth resistance curve with C^* as a driving force; (c2): creep crack growth rate (mm/h) versus time (hours), (d2): loadline displacement rate (mm/h) versus time (hours).

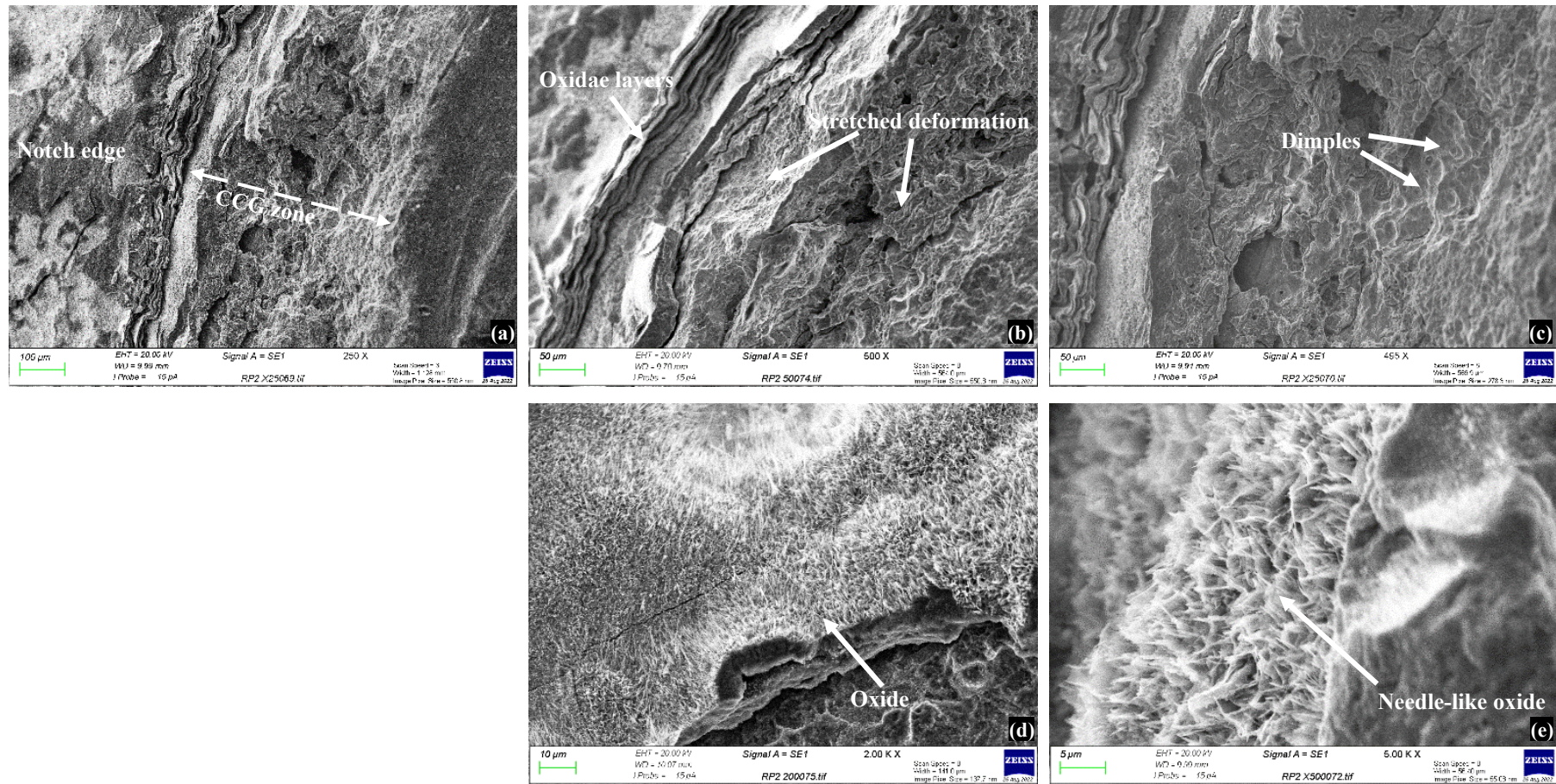


Figure 4.59 SEM fractography of specimen C-PS3 (200MPa,650°C), (a) low magification image of CCG zone invloving notch edge; (b)-(c) high magnification images of CCG zone indicating ductile dimples fracture mechanism; (d)-(e): high magnification images of oxide layer indicating a dense fromation of needle-like oxides.

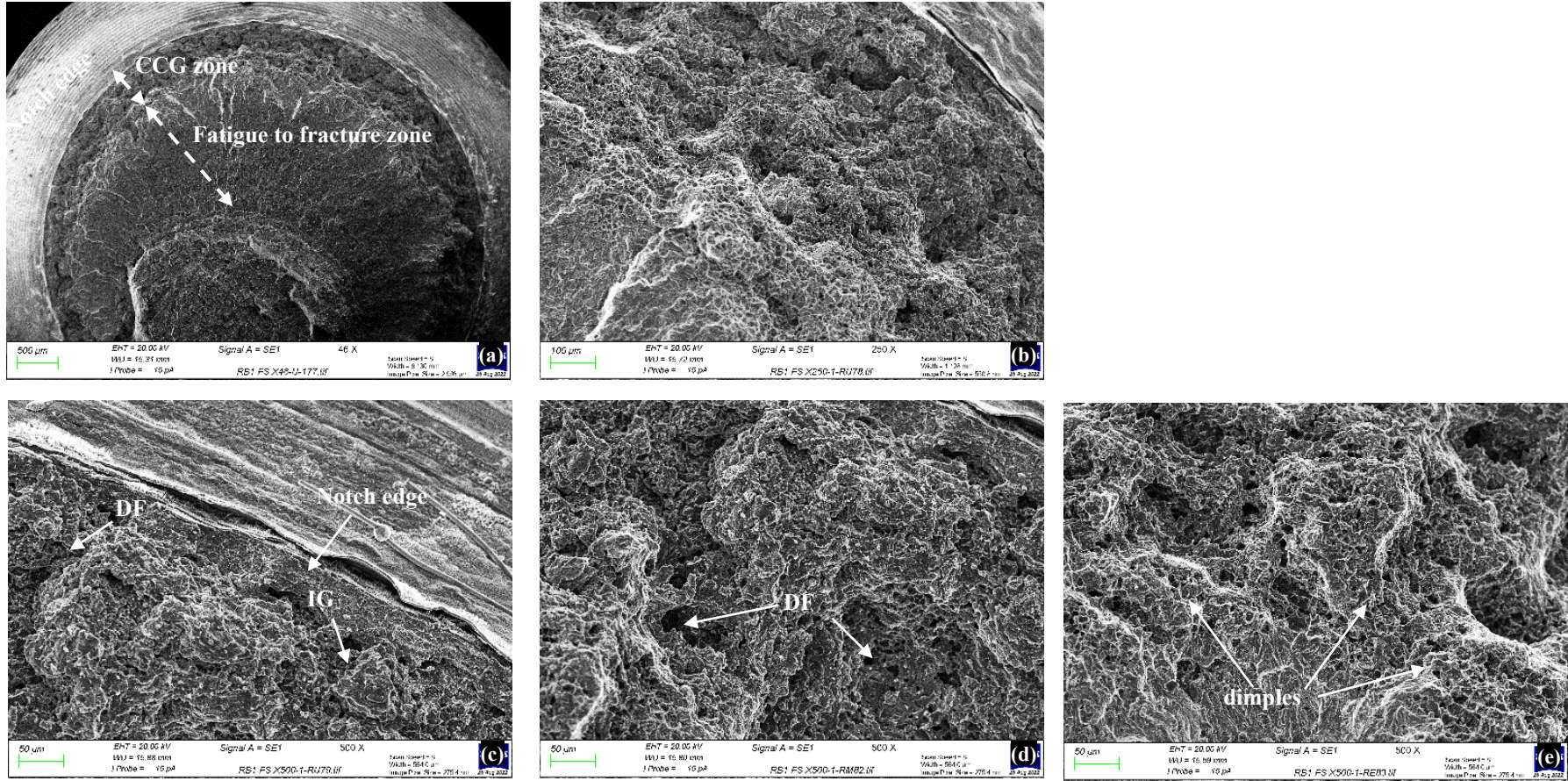


Figure 4.60 SEM fractography of specimen C-BS2 (200MPa,650°C), (a) low magification image of CCG zone invloving notch edge; (b) high magnification images of CCG zone ; (c): high magnification image of starting stage of crack grwoth, complete notch edge can be seen and intergaunlar fracture can be observed; (d) high magnificaiton image of middle stage of crack grwoth, ductile fracture is frequently obsreved; (e) the end stage of crack grwoth, ductile dimples was observed.

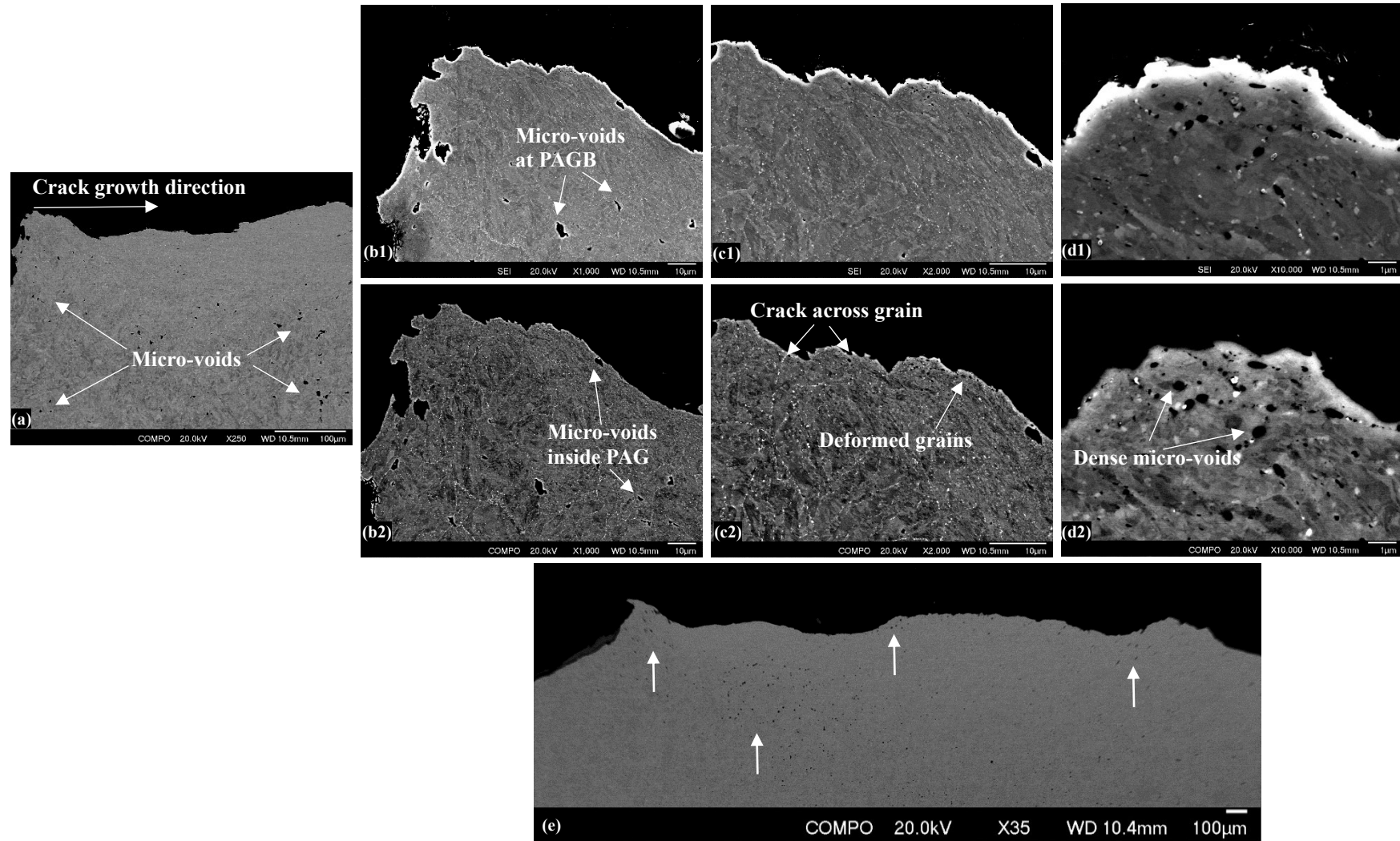


Figure 4.61 SEM micrographs of cross section through C-PS3 (200MPa,650°C), (a) low magification image of CCG zone invloving notch edge; (b1)-(d2): high magnification images of different stages of crack grwoth; (e) SEM backscattered (COMPO) image of whole fracture suface indicaitng the distribution of micro-voids in this specimen.

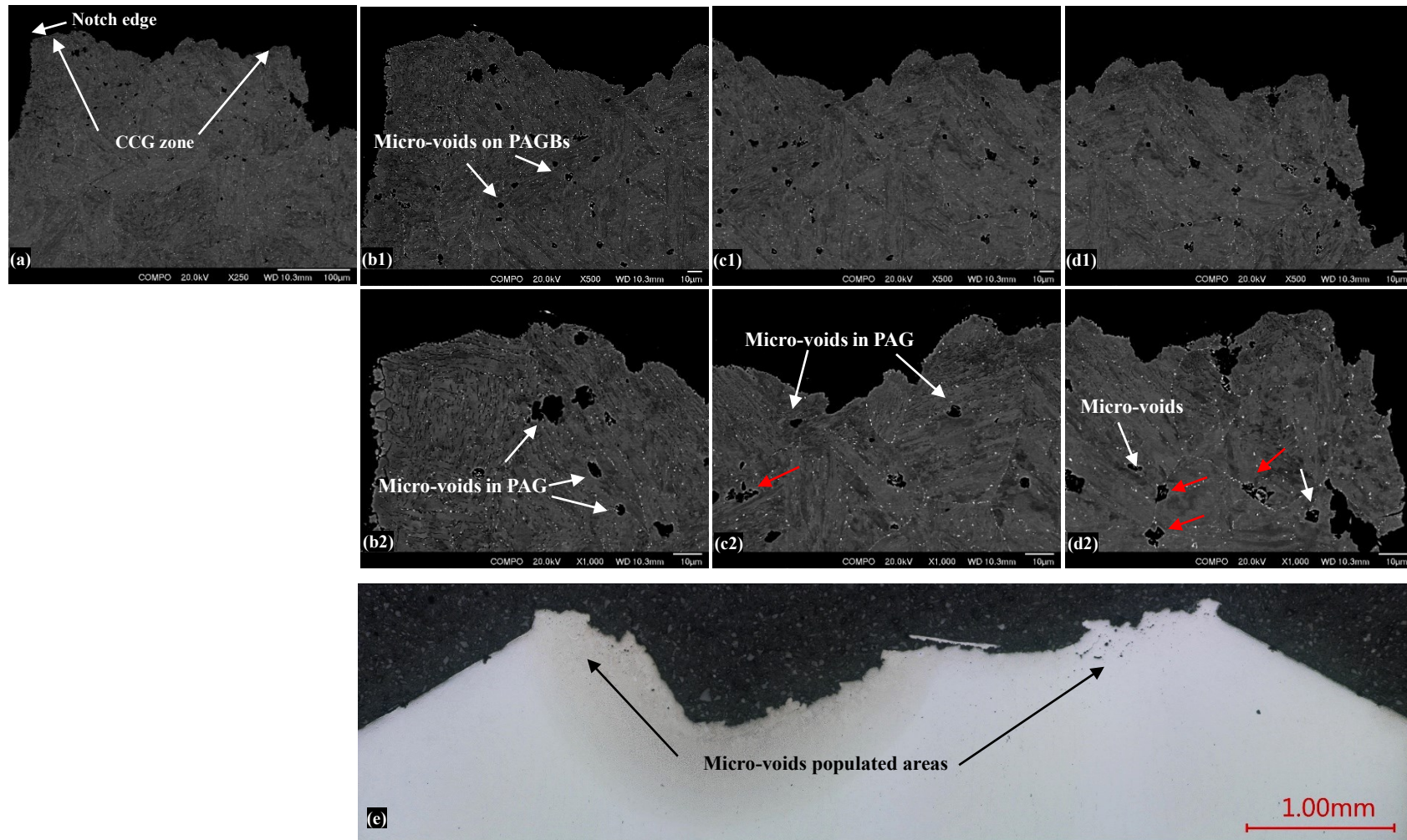


Figure 4.62 OM and SEM micrographs of cross section through C-BS2(200MPa,650°C), (a) low magification image of CCG zone invloving notch edge; (b1)-(b2): different magnification images of crack grwoth initiation area; (c1)-(c2) different magnification images of crack grwoth middle stage; (d1)-(d2) different magnification images of crack growth final stage; (e) optcial image (OM) indicating dense micro-voids distributed below CCG fracture surface.

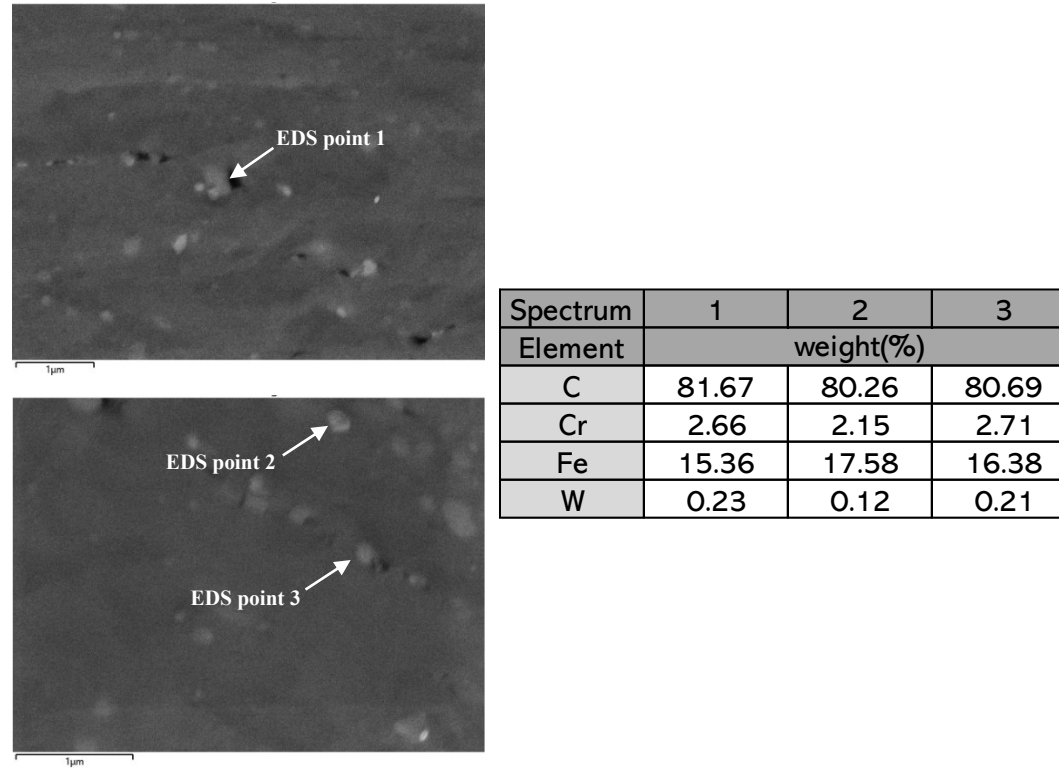
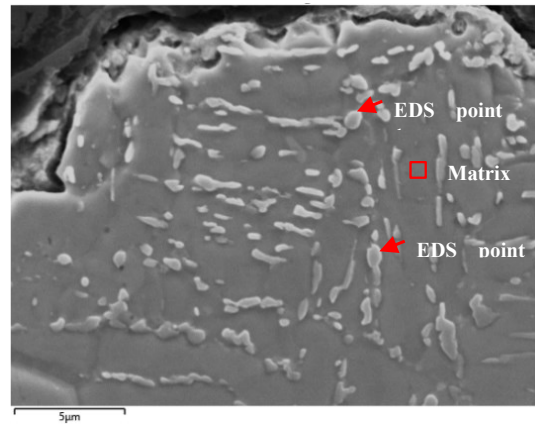


Figure 4.63 SEM image showing precipitates along prior austenite grain boundaries and EDS spectra of these precipitates in C-PS3 indicating W-rich particles along boundaries.



Spectrum	1	2	Matrix
Element	weight(%)		
C	12.98	/	15.6
N	20.77	25.94	/
Cr	30.56	55.51	6.48
Fe	35.39	14.66	75.07
V	/	2.92	/
Co	/	/	2.59
Mo	/	0.4	/
W	0.3	0.57	0.27

Figure 4.64 SEM image showing precipitates along prior austenite grain boundaries and EDS spectra of these precipitates in C-BS2 indicating (Cr,W)-rich particles along boudaries.

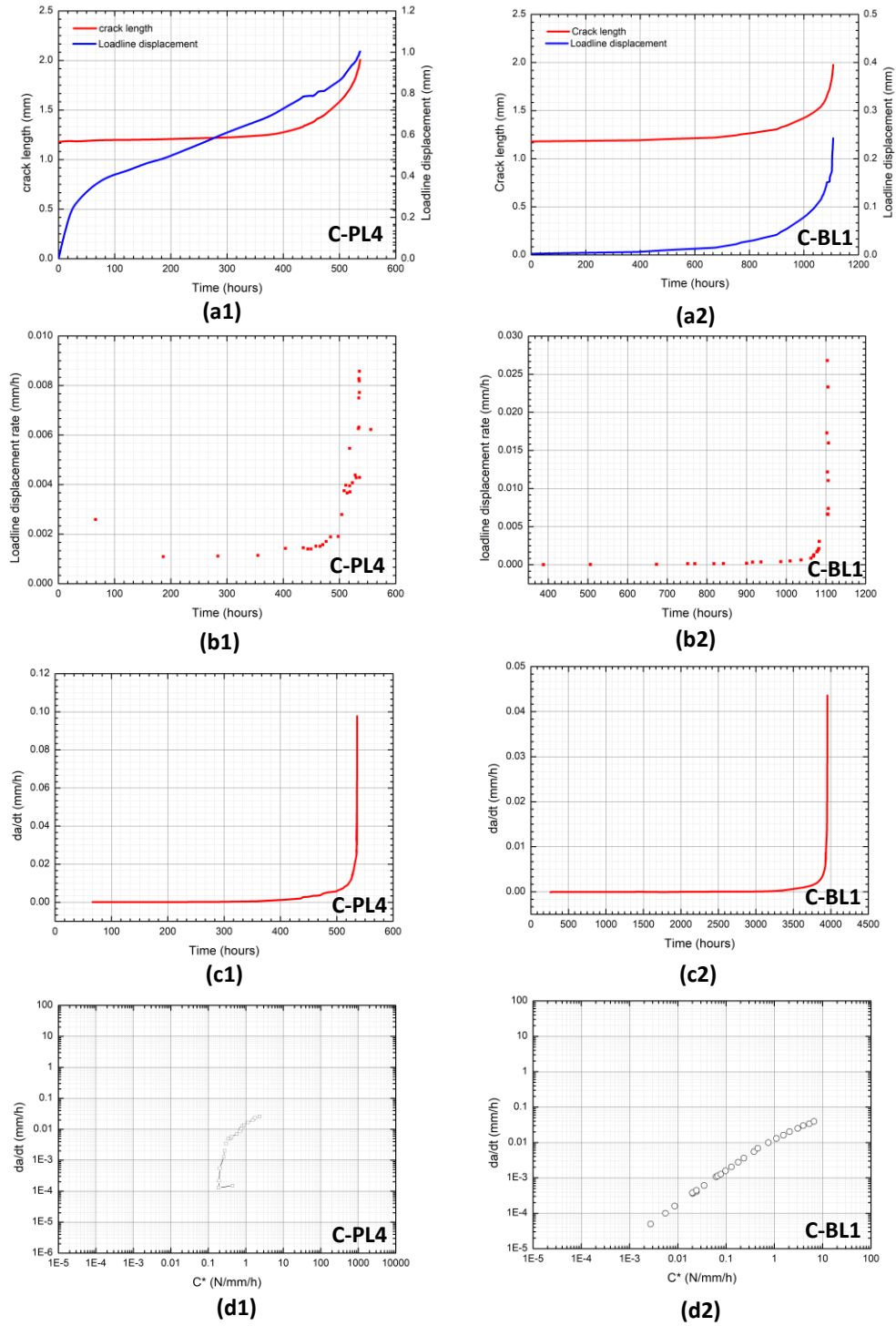


Figure 4.65 creep crack growth plots of testpieces (C-PL4&C-BL1) test at 150 MPa at 650°C, (a1)-(a2): creep crack length (mm) and loadline displacement (mm) versus time;(b1)-(b2): loadline displacement rate (mm/h) versus time (hours); (c1)-(c2): creep crack growth rate (mm/h) versus time (hours), (d1)-(d2): creep crack growth resistance curve with C^* as a driving force.

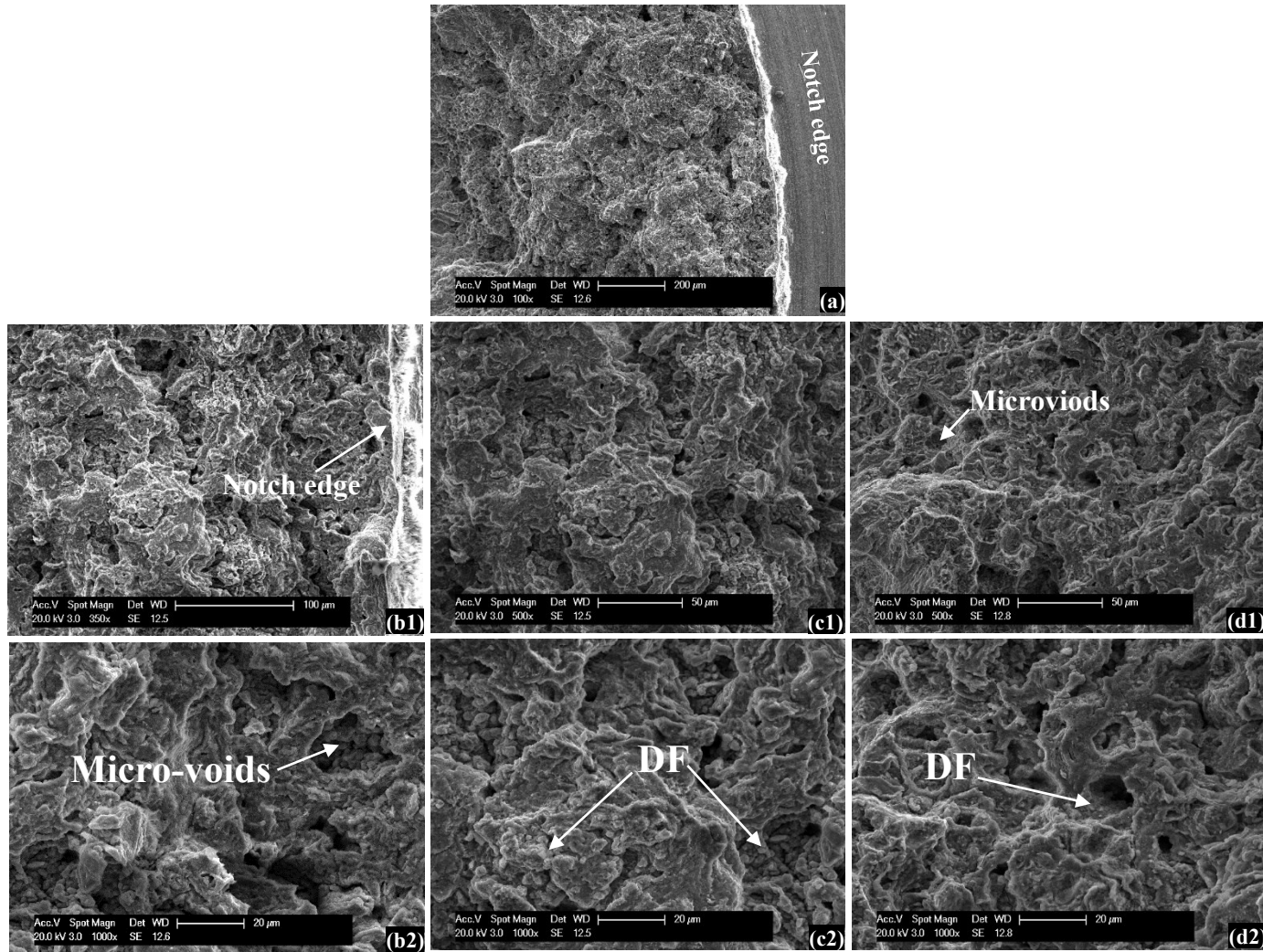


Figure 4.66 SEM fractography of specimen C-PL4 (150MPa, 650°C), (a) low magnification image of fracture surface; (b1)-(b2) the beginning stage of CCG; (c1)-(c2) the middle stage of CCG; (d1)-(d2): the final stage of CCG, dimples were found.

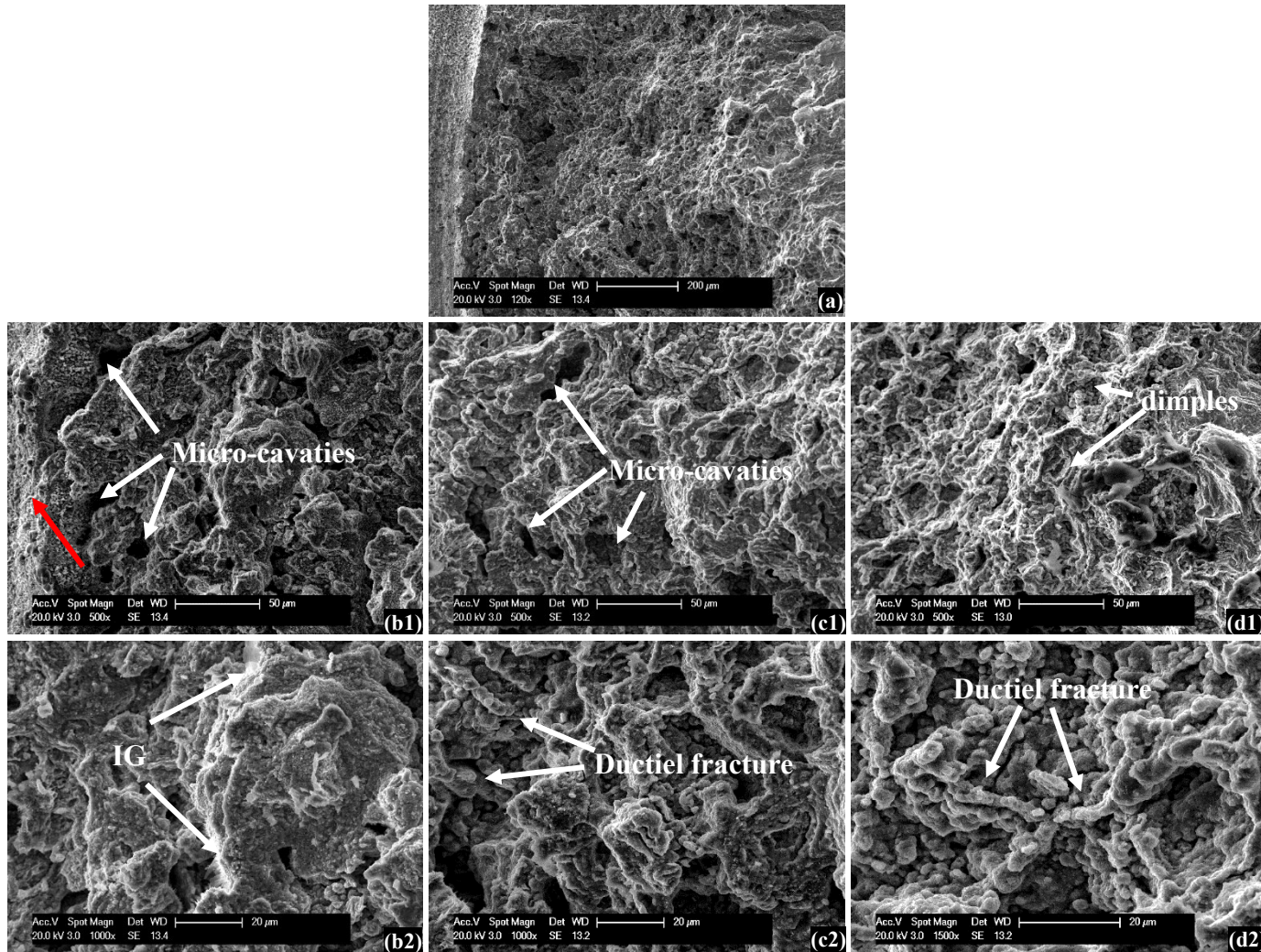


Figure 4.67 SEM fractography of specimen C-BL1 (150MPa, 650°C), (a) low magnification image of fracture surface; (b1)-(b2) the beginning stage of CCG, coarsened micro-cavities and IG were found; (c1)-(c2) the middle stage of CCG indicating a ductile fracture mechanism; (d1)-(d2): the final stage of CCG, ductile dimples were found.

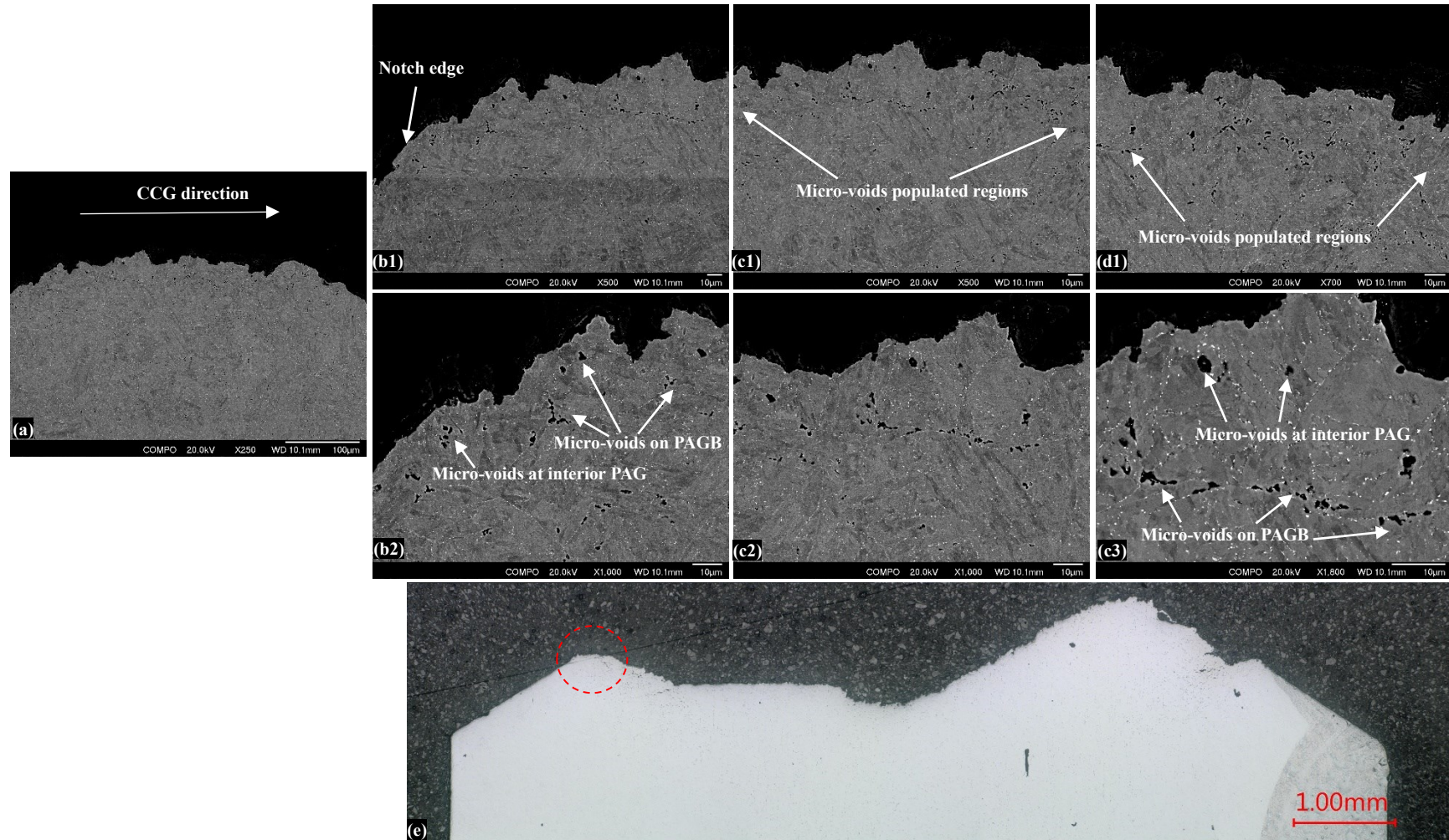


Figure 4.68 OM and SEM micrographs of cross section through C-PL4 (150MPa,650°C), (a) low magification image of CCG zone involvling notch edge; (b1)-(b2): different magnification images of crack grwoth initiation area; (c1)-(c3) different magnification images of crack grwoth middle stage; (d1) high magnification image of crack growth final stage; (e) optcial image (OM) indicating micro-voids distributed across the specimen.

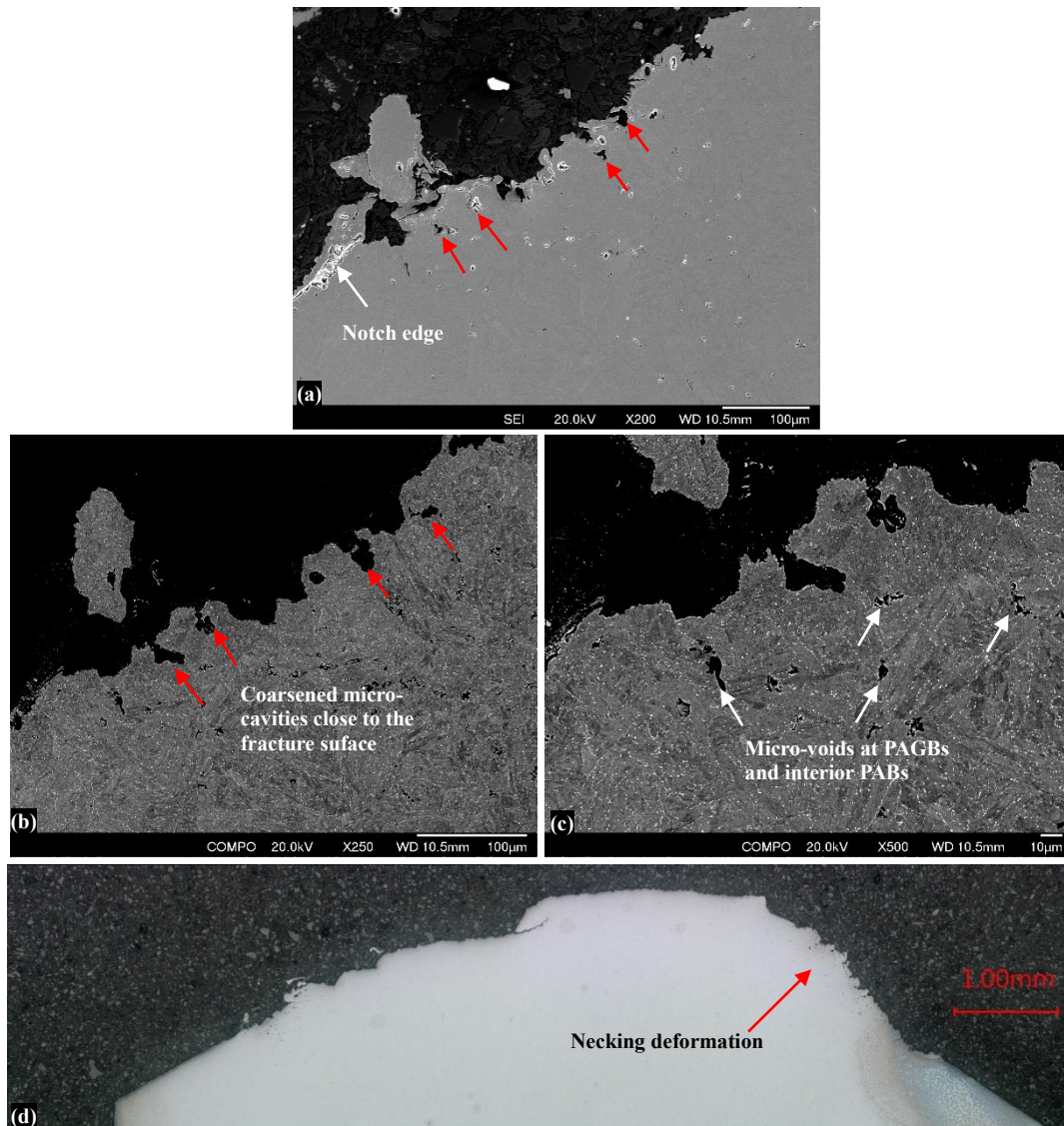
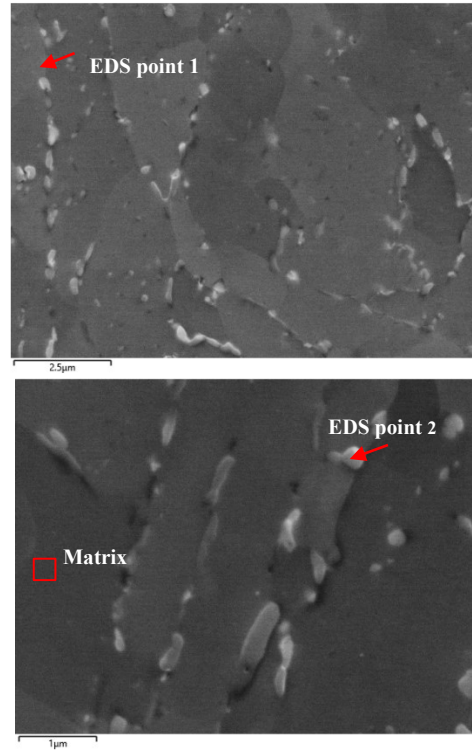


Figure 4.69 OM and SEM micrographs of cross section through C-BL1(150MPa, 650°C), (a)-(b) low magification image of CCG zone invloving notch edge, coarsened micro-cavities found close to the fracture surface; (c) high magnification image of the beginning stage of CCG; (d) optical micrograph (OM) indicatiing micr-voids primarily distributed within CCG region, necking deformation was found.



Spectrum	1	2	Matrix
Element	weight(%)		
C	18.63	15.31	15.31
N	/	13.4	/
Cr	7.98	25.16	5.96
Fe	61.66	43.01	78.72
Mo	2.29	0.44	/
W	9.45	1.11	/

Figure 4.70 SEM image showing precipitates along lath boundaries and EDS spectra of these precipitates in C-PL4.

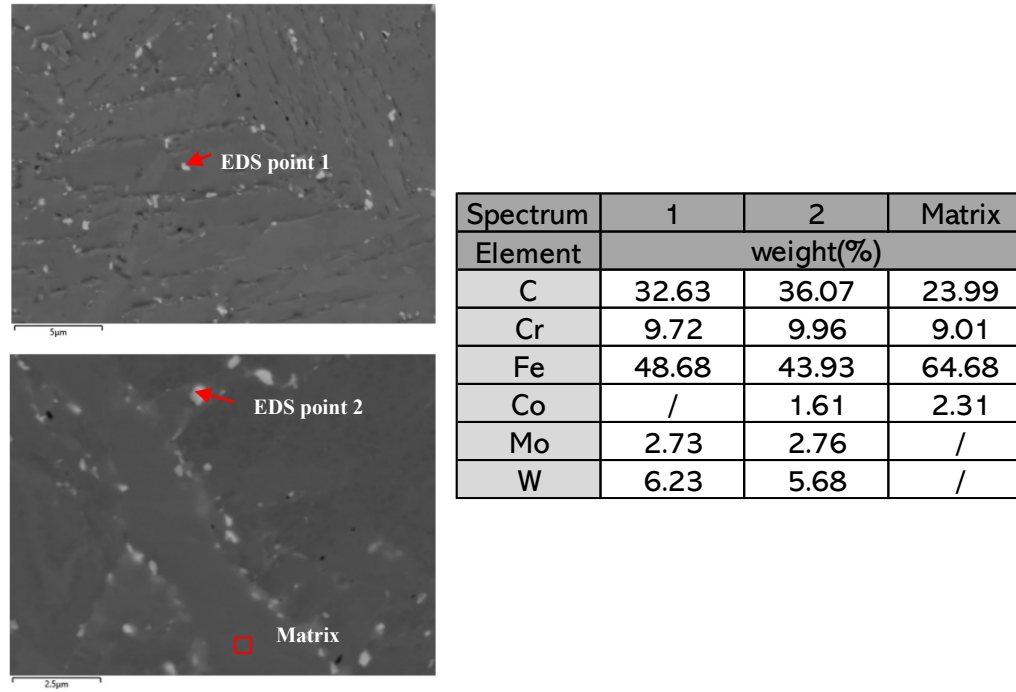


Figure 4.71 SEM image showing precipitates along lath boundaries and EDS spectra of these precipitates in C-BL1 indicating (Cr,W)-rich particles along boundaries and interior the grain with W content.

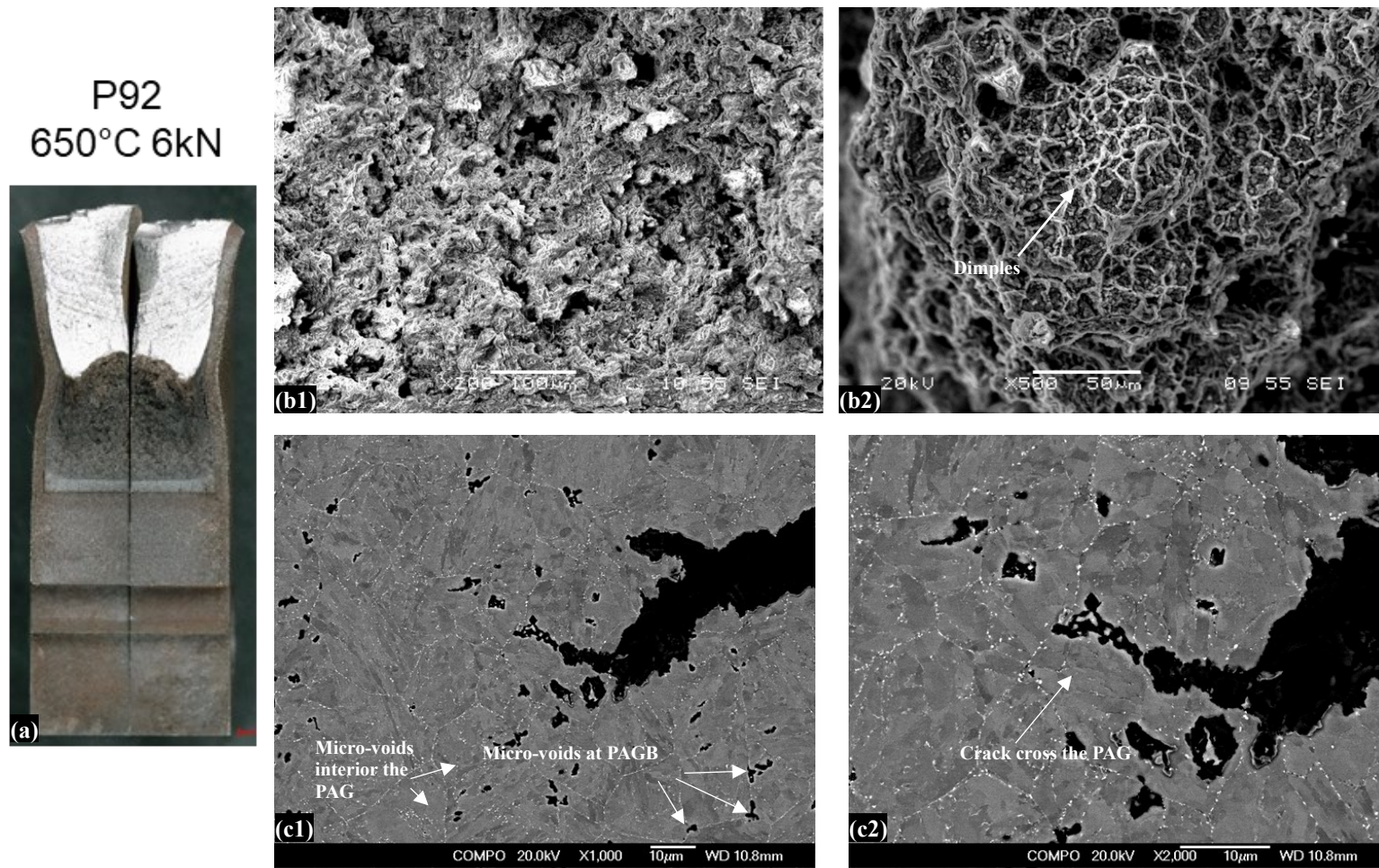


Figure 4.72 OM and SEM fractorgraphy, SEM images of SEM micrographs of cross section through a P92 CT testpiece (650°C, 6kN).

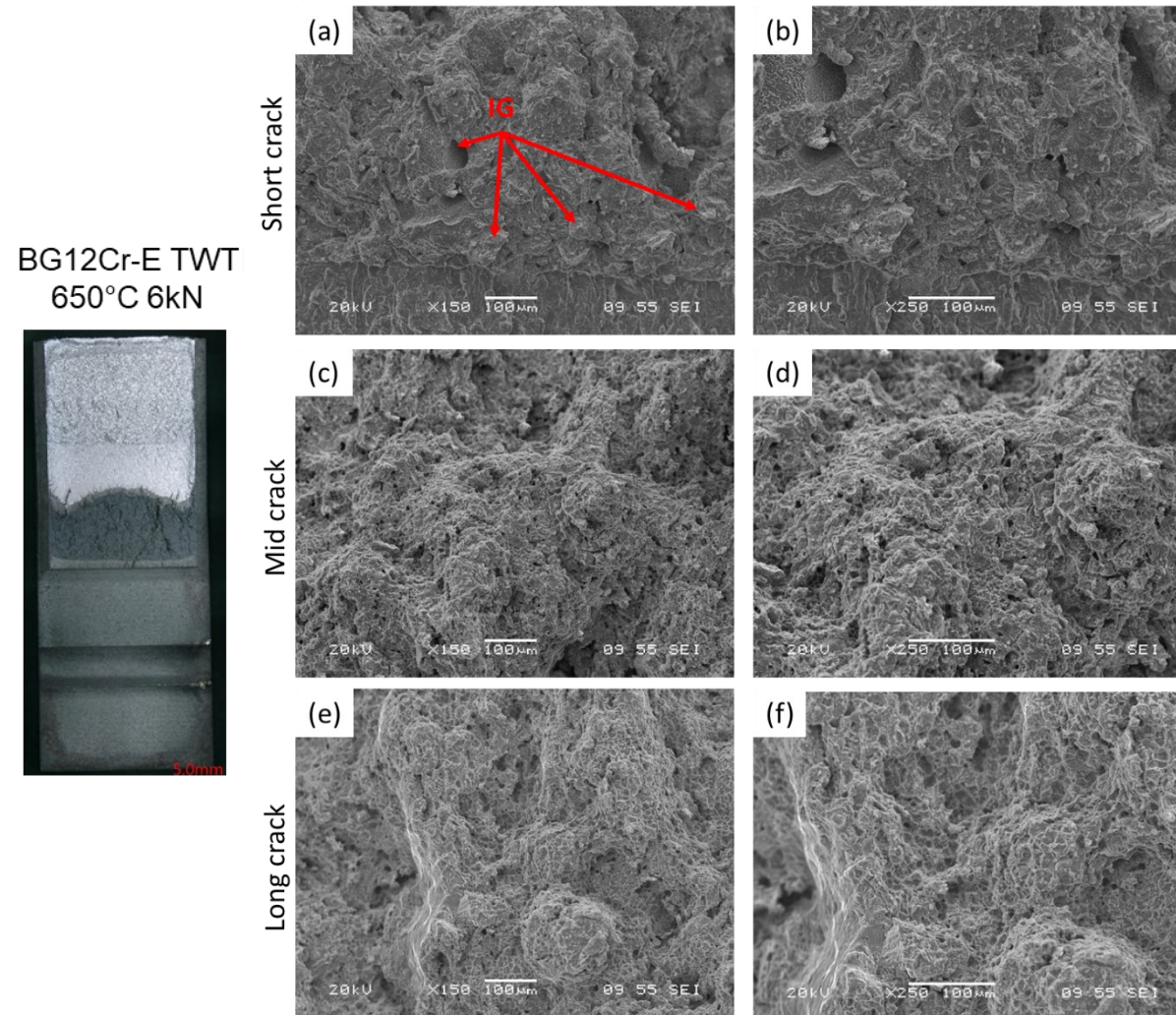


Figure 4.73 OM and SEM fractography of cross section through a BG12Cr CT testpiece indicating a failure mechanism transition from intergranular fracture to transgranular fracture(650°C, 6 kN).

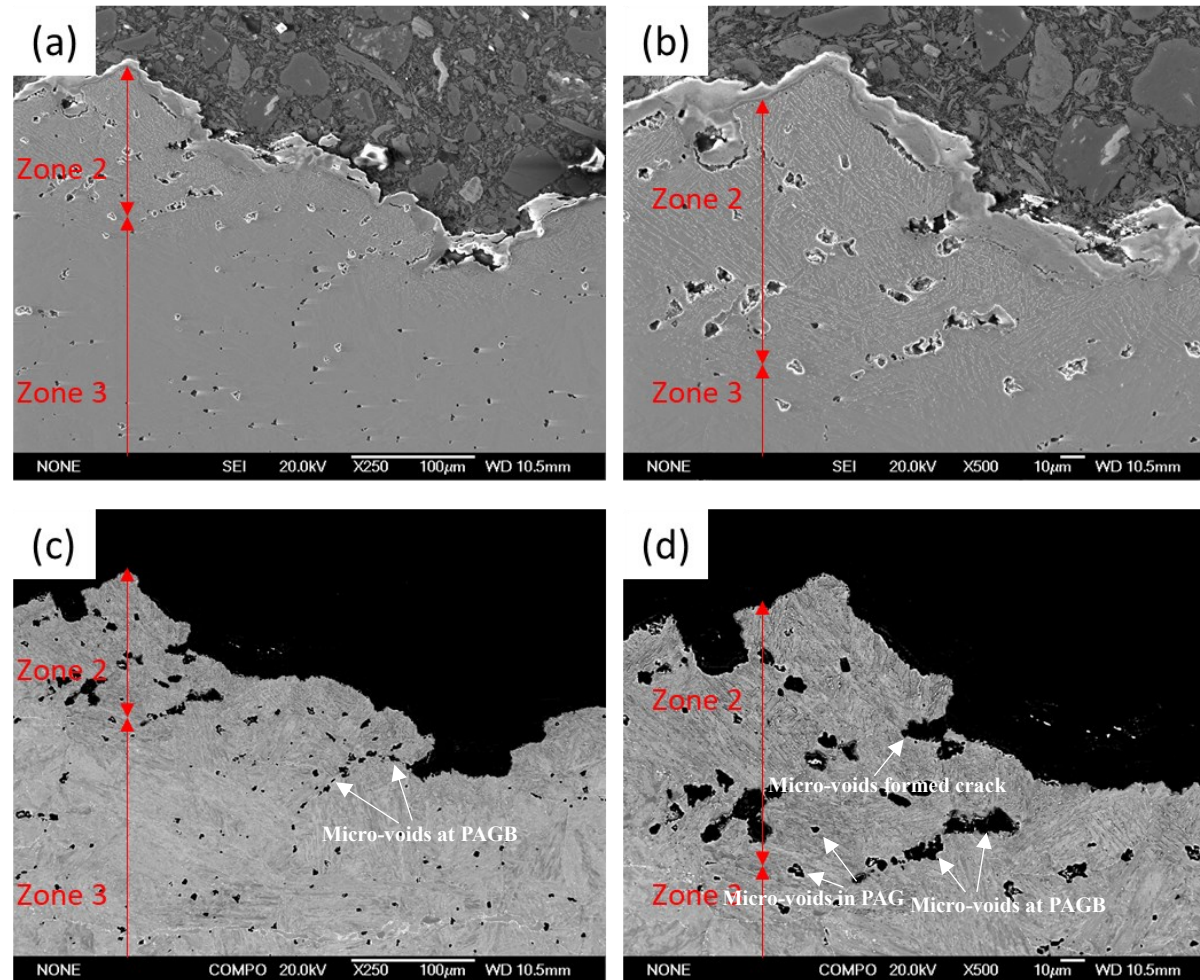


Figure 4.74 Secondary and backscatter electron images of the cross-section of the initial short crack in BG12Cr-E TWT tested at 650°C with 6 kN(in CT geometry, the results was reprocessed with the agreement of the author-Ke Liu).

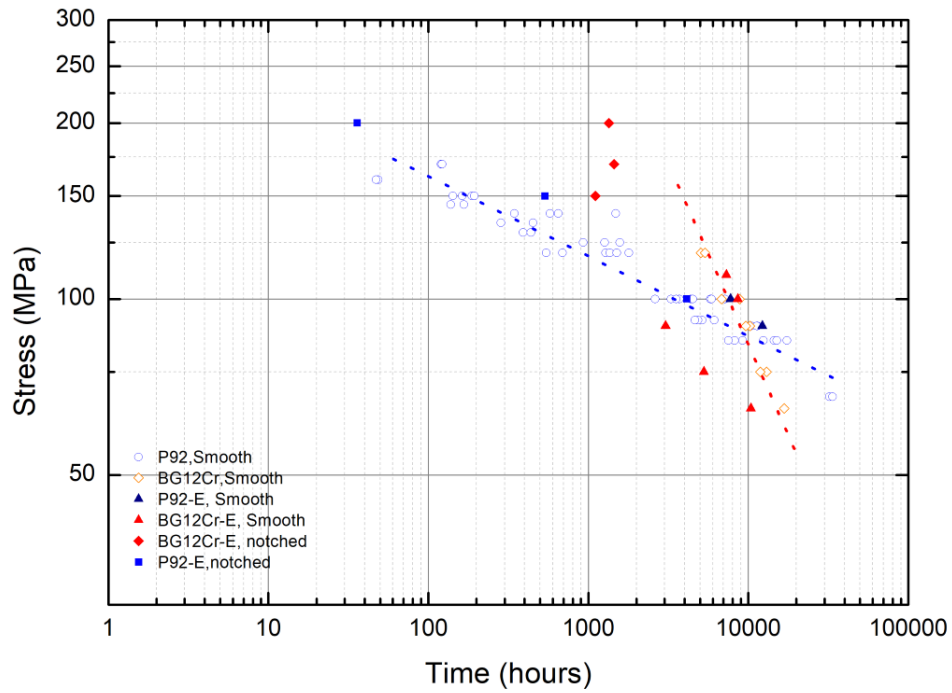


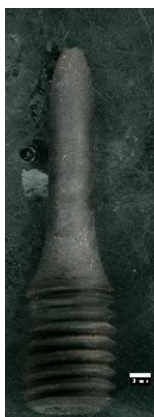
Figure 4.75 Stress-rupture curves of P92 and BG12Cr at 650°C with different stress levels (Except notched specimens' data, the others were supported by Baowu Steel).



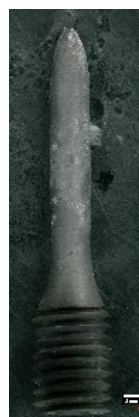
P92-E-90MPa



P92-E-100MPa



BG12Cr-E-65MPa



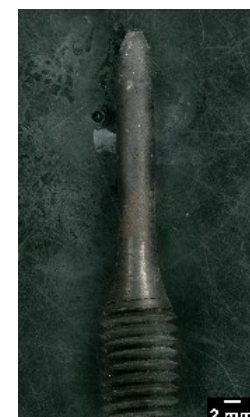
BG12Cr-E-75MPa



BG12Cr-E-90MPa

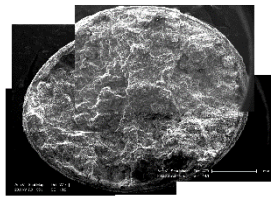


BG12Cr-E-100MPa



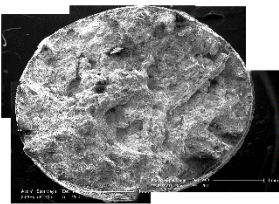
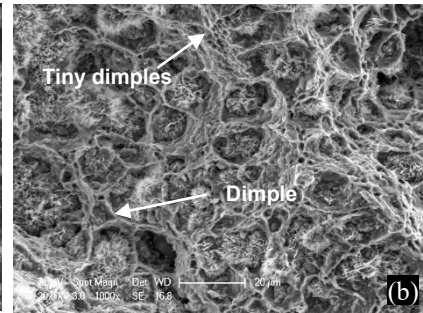
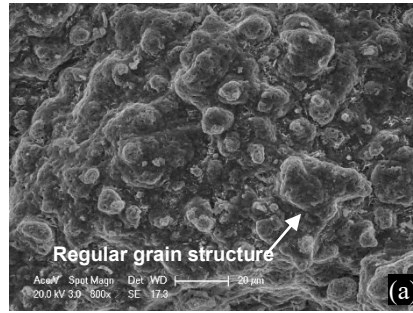
BG12Cr-E-110MPa

Figure 4.76 Optical photography of received stress-ruptured testpieces supported by Baowu Steel.



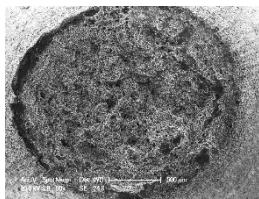
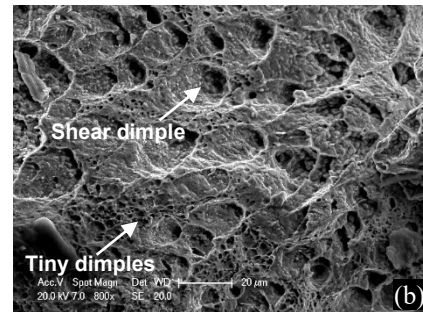
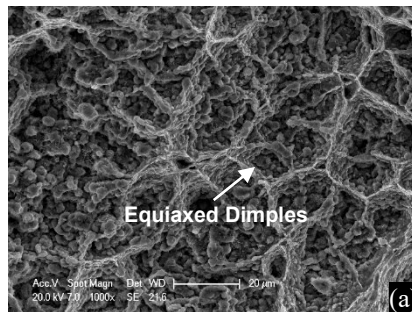
P92-E-90MPa

(1)



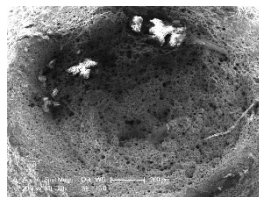
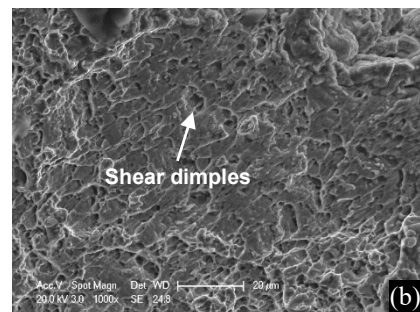
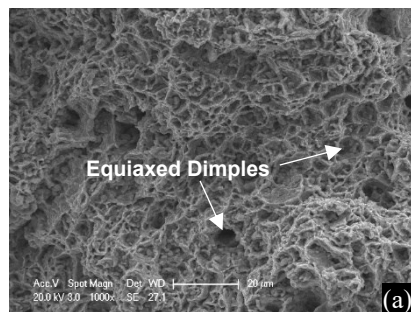
P92-E-100MPa

(2)



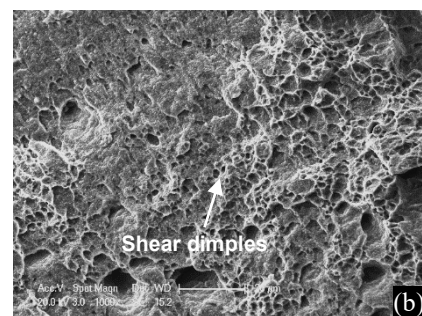
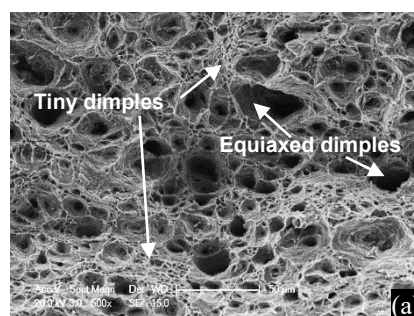
BG12Cr-E-65MPa

(3)



BG12Cr-E-75MPa

(4)



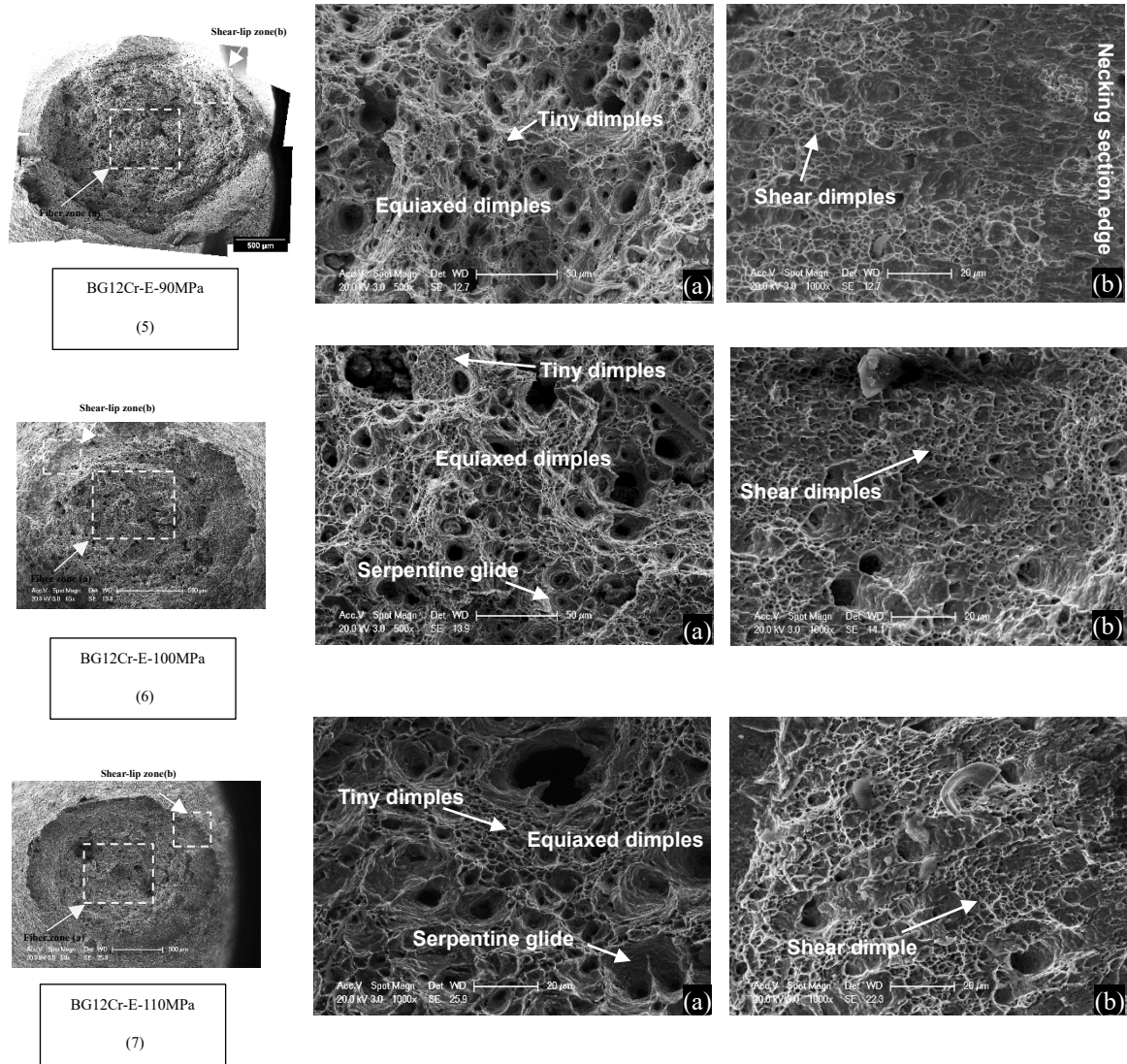


Figure 4.77 SEM fracture surface images of all the stress rupture testpieces tested at 650°C under different stress levels: (1)-(2): SEM fractography of P92 specimens, no necking deformation observed, (a): magnified view of the fibre zone, (b): magnified image showing shear dimples ; (3)-(7) SEM fractography of BG12Cr specimens macro view presenting elliptical necking, (a) magnified view of the fibre zone showing equiaxed dimples, (b) magnified image showing shear dimples characteristic on the shear-lip zone.

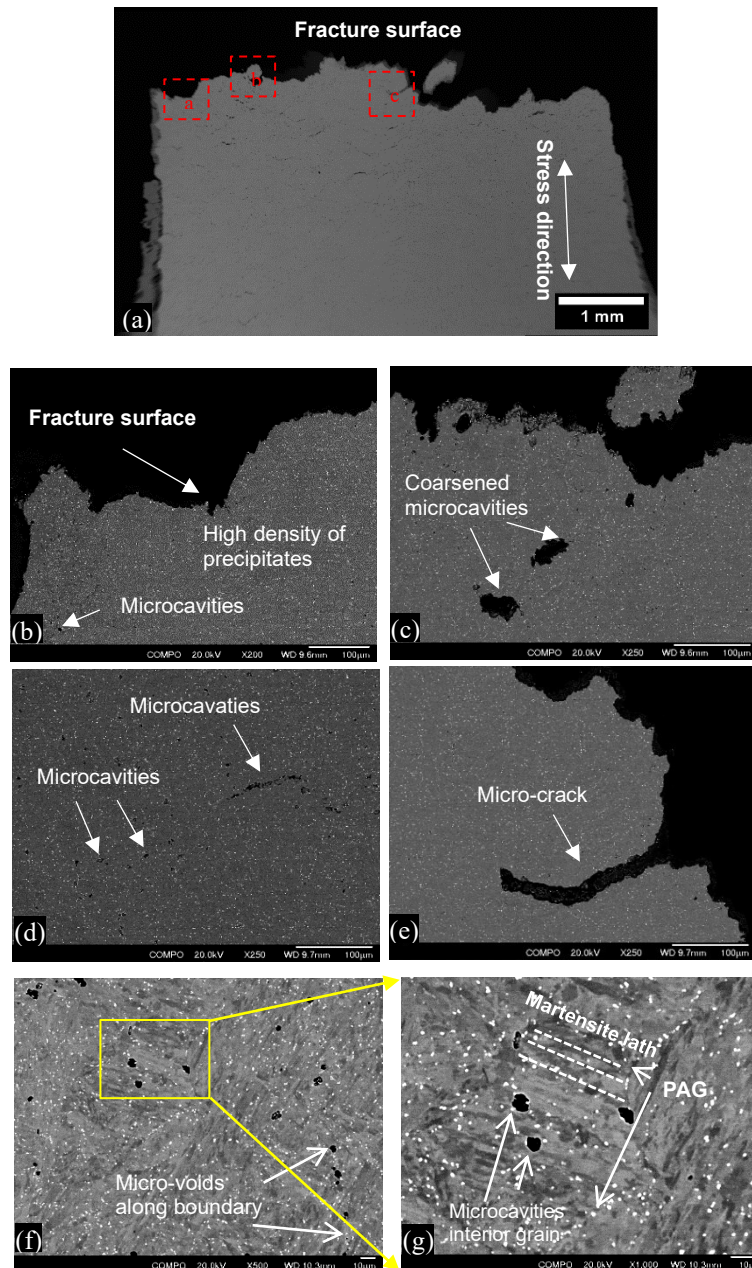


Figure 4.78 Backscattered SEM micrographs taken from a longitudinal cross-section of a P92 stress-ruptured sample tested at 650°C, 90 MPa, (a): low magnification of cross-sectioned specimen; (b): magnified view of area-a, (c): magnified view of area-b consisting of coarsened microcavities close to the fracture surface, (d): magnified view of microcavities in area-b, (d): magnified view of micro-crack in region-c, (f)-(g): magnified views of microcavities along boundaries and the interior the grains far away from fracture surface.

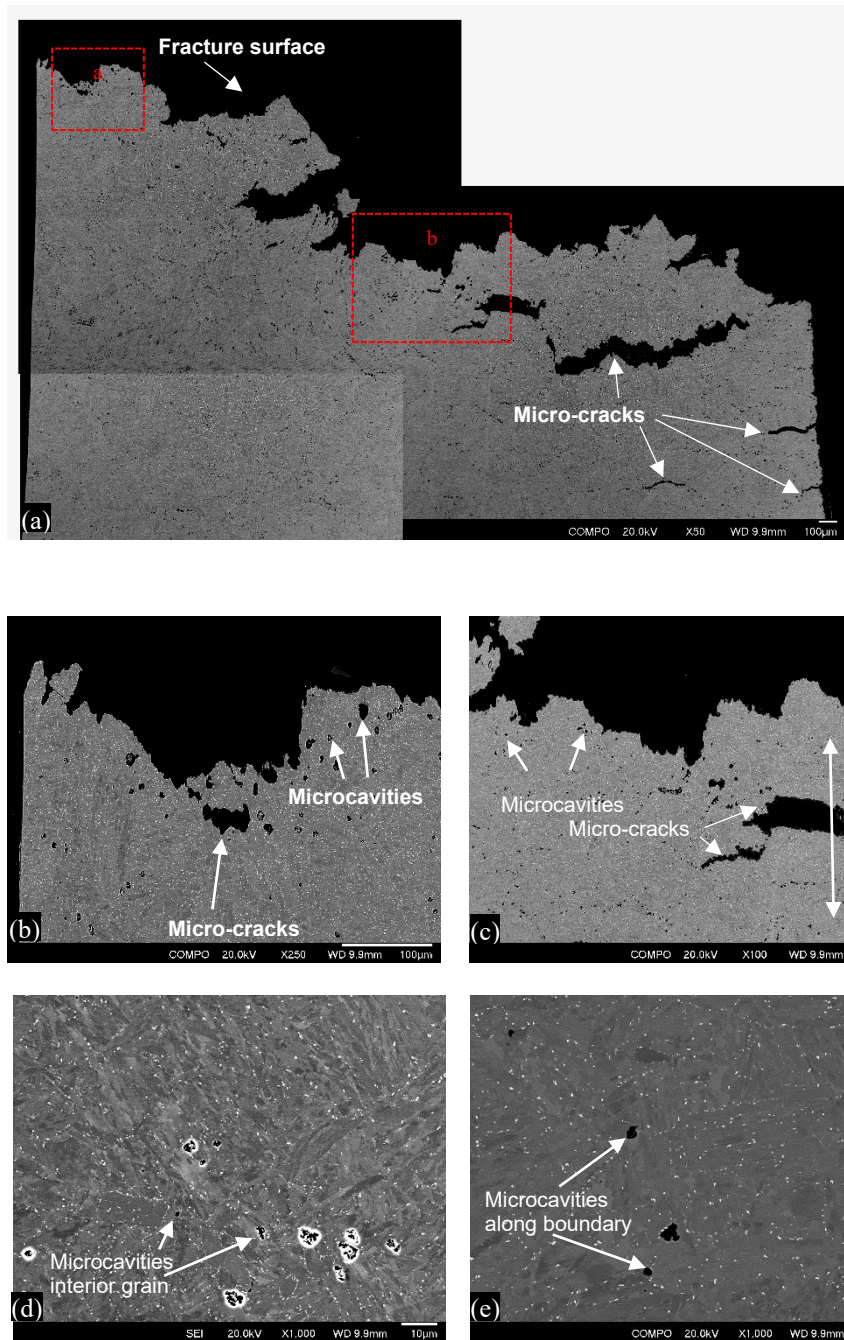


Figure 4.79 Backscattered SEM micrographs taken from a longitudinal cross-section of a P92 stress-ruptured sample tested at 649°C, 100 MPa. (a): low magnification of cross-sectioned specimen; (b): micro-voids along grain boundaries found close to the fracture surface; (c): micro-voids found close to the fracture surface (white arrow) and sheared micro-voids close to the fracture surface; (d): a micro crack below the fracture surface; (e): micro-voids along grain boundaries far away from the fracture surface.

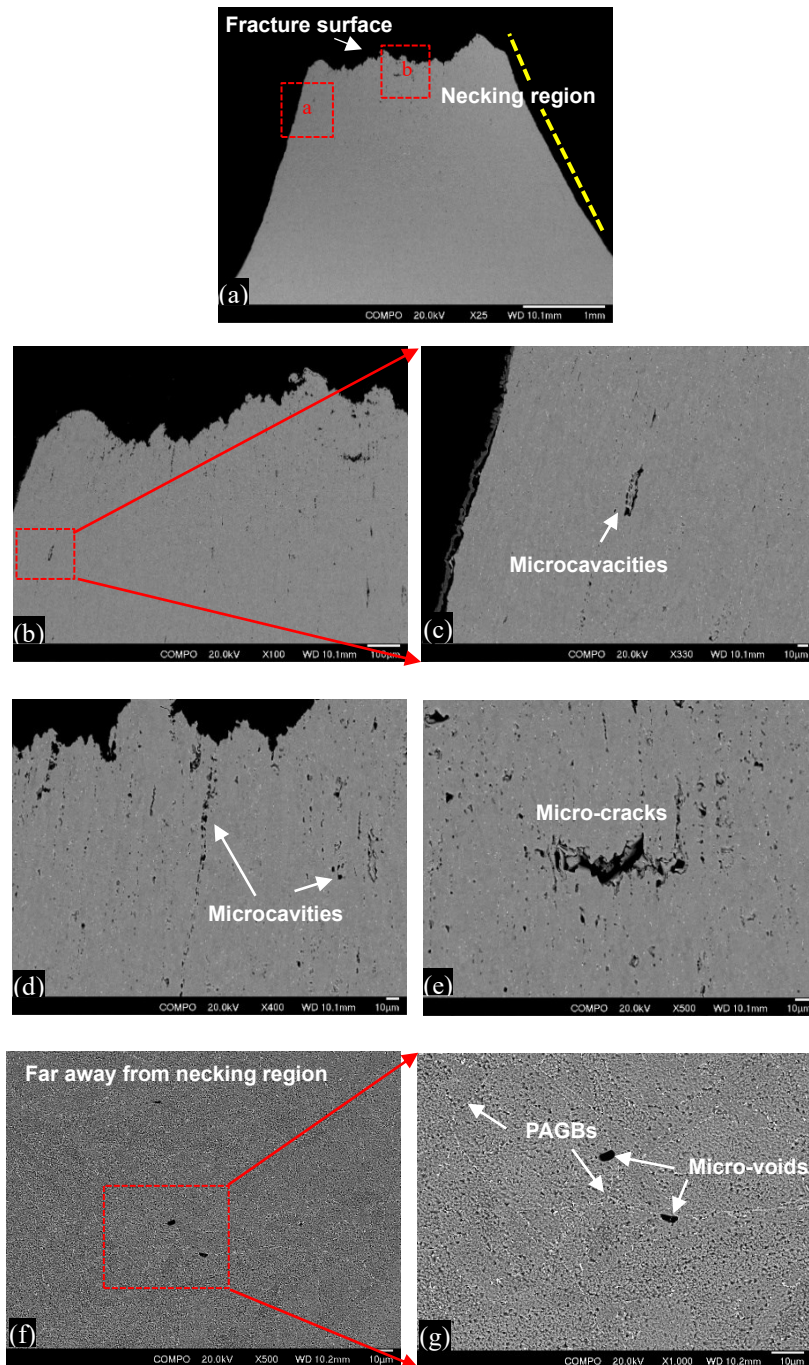


Figure 4.80 Backscattered SEM micrographs taken from a longitudinal cross-section of a BG12Cr stress-ruptured sample tested at 650°C, 65 MPa. (a): low magnification of cross-sectioned specimen; (b): Close-up of regions close to the fracture surface, micro-cracks and microcavities were found (see arrows); (c): martensite cracking far away from the fracture surface; (d): dense microcavities close to the fracture surface; (e): close-up of micro-crack with width of ~50 μm .

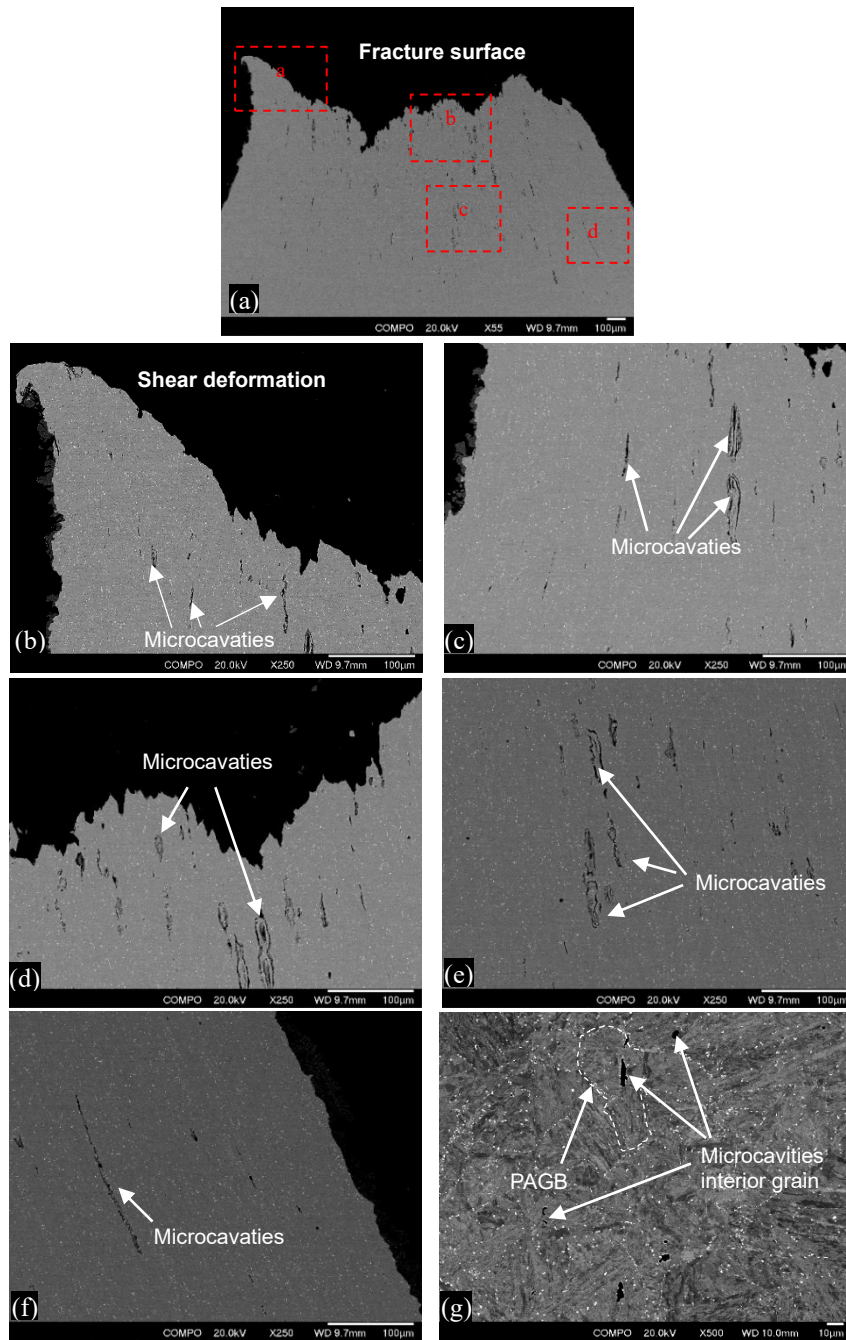


Figure 4.81 Backscattered SEM micrographs taken from a longitudinal cross-section of a BG12Cr stress-ruptured sample tested at 650°C, 75 MPa. (a): low magnification of cross-sectioned specimen; (b)-(c): close-up of region-a, microcavities can be observed close to fracture surface; (d)-(e): magnified view of areas below fibre zone showing dense microcavities; (f): magnified view of microcavities far away from the fracture surface; (g): magnified view of microcavities in the interior of a grain far away from the fracture surface.

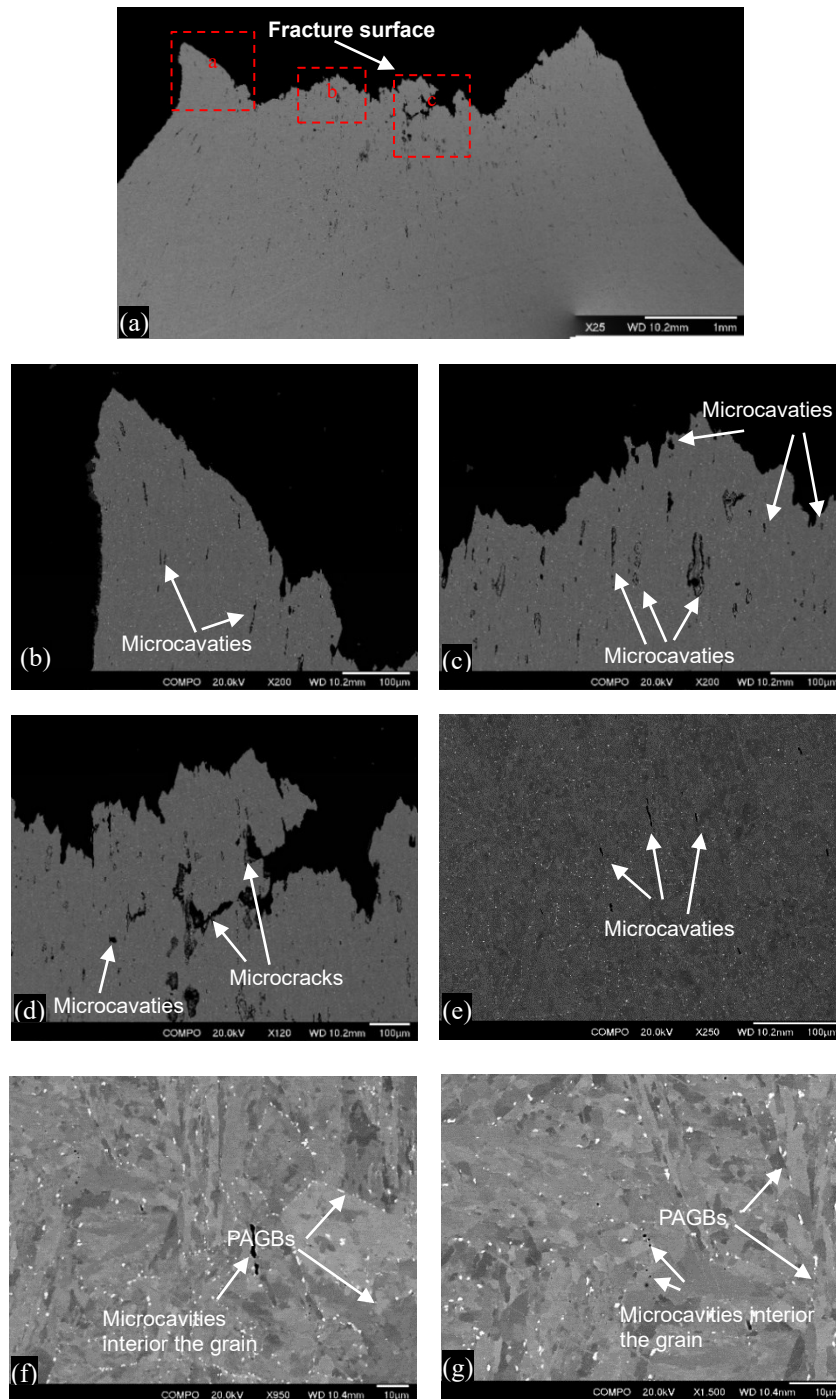


Figure 4.82 Backscattered SEM micrographs taken from a longitudinal cross-section of a BG12Cr stress-ruptured sample tested at 650°C, 90 MPa, (a): low magnification of cross-sectioned specimen; (b): close-up of area-a; (c): close-up of area-b; (d): close-up of area-c: dense coarsened microcavities; (e) magnified view of region below area-c; (f)-(g), magnified view of region far away from the deformed region: microcavities formed in the interior the PAGs.

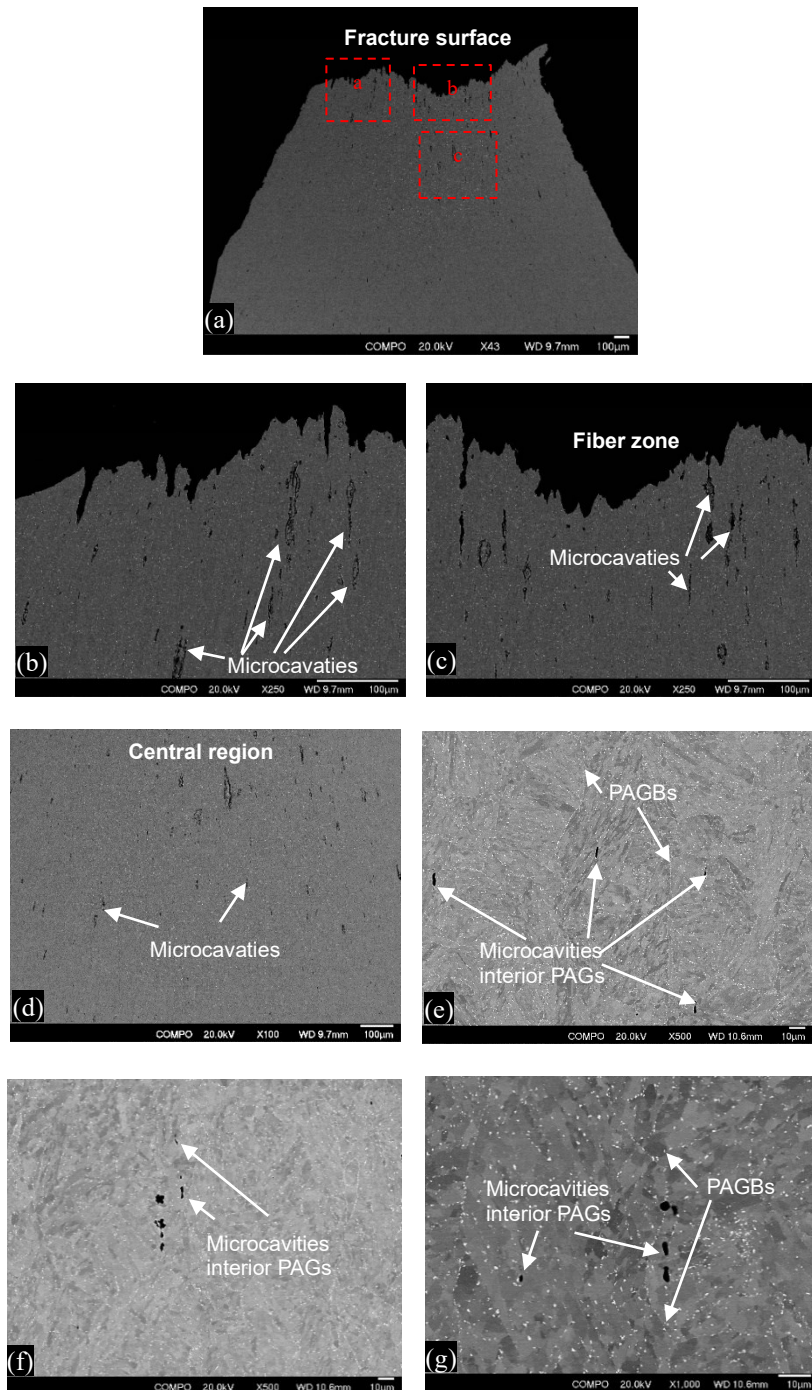


Figure 4.83 BSE images taken from a longitudinal cross-section of a BG12Cr stress-ruptured sample tested at 650°C, 100 MPa, (a): low magnification of cross-sectioned specimen; (b): magnified view of region-a: microcavities close to fracture surface; (c): close-up image of region-b: dense microcavities; (d): close-up of region-c, micro-voids found far away from fracture surface; (e)-(g): magnified view of regions far away from the fracture surface: microcavities in the interior of a grain.

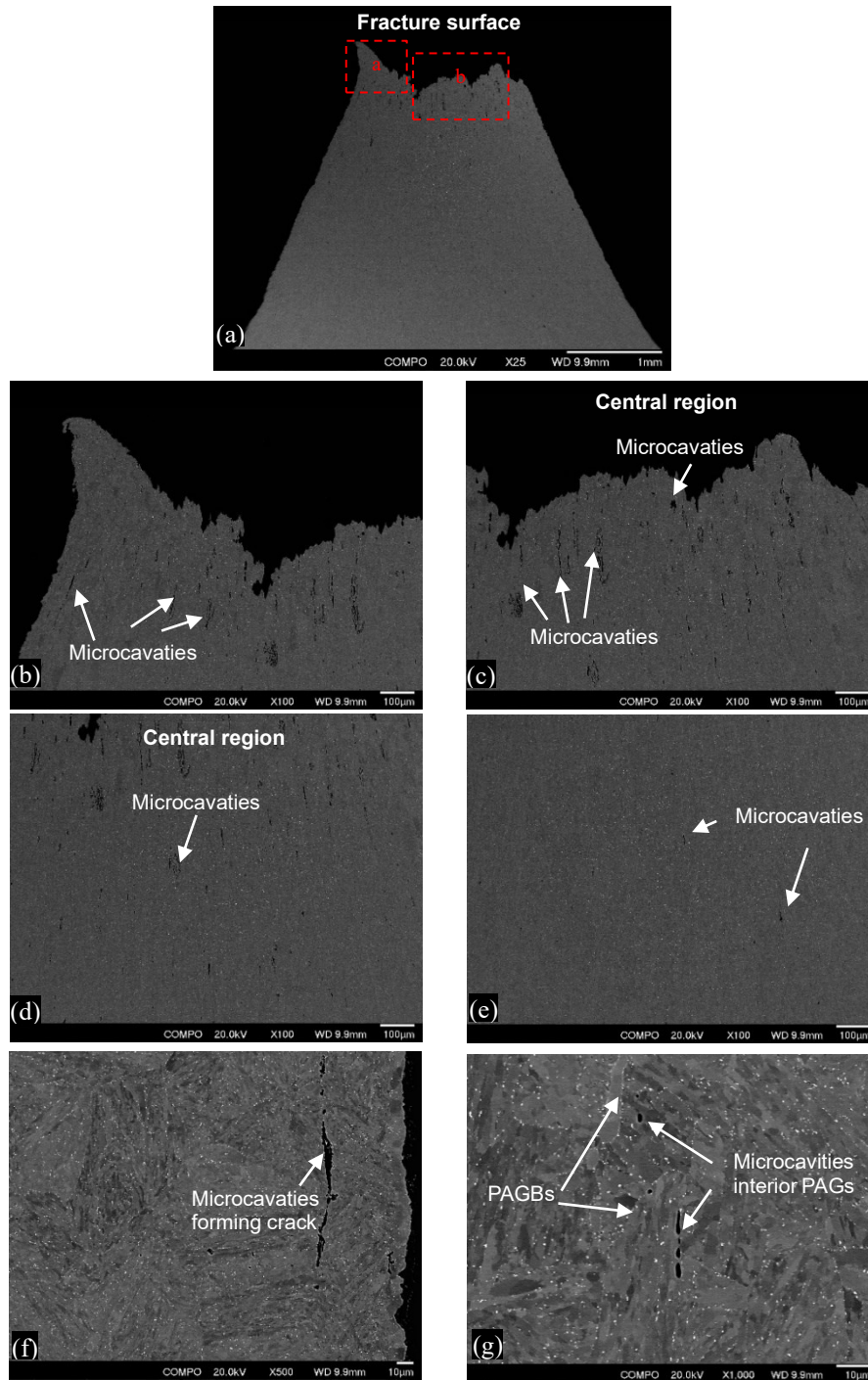


Figure 4.84 BSE images taken from a longitudinal cross-section of a BG12Cr stress-ruptured sample tested at 625°C, 110 MPa, (a): low magnification of cross-sectioned specimen; (b): magnified view of region-a: dense microcavities; (c)-(e): magnified views of region-b: dense microcavities in the central region; (f)-(g) magnified views of a region far away from fracture surface: microcavities close to the edge of the specimen and microcavities formed inside PAGs.

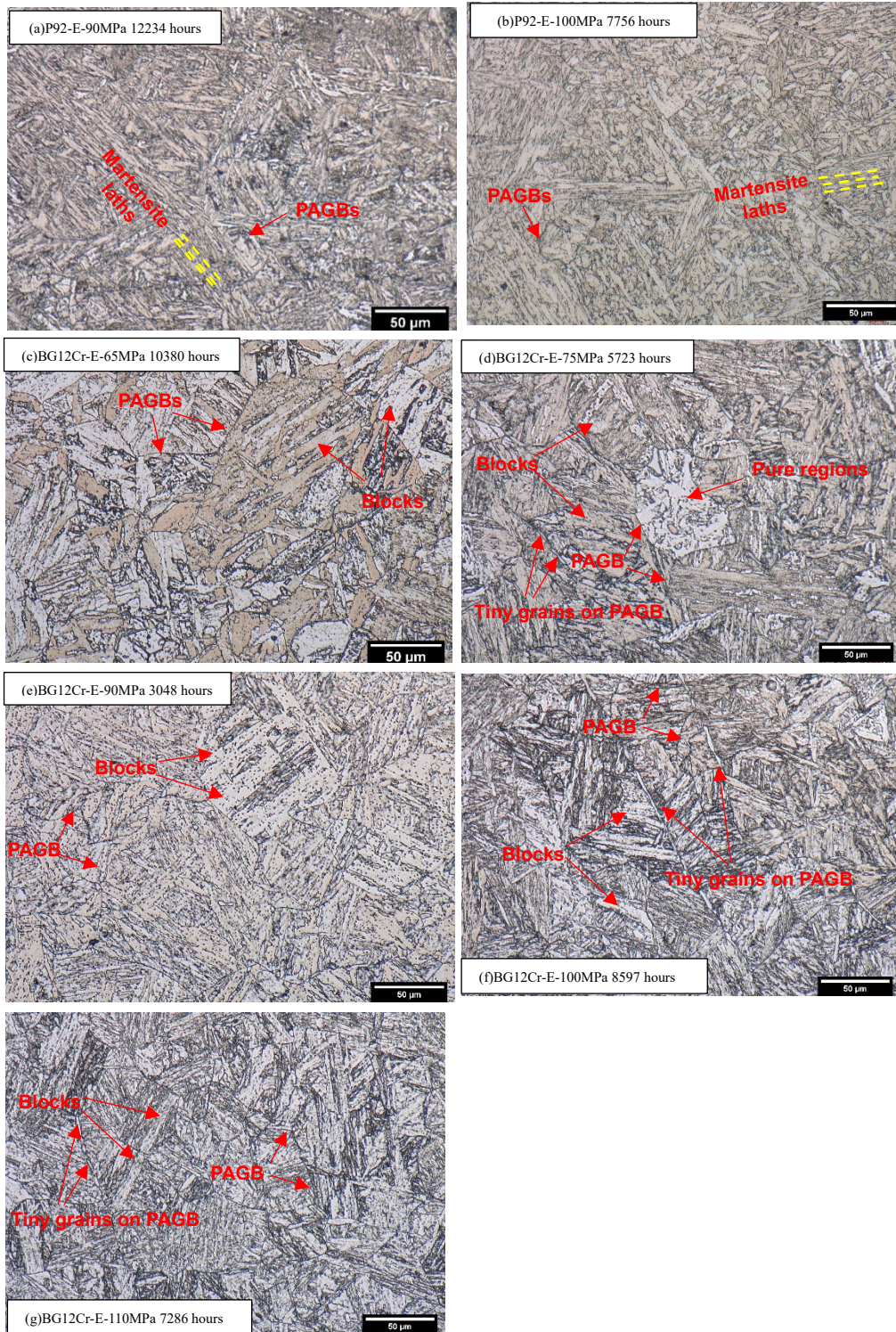
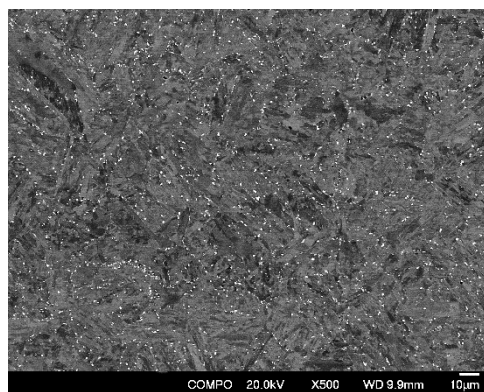
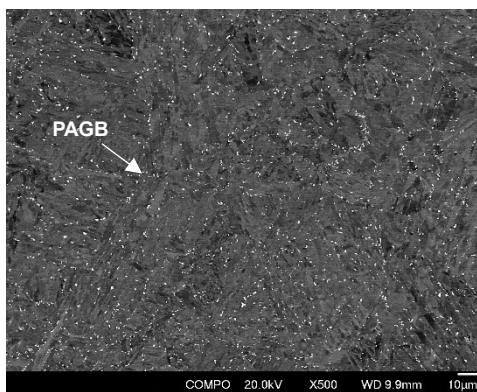


Figure 4.85 Optical micrographs of thread regions from long-term stress-ruptured samples tested at 650°C

(1)P92-E-90MPa
Tr: 12234 hours

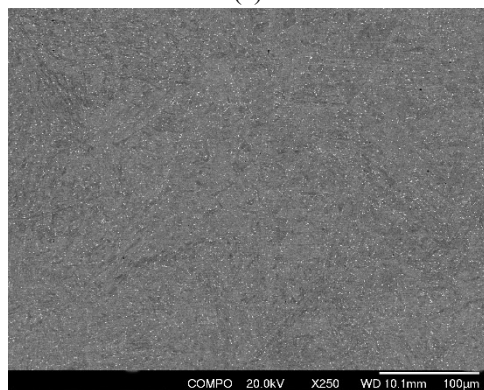


(a)

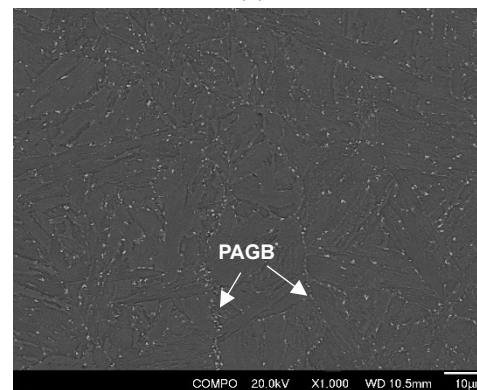


(b)

(2)P92-E-100MPa
Tr: 7756 hours

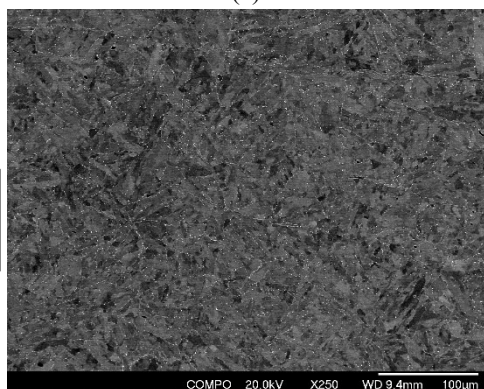


(a)

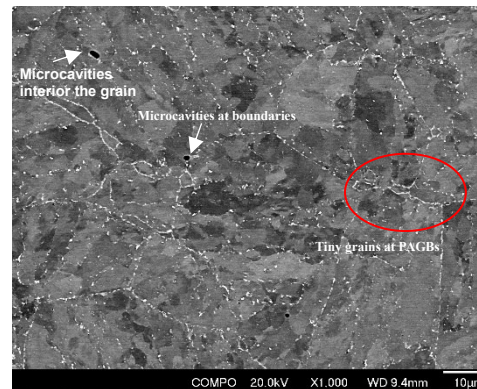


(b)

(3) BG12Cr-E-65MPa
Tr: 10380 hours



(a)

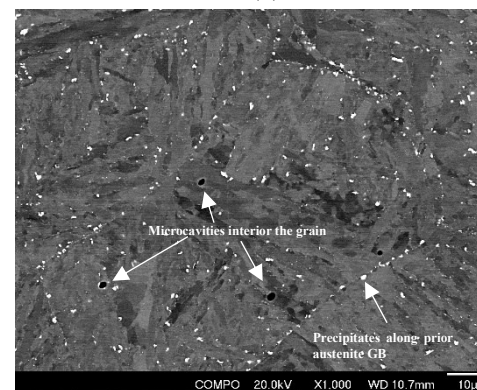


(b)

(4) BG12Cr-E-75MPa
Tr: 5723 hours



(a)



(b)

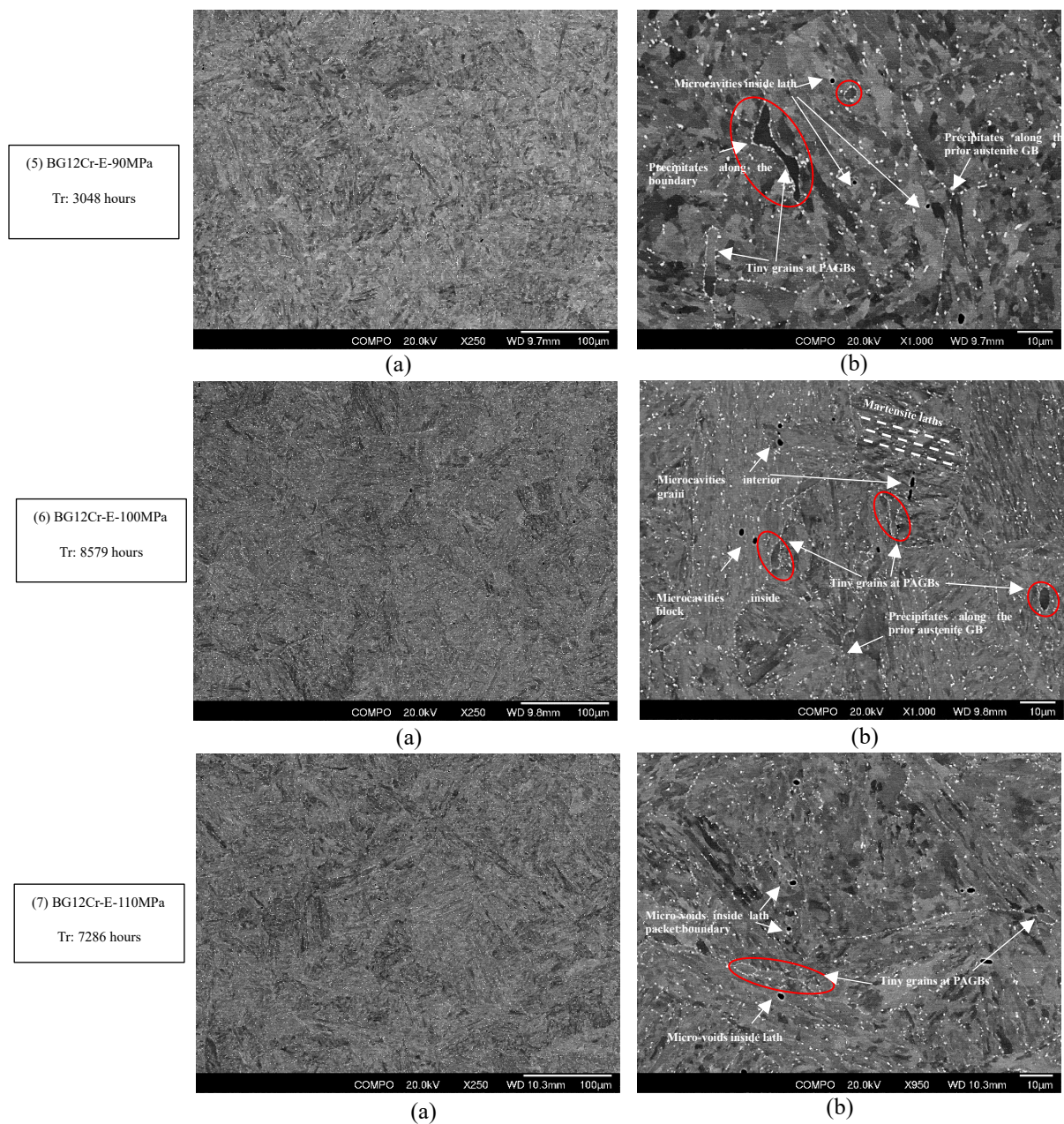


Figure 4.86 BSE images taken from the underformed regions of all the stress-ruptured samples.

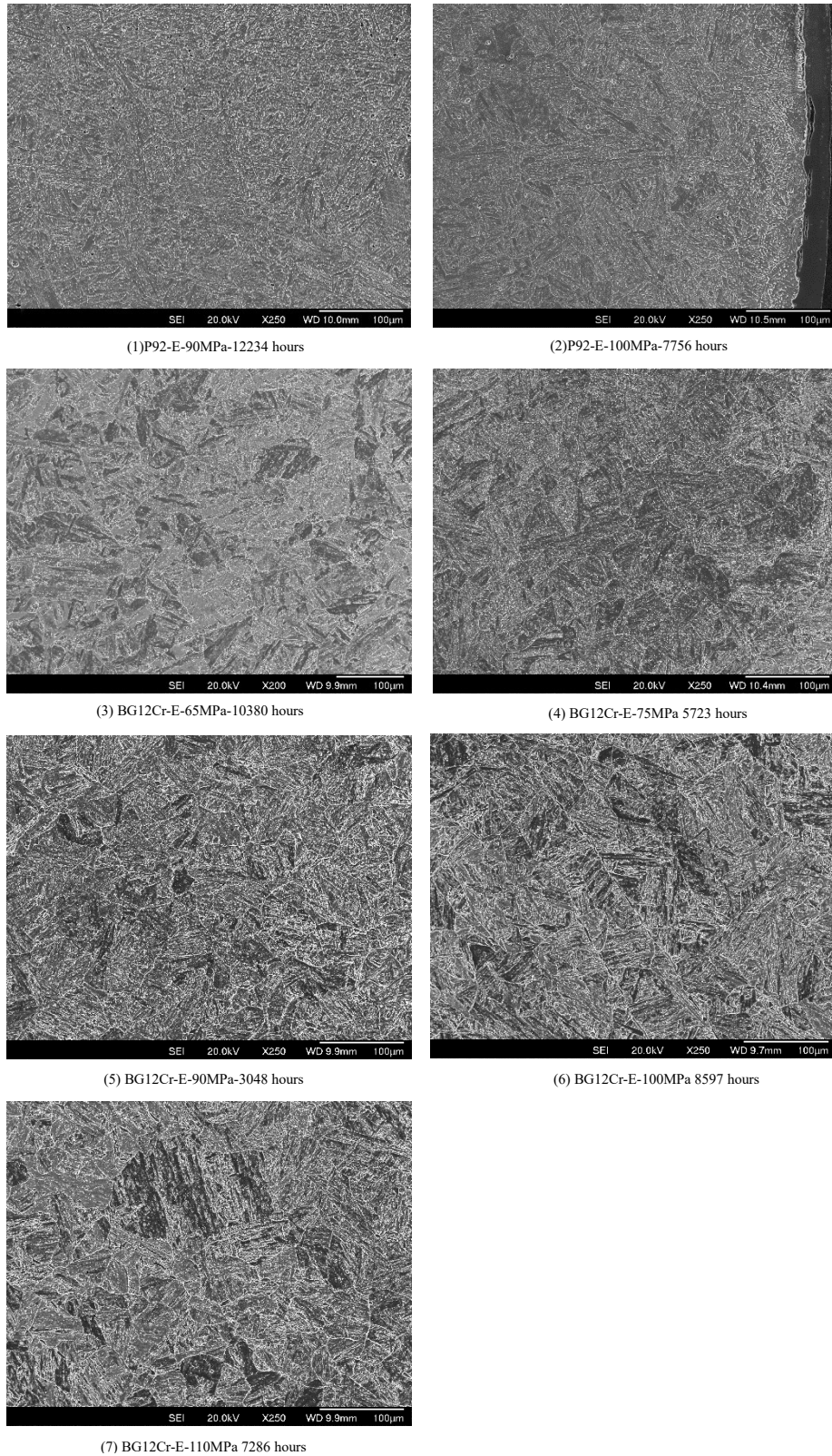


Figure 4.87 SE images taken from the undeformed regions of all the stress-ruptured samples after etching.

Chapter 5

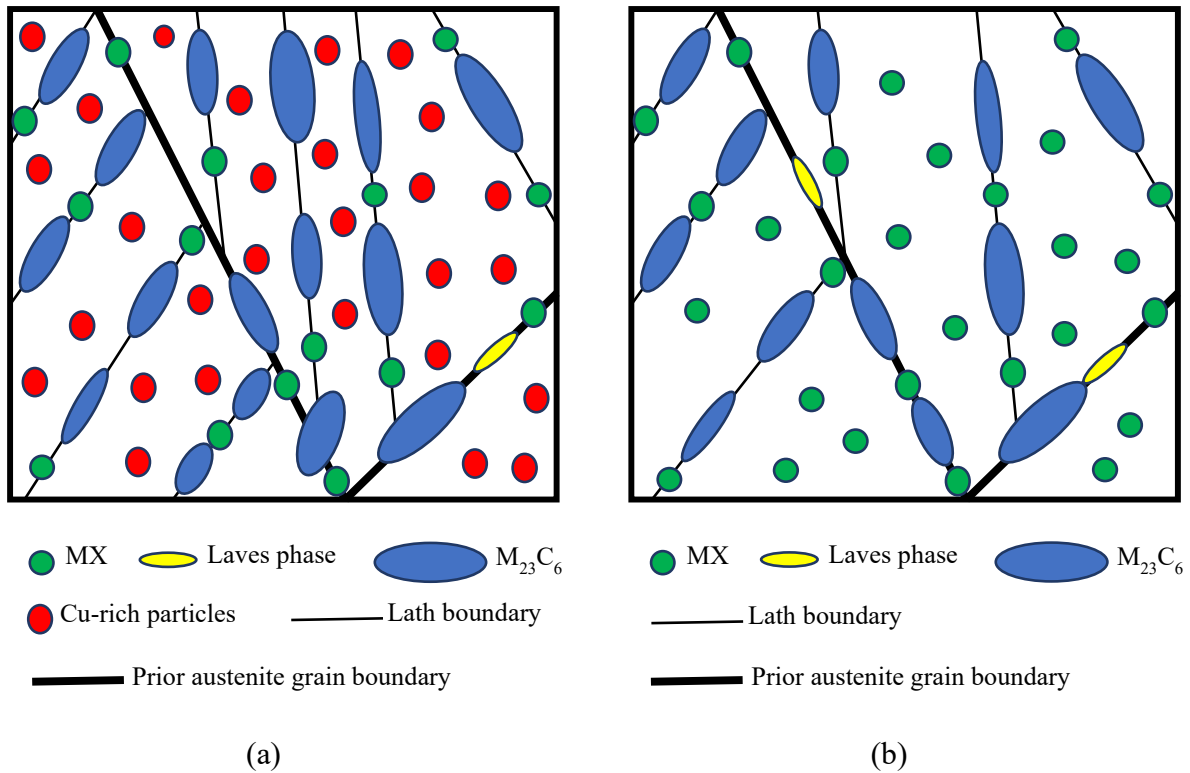


Figure 5.1 Schematic drawing of the as-received martensite heat resistant steels microstructure, (a) as-received BG12Cr, (b) as-received P92.

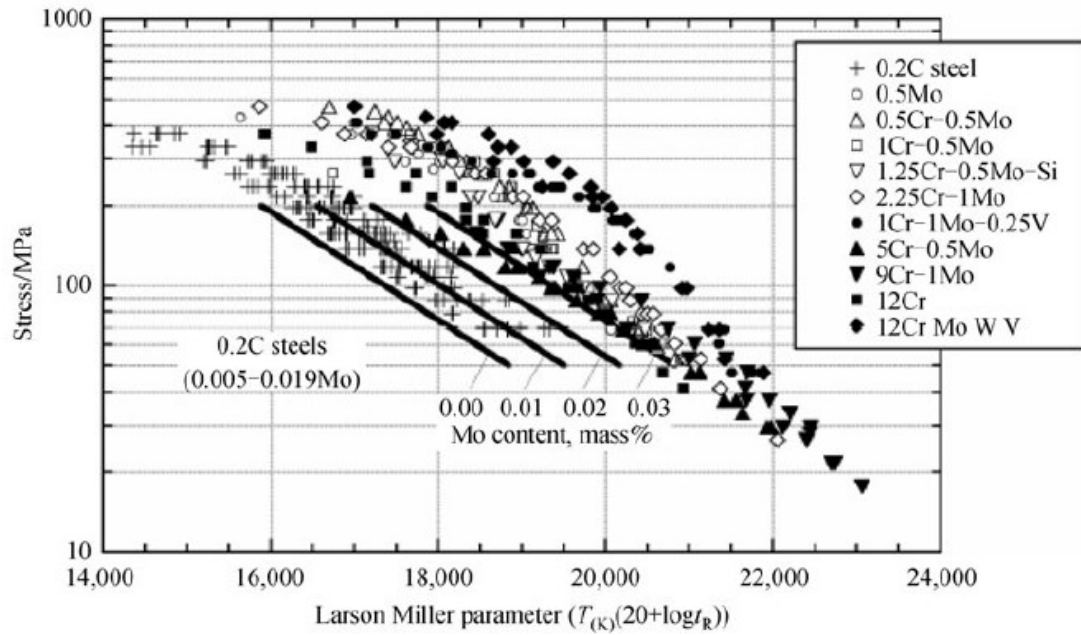


Figure 5.2 Creep rupture strength versus Larson Miller parameter curves of different ferritic heat-resistant steels [31-32].

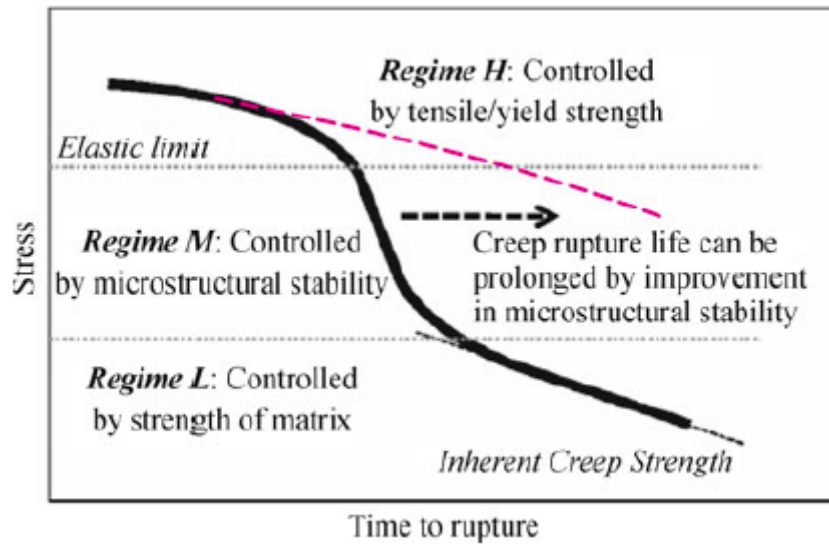


Figure 5.3 Schematic drawing of different regimes in creep strength mechanism map [31-32].

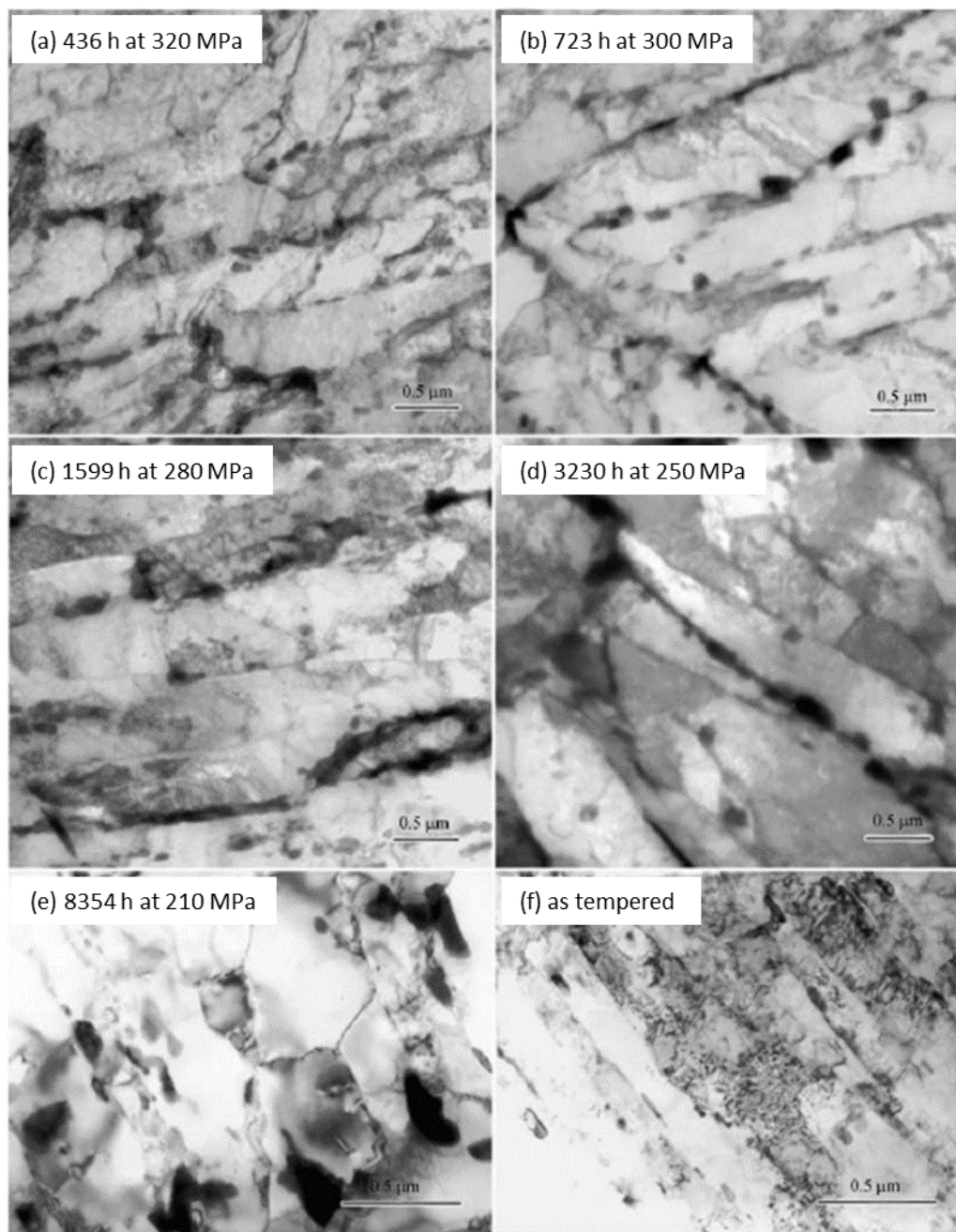


Figure 5.4 TEM images of the 10%Cr steel under 600°C with different stress levels [33].

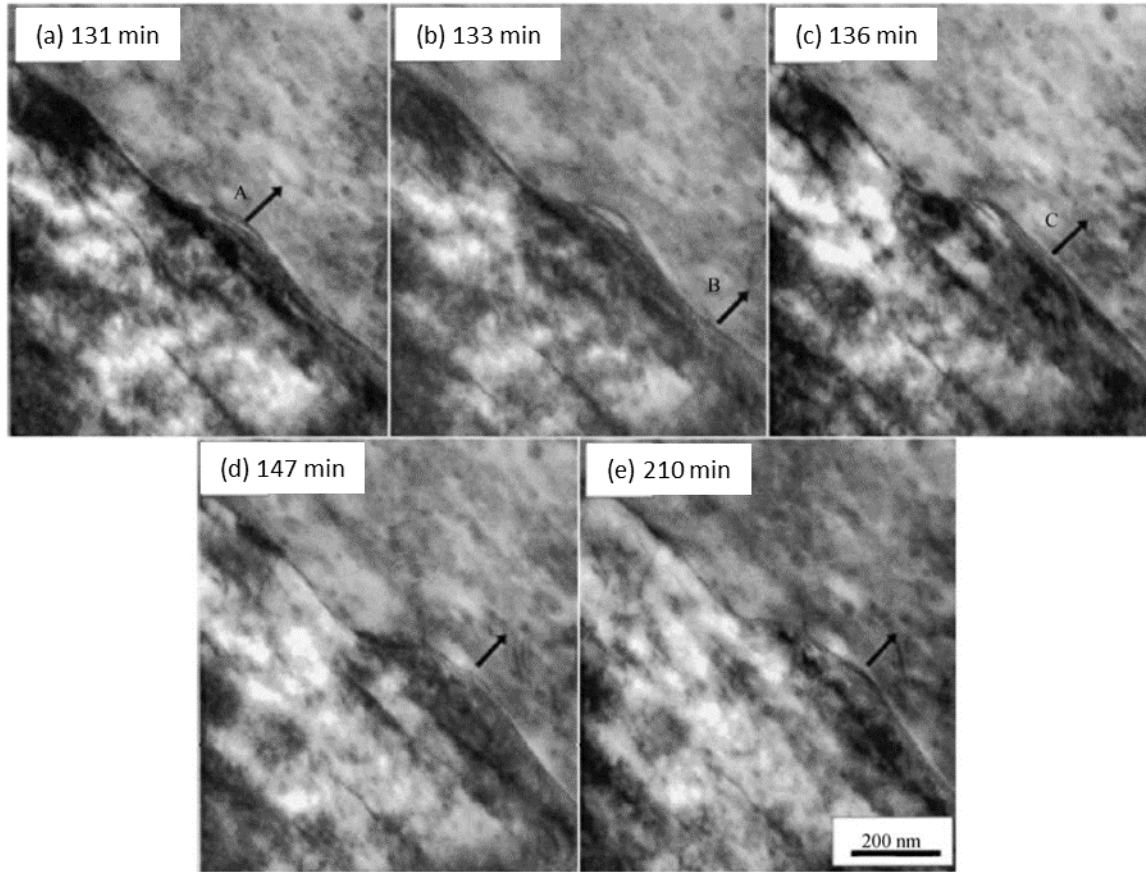


Figure 5.5 TEM micrographs presenting lath booundary migration behaviour during tempering at 667°C with different time range from 131 min to 210 min [33, 34].

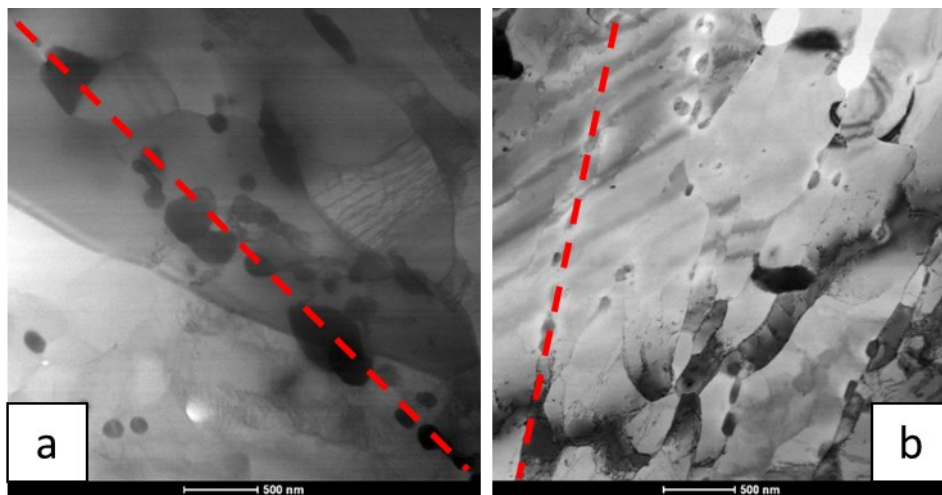


Figure 5.6 TEM images showing the disappearance of PAGBs in: a) PT BG12Cr, b) PT P92.

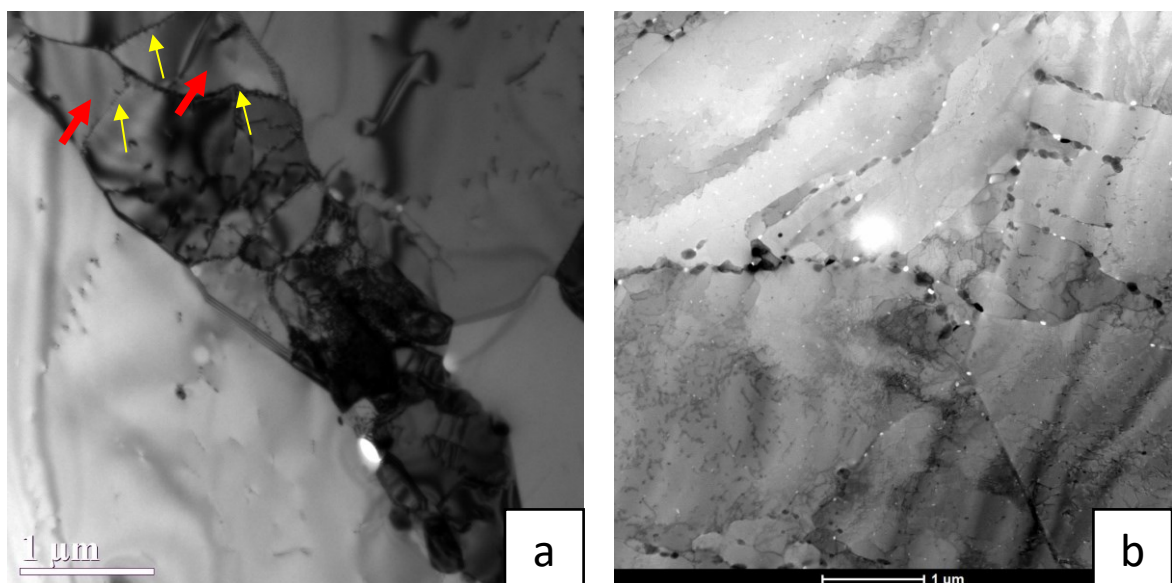


Figure 5.7 TEM images showing the emergence of subgrains in: a) PT BG12Cr, b) AS BG12Cr.

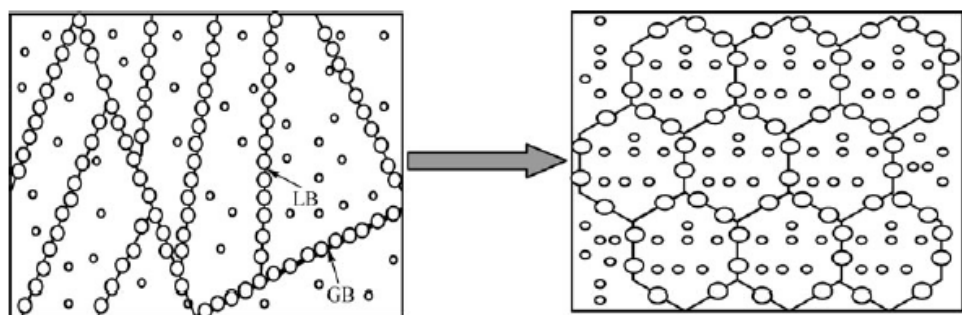


Figure 5.8 Schematic of martensitic laths to subgrains transformation mechanism [33].

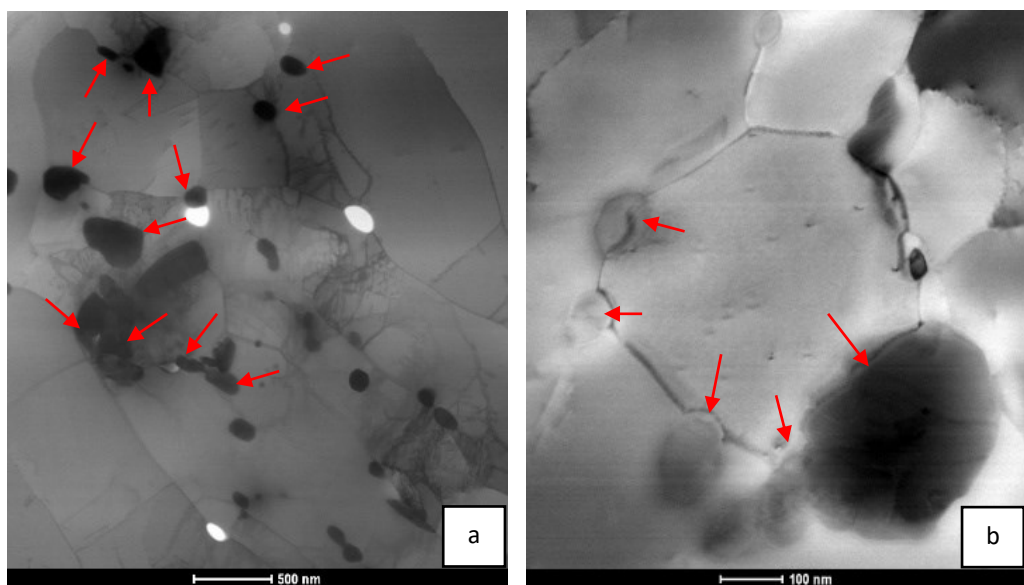


Figure 5.9 TEM images showing the pinning precipitates ($M_{23}C_6$) on subgrain boundaries in: a) PT BG12Cr, b) AS BG12Cr.

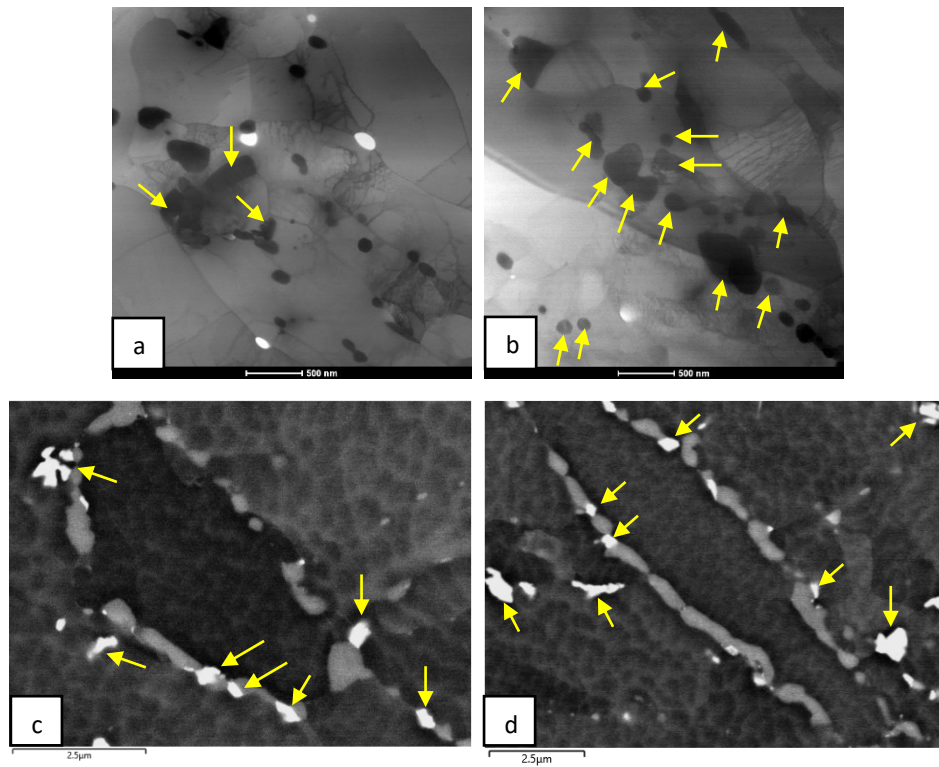


Figure 5.10 TEM images showing the precipitation of Laves phase boundaries in: (a)-(b) PT BG12Cr, EDS micrographs showing (W,Mo)-rich Laves phase distribution around some small grain boundaries in PT BG12Cr.

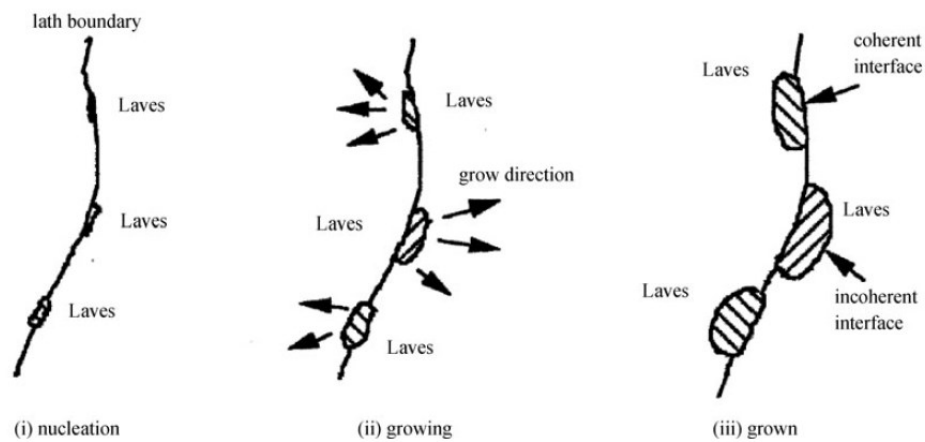


Figure 5.11 Schematic drawing of Laves phase precipitation mechanism showing three stages (nucleation stage, growth stage and grown stage) [33].

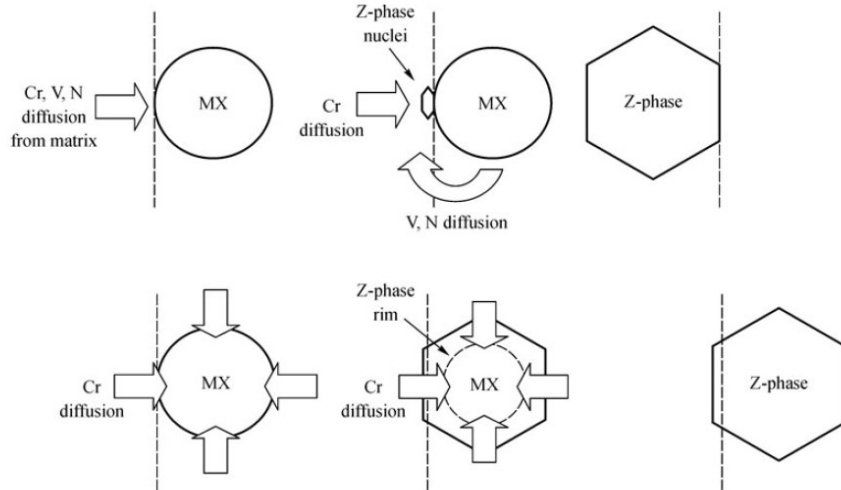


Figure 5.12 Schematic illustration of Z-phase nucleation possible models [33].

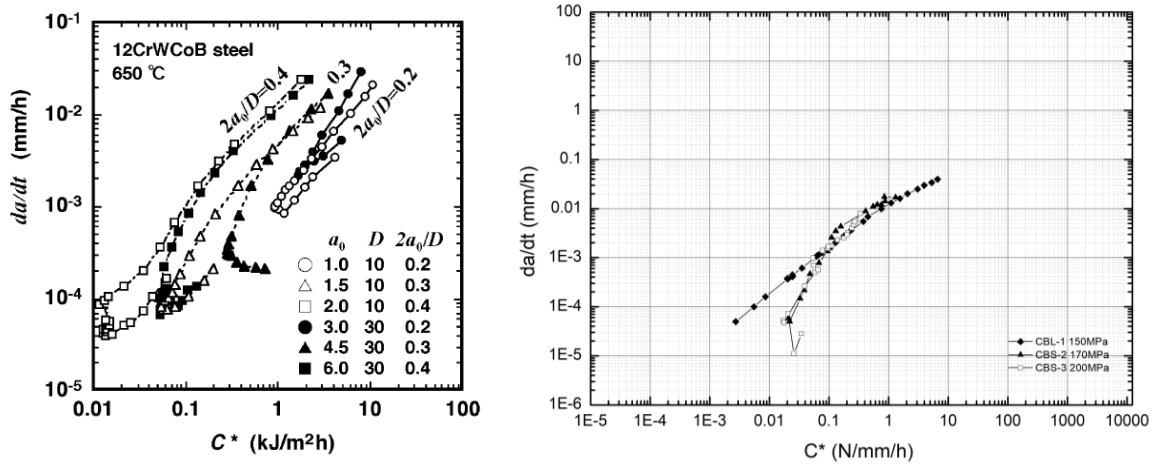


Figure 5.13 Creep crack growth rate vs. C^* plots of 12CrWCoB steel [130] and BG12Cr steel in ring-notched geometry at 650°C with loading conditions.

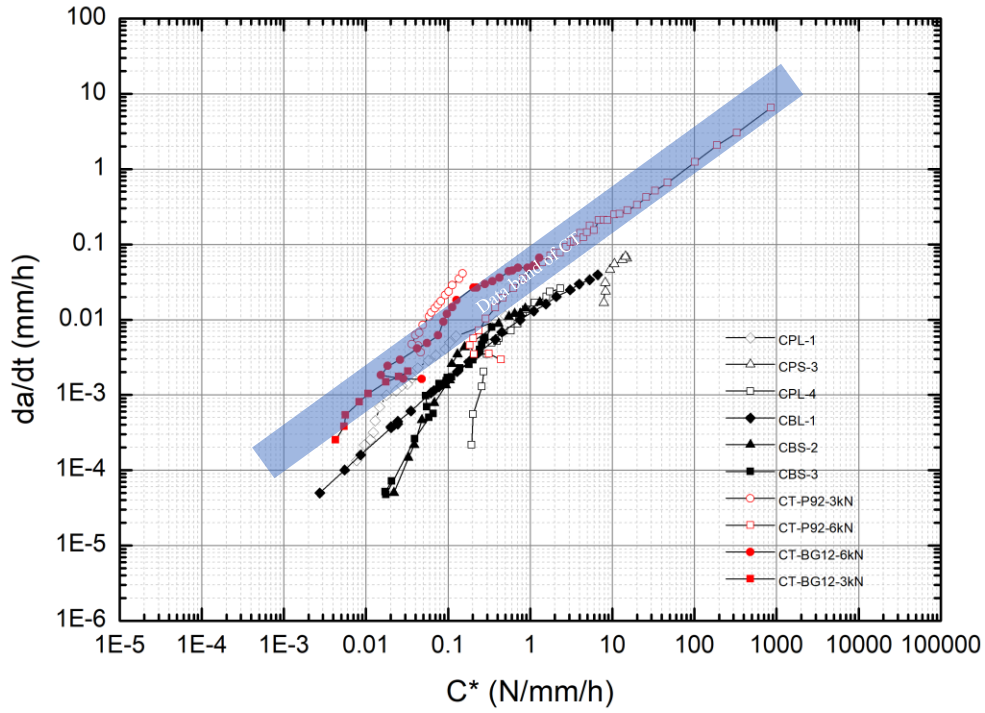


Figure 5.14 Creep crack resistance curves with C^* as driving force parameter for BG12Cr and P92 steel at 650°C in two different geometries: 1) CT, 2) ring notched geometry.

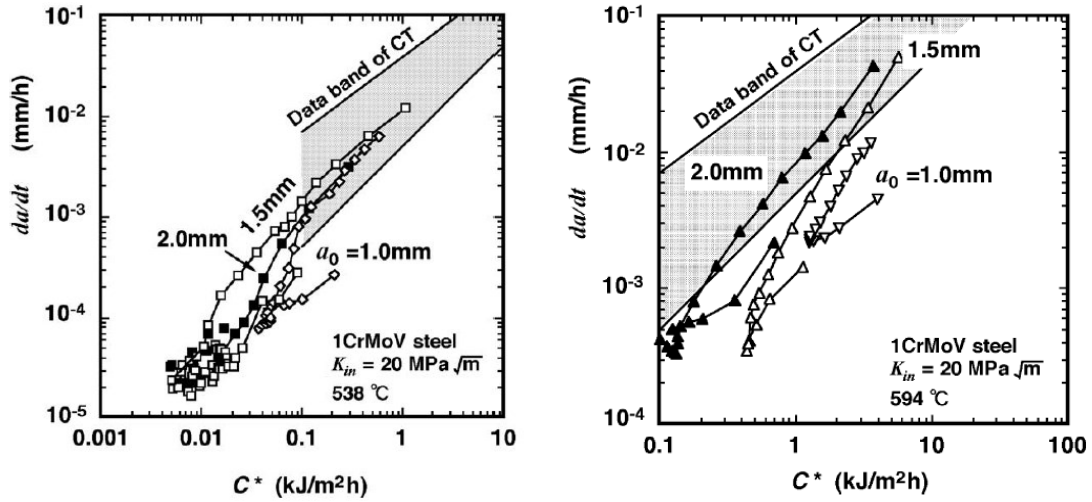


Figure 5.15 Creep crack growth rate vs. C^* parameter for ring-notched testpieces of 1CrMoV steel under different temperatures: 1) 538°C, 2) 594°C [130].

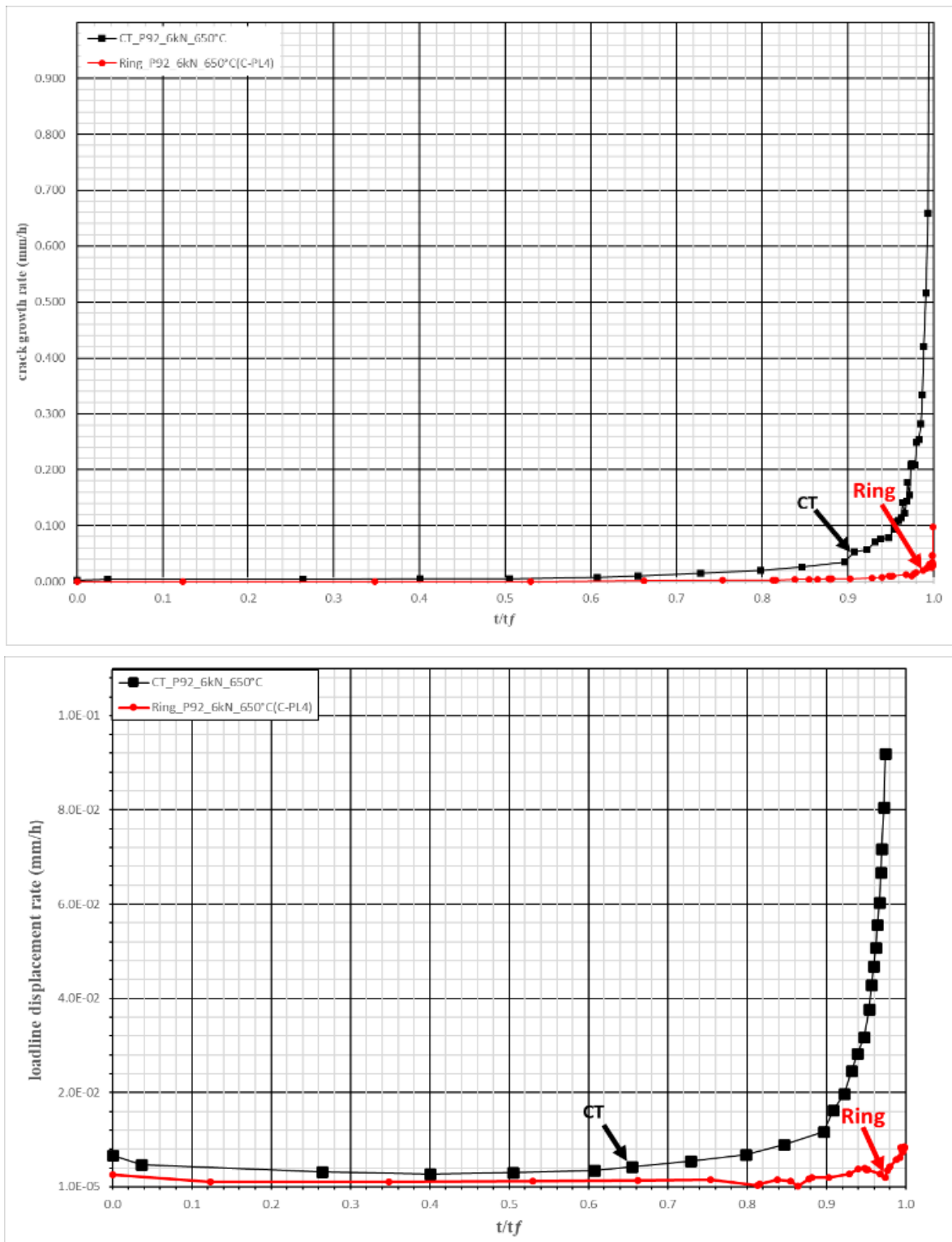


Figure 5.16 Relations between crack growth rate and displacement rate vs normalized time by CCGT time indicating the difference behaviour between two different geometries of P92 steel under the same testing conditions (CPL-4 versus CT-P92-6kN).

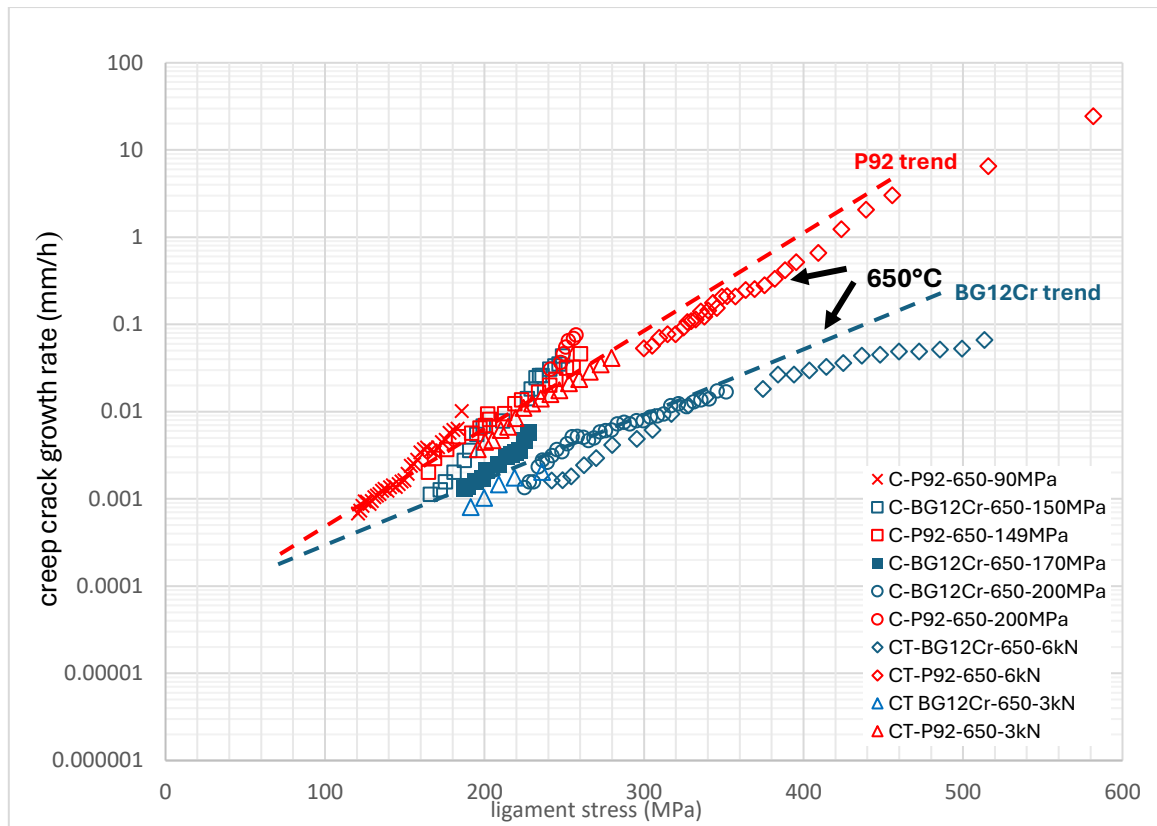


Figure 5.17 Creep crack growth rate (mm/h) versus ligament stress (MPa) plots of ring-notched testpieces at 650 °C.

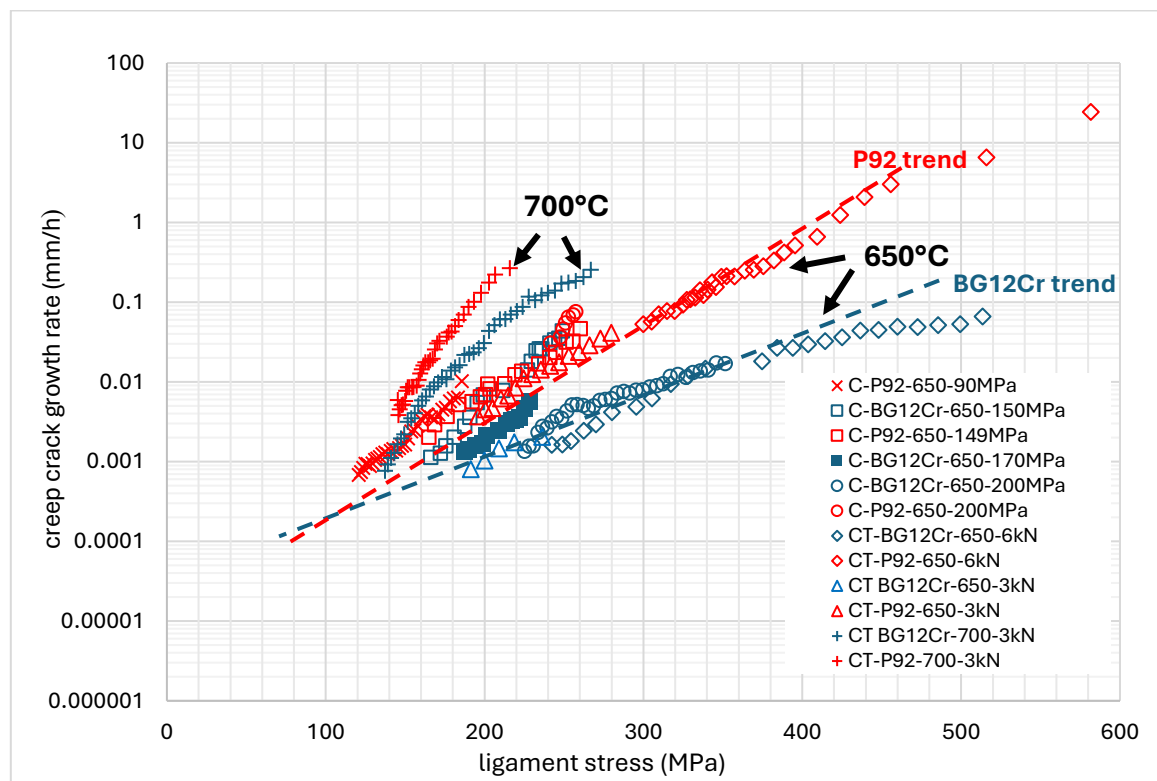


Figure 5.18 Creep crack growth rate (mm/h) versus ligament stress (MPa) plots of CT testpieces at different temperatures.

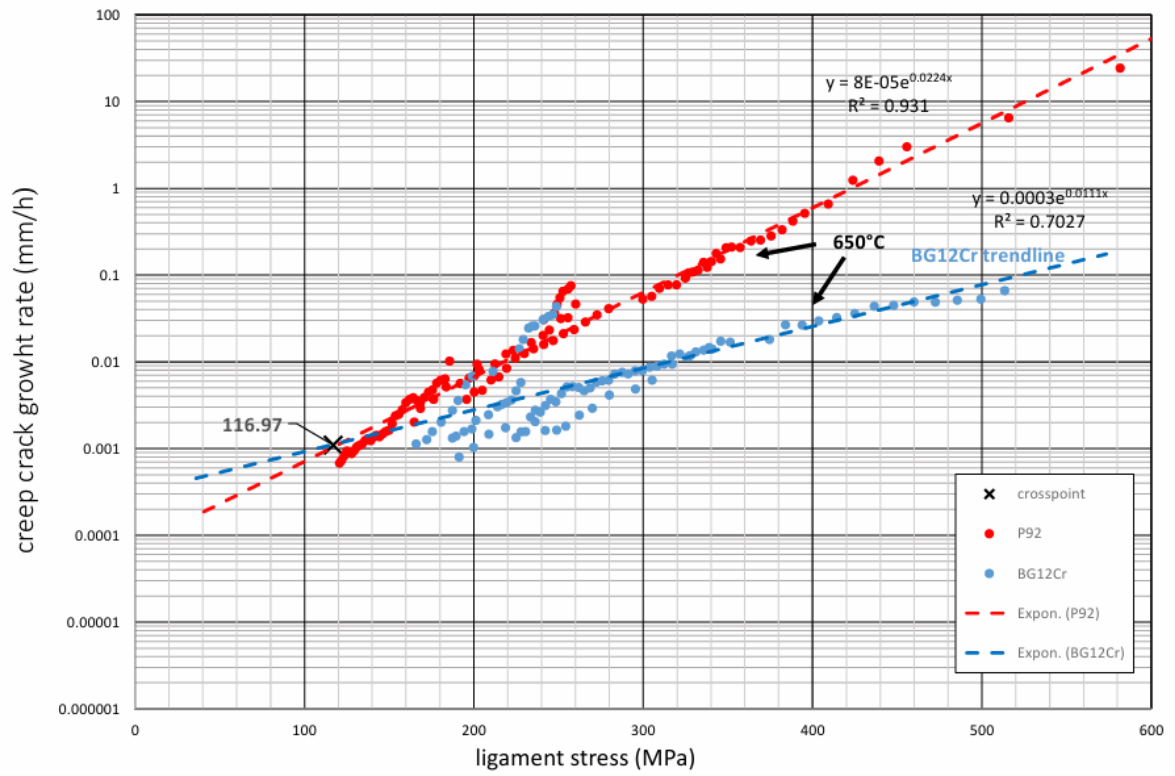


Figure 5.19 Creep crack growth rate (mm/h) versus ligament stress (MPa) plots of testpieces with different geometries at 650 °C (trendline version).