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Report on microstructure analysis of irradiate graphite at low dose

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Summary

In the European graphite irradiation programme, the irradiation behaviour of modern graphite grades is investigated at two irradiation temperatures, 750°C and 950°C. Following up on the irradiation experiments performed in the HTR-M and RAPHAEL projects, a low dose irradiation experiment at 750°C was performed to investigate materials properties changes induced by neutron irradiation. This report describes the microstructural analysis using optical microscopy and scanning electron microscopy on samples from the INNOGRAPH-1C irradiation experiment at 750°C.

Approval

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Optical microscopy and SEM

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Contents

Summary

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1 Introduction

Graphite is a suitable material to be used as a neutron moderator and reflector in nuclear reactors. Graphite has been used in Advanced Gas-Cooled Reactors, Magnox reactors, RBMK's (a Russian acronym meaning "reactor cooled by water and moderated by graphite"), Research Reactors, Materials Test Reactors and High Temperature Reactors (HTR's). Since the end of the 20th century there has been a renewed interest in HTR's. Two prototypes, one in Japan (HTTR) and one in China (HTR-10) are in operation. A demonstration plant is under construction in China (HTR-PM). The Next Generation Nuclear Plant NGNP in the US is in development. The European Commission is supporting research projects for the development of HTR technology to determine the technological requirements for designing and constructing an HTR in Europe.

Much research has been done on the behaviour of graphite grades in a nuclear reactor environment, because graphite is already being used in reactors for decades. However, these graphite grades are no longer commercially available since the raw materials no longer exist. In addition, most data from the past are from low temperature experiments (<550°C), whereas for HTR's the graphite temperatures will generally be higher than 550°C. In order to be able to design a European HTR, it was decided by the European commission to create a database that contains data of the materials behaviour of available graphite grades under neutron irradiation at temperatures relevant for the HTR's. This will allow the 'best' graphite(s) to be chosen, and provide the necessary data to allow the core design to be carried out.

The irradiation behaviour of HTR graphite grade candidates is investigated in the European programme on High Temperature Technology, started in 2001. Within the HTR-M1 and RAPHAEL-IP framework, graphite irradiation experiments were performed at 750°C and 950°C in the High Flux Reactor (HFR) in Petten. These were named INNOGRAPH-1B (750°C, high dose), INNOGRAPH-2A (950°C, medium dose) and INNOGRAPH-2B (950°C, high dose). In every experiment a few graphites were also irradiated at lower temperatures of 650°C (in the 1B) and 850°C (in the 2A and 2B). Figure 1-1 shows schematically the volume change of graphite with neutron fluence at the two main irradiation temperatures. The boxes on the curves indicate the five different irradiation experiments. Box 1A indicates the low dose irradiation at 750°C named INNOGRAPH-1A. This has been performed within HTR-M1 in the 5th Framework Programme and had a target dose of 8 dpa [1]. Box 1B indicates INNOGRAPH-1B, the high dose (up to ~25 dpa) experiment at the same temperature. Boxes 2A and 2B indicate the medium and high dose experiments at 950°C, INNOGRAPH-2A and INNOGRAPH-2B respectively. Results of these experiments are reported in [2]. Box 1C indicates the low dose irradiation experiment at 750°C that is performed in the framework of the ARCHER project. In this report, the results of optical microscopy and scanning electron microscopy on samples from the INNOGRAPH-1C irradiation are presented.

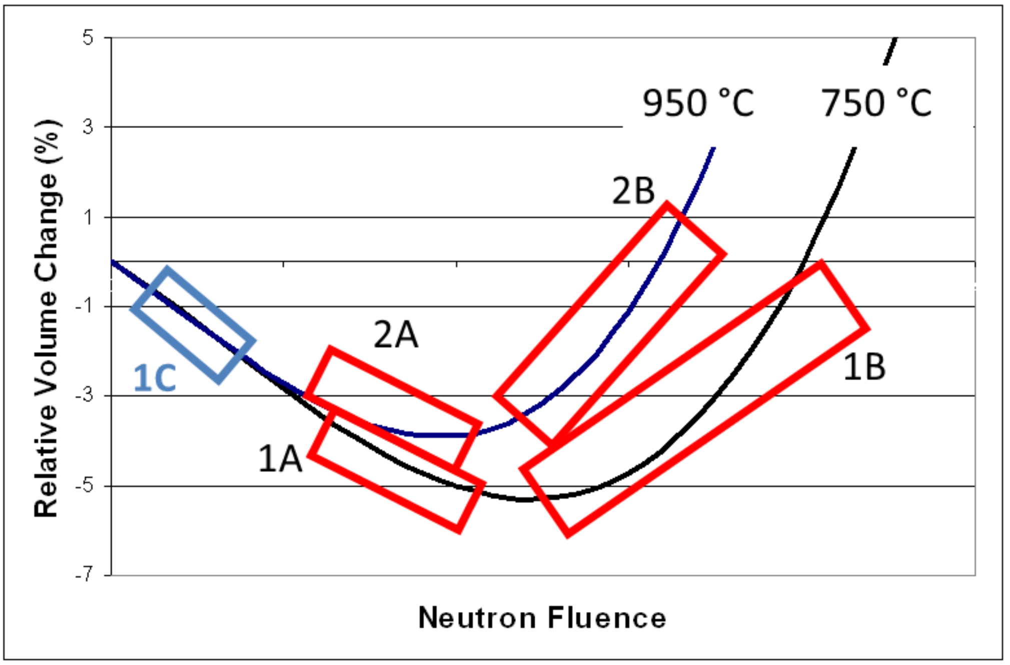

Figure 1-1 Schematic overview of the five INNOGRAPH irradiation experiments

2 Experiment and measurement techniques

2.1 Irradiation experiment

2.1.1 Graphite grades

The INNOGRAPH-1C is a follow-up experiment of the INNOGRAPH-1A and -1B experiments. Therefore, graphite grades are selected based on their presence in these preceding experiments [3]. The selection of the graphite grades for the INNOGRAPH irradiation experiments is based on several factors such as thermal and mechanical properties, impurity levels and availability [1]. The four major grades, produced by SGL Carbon and Graftech are chosen in such a way that the graphites cover a variety of microstructures. This is achieved by selecting grades based on different raw materials, i.e. coal tar pitch coke or petroleum coke, and different manufacturing methods, i.e. extrusion, isostatic-moulding or fibro-moulding.

The minor grades include graphites from three different manufacturers, SGL Carbon, Graftech and Toyo Tanso. The minor grades include iso-moulded graphites and graphites based on needle coke. The graphites used in the experiments are listed in Table 1.

Next to the major and minor grades that also were presented in the preceding irradiation experiments, a number of “specials” were present in the INNOGRAPH-1C. These include fuel matrix material supplied by Furnaces Nuclear Applications Grenoble (FNAG), graphite grades supplied by GrafTech and SGL, and Highly Oriented Pyrolitic Graphite (HOPG).

Table 1 Overview of the graphite grades selected for the INNOGRAPH-1C irradiation experiment

Grade	Manufacturer	Coke	Process	Major/minor
NBG-10	**SGL**	**Pitch**	**Extrusion**	**Major**
NBG-18	**SGL**	**Pitch**	**Vibro-moulding**	**Major**
PCEA	**GrafTech**	**Petroleum**	**Extrusion**	**Major**
PPEA	**GrafTech**	**Pitch**	**Extrusion**	**Major**
IG-110	Toyo Tanso	Petroleum	Iso-moulding	Minor
IG-430	Toyo Tanso	Pitch	Iso-moulding	Minor
LPEB/BAN	GrafTech	Needle	Extrusion	Minor
LPIB	GrafTech	Needle	Iso-moulding	Minor
NBG-17	SGL	Pitch	Vibro-moulding	Minor
NBG-20	SGL	Petroleum	Extrusion	Minor
NBG-25	SGL	Petroleum	Iso-moulding	Minor
PCIB	GrafTech	Petroleum	Iso-moulding	Minor

2.1.2 Specimen geometry

All specimens loaded in the INNOGRAPH experiments are cylindrical. The diameter of all specimens is 8 mm and the length is 6 mm. Specimens of these sizes are small enough to allow a sufficient number of specimens in the irradiation rig, and large enough to perform reasonably accurate measurements. The specimens are suitable for all type of measurements (dimensional changes, dynamic Young's modulus, coefficient of thermal expansion and thermal diffusivity).

The cylindrically shaped specimens are flattened by milling a plane with a width of 3 mm along the length. Figure 2-1 schematically shows the flattening. Producing a plane with a width of 3 mm mills only 1% of the volume of the cylinders. The benefits of this plane are threefold. The first benefit is the possibility to indicate the z-direction from the original graphite blocks in the specimen. The plane is milled perpendicular to the *z*-direction if the axial direction of the cylinder is parallel to the *xy*-direction, and therefore the *z*-direction in the specimen is known after fabrication. The plane is milled on an arbitrary position if the axial direction of the cylinders is parallel to the *z*-direction, because in that case the *z*-direction is already indicated by the axial direction.

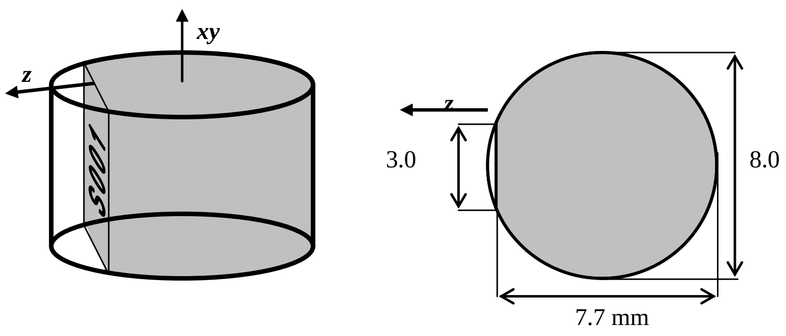

Figure 2-1 Flattening of the specimens. The figure on the left-hand side exaggeratedly shows the flatting of the cylinder. The figure on the right-hand side is a top-view of the specimen

The second benefit of the plane is that it gives an area to engrave the specimen with a unique code. This code is necessary to easily identify a specimen. The code consists of four characters. The code is formed by three numbers and one letter. The letter indicates the graphite manufacturer (S= SGL, U= UCAR (Graftech) and T= Toyo Tanso). The letter Z is used for a number of special samples by various manufacturers.

A third benefit is that the milled plane can act as a marking that defines the measuring positions during the dimensional measurements before and after irradiation.

2.1.3 INNOGRAPH-1C low dose irradiation experiment at 750°C

The INNOGRAPH-1C rig consists of a stack of eight TZM (0.5Ti, 0.08Zr, 0.42 Mo) drums that are used as specimen-holders (Figure 2-2). The rig contained seven drums with a height of 60 mm and one drum with a height of 30 mm. In each drum, three channels are present to contain the samples. In between drums, a thin graphite foil is placed to prevent specimens moving axially into the drum below. The high density of TZM makes it possible to reach the required nominal temperature of 750°C. Because of the relatively low neutron flux in the top drum, this drum cannot reach the 750°C. However, this drum is still usable to irradiate graphite specimens at a slightly lower temperature. The top drum is separated from the drums below by a thermal barrier and is designed to have a temperature of 650°C. For the rest of the rig the design temperature is 750°C. The capsule is instrumented with 24 thermocouples that are placed on different radial and axial positions to monitor the temperature during irradiation. Temperature was controlled by changing the He/Ne gas mixture in the second containment and the vertical displacement of the experiment in the reactor core. The temperature was controlled for the 750°C drums; the temperature for the top drum at 650°C is not controlled.

In the centre of each drum a hole was drilled to place neutron fluence detector sets. Five fluence detector sets were distributed axially over the rig with TZM fillers in between. Four fluence detector sets are distributed radially in two drums to monitor the radial gradients. The detector sets are analysed after the experiment to determine the neutron fluence.

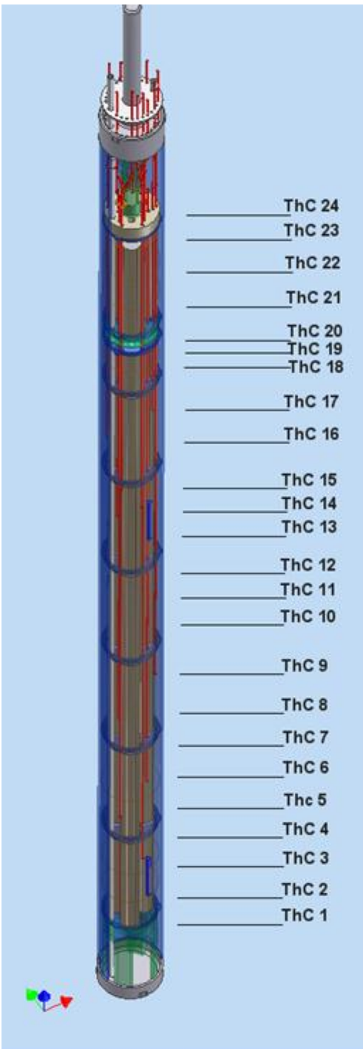

Figure 2-2 Drawing of the INNOGRAPH-1C irradiation rig

2.2 Microstructural analysis

2.2.1 Test matrix

The microstructural analysis consists of optical microscopy (OM) and scanning electron microscopy (SEM). For both techniques, the same specimens are used. In total, seven irradiated graphite specimens are selected. This constitutes one sample per graphite grade that also has been examined as part of the medium and high dose experiments at 750°C [4], except for minor grade IG-110. For the microstructural analysis, the samples are prepared by grinding and polishing. Thus, optical microscopy and SEM are destructive investigative techniques. Therefore, the samples selected have their standard PIE measurements completed [5]. Table 2 shows the selected samples.

Table 2 INNOGRAPH-1C graphite specimens selected for optical microscopy and SEM

Specimen code	Graphite grade	Orientation	Location	dpa
0S27	NBG-10	AG	heart	2.2
1S02	NBG-17	WG	heart	2.1
2S01	NBG-18	WG	heart	2.2
3S37	NBG-25	WG	heart	2.2
0U32	PCEA	AG	heart	2.2
1U07	PCIB	AG	heart	2.2
2U03	PPEA	WG	heart	1.7

2.2.2 Sample preparation

Samples are placed in a sample holder that can be used in both the optical microscope and the SEM. The samples are fixed in the sample holder using an two-component resin with fluorescent dye. The samples are mounted such that they protrude 0.5 mm from the holder. After curing of the resin, the sample is ground down to the level of the sample holder using silicon carbide paper. Next, the sample surface is polished using increasingly fine oil based diamond paste down to 1 µm grain size. All preparation is performed in glovebox.

The sample orientation in the test matrix (Table 2) indicates the direction of the sample with respect to the grain direction in the graphite source material. The length of the cylindrical sample is parallel to the indicated direction. Thus, the optical and scanning electron micrographs will show a plane orthogonal to the direction indicated in Table 2.

2.2.3 Optical microscopy

Optical microscopy is performed on an Olympus BX51 optical microscope. The microscope is placed in a glovebox to allow for radioactive samples to be investigated. Sample positioning is performed using a computer-controlled xy-stage. Bright field, polarised light, and fluorescence microscopy can be performed on the BX51. For bright field and polarised light microscopy, a 120W metal halide lamp is present. For fluorescence microscopy, a100W Halogen X-cite 120Q lamp with appropriate fluorescence filters is available. Objectives available on the Olympus BX51 are 5x, 10x, 20x, 50x, and 100x magnification long working distance objectives. Micrographs are obtained using a 5 megapixel, 14 bit CCD camera and “Stream motion” software by Olympus. The full image resolution is 2576 by 1932 pixels. For the microstructure analysis, first bright field and polarised light micrographs of the whole sample cross-section are made. These micrographs are composed of approximately 35 micrographs at 5x magnification. Next, micrographs are made on 5x up to 50x magnification on 3 different locations on the sample. Next to bright field also polarised light micrographs are acquired for magnifications of 5x to 50x.

2.2.4 Scanning electron microscopy

At NRG, a JEOL JSM 6490 Scanning Electron Microscope (SEM), equipped with several detectors (EDS, WDS and EBSD), is available. The SEM is placed in alpha tight hot-cell to allow analysing radioactive samples emitting α, γ and β radiation, therefore both nuclear structural materials and nuclear fuels can be analysed. Irradiation sensitive parts of the SEM are removed or protected and changes have been made to the SEM to allow remote handling and to allow maintenance of the important parts.

The SEM has a Tungsten filament and an acceleration voltage between 0.3- 30 keV. It can be operated in both low and high vacuum mode. Low vacuum mode is useful for poor conducting samples. A resolution of 3.0 nm can be reached in the high vacuum mode at 30 keV. As graphite is conductive, high vacuum mode can be used. The resin used for sample fixation can be charged somewhat by the electron beam. To reduce charging, a conductive gold-palladium layer is sputtered onto the samples. The thickness of this Au-Pd layer is in the order of a few nanometers.

3 Results

3.1 Optical microscopy

All seven micrographs were succesfully prepared for microscopy. In paragraph 3.1.1 to 3.1.7 the results of the optical microscopy are presented with one sample per paragraph.

3.1.1 NBG-10 micrographs

Figure 3-1to Figure 3-4 show the micrographs of irradiated NBG-10 sample 0S27 at 2.2 dpa.

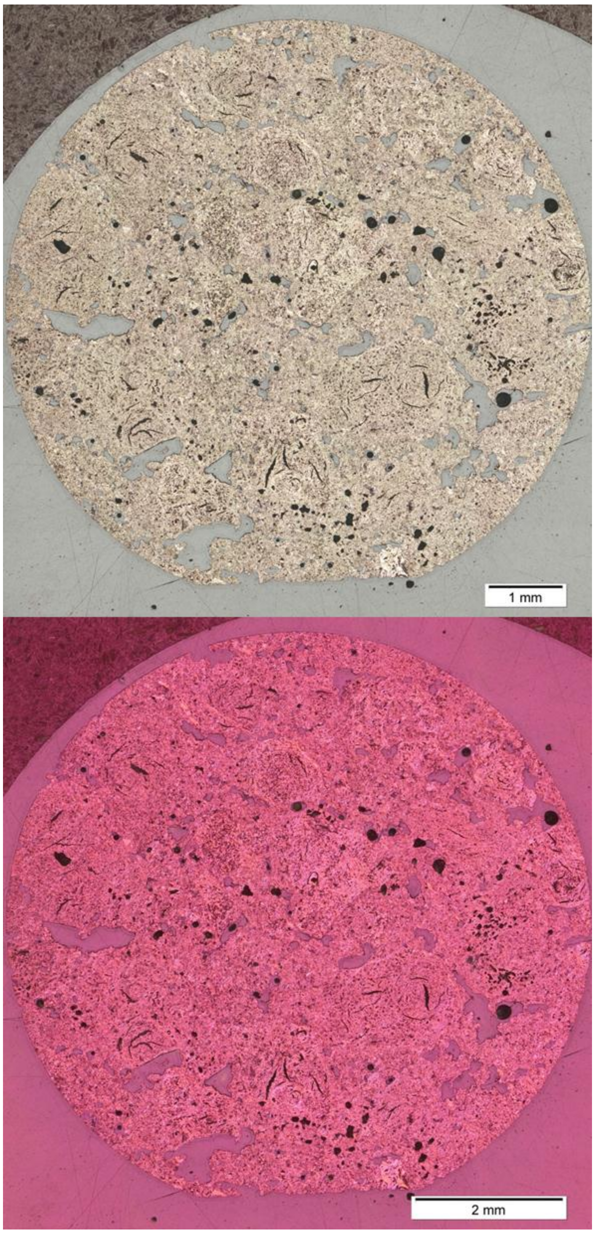

Figure 3-1 Bright field (top panel) and polarisation (bottom panel) overview micrographs of NBG-10 specimen 0S27 irradiated to 2.2.

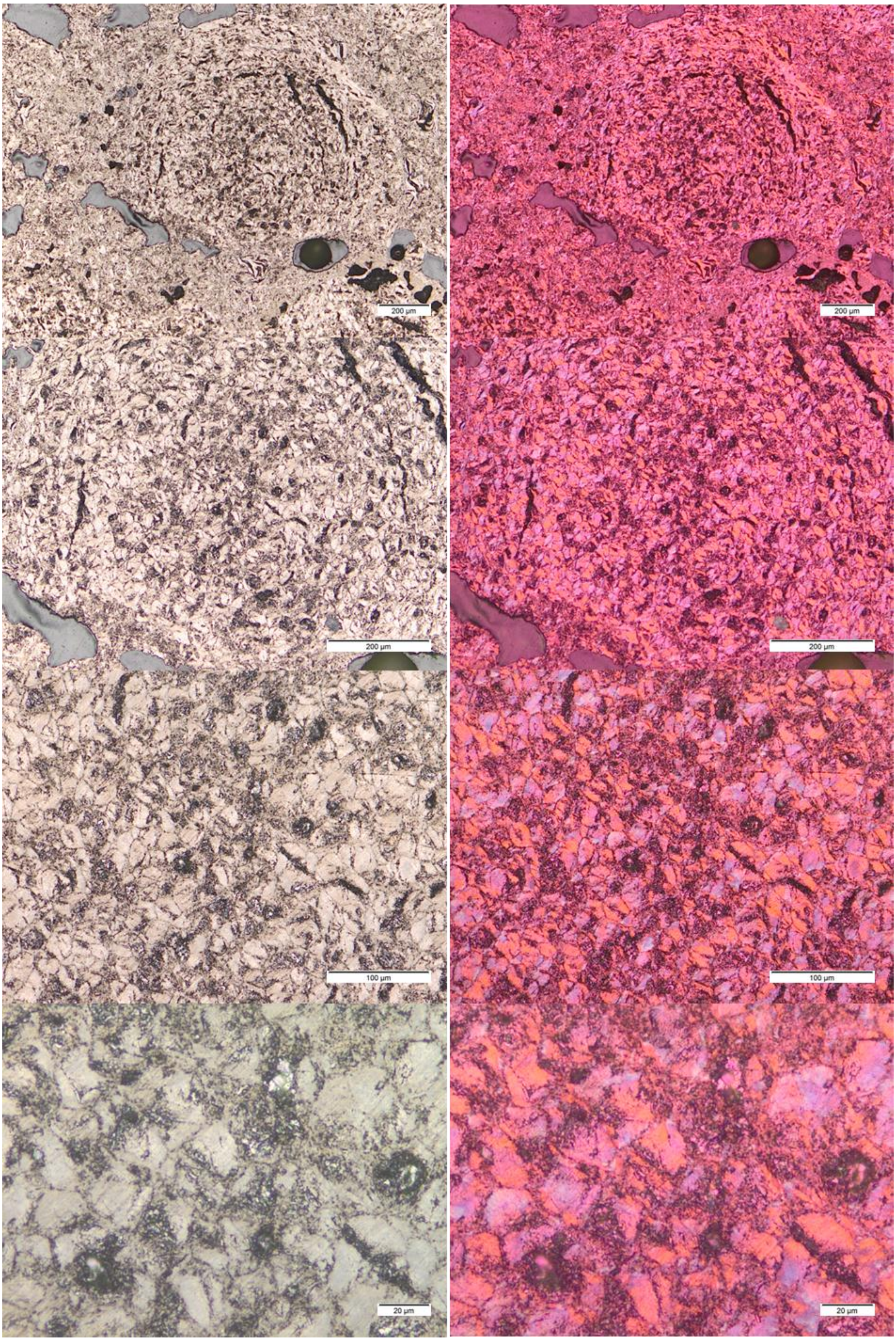

Figure 3-2 Bright field (left) and polarisation (right) micrographs of NBG-10 specimen 0S27

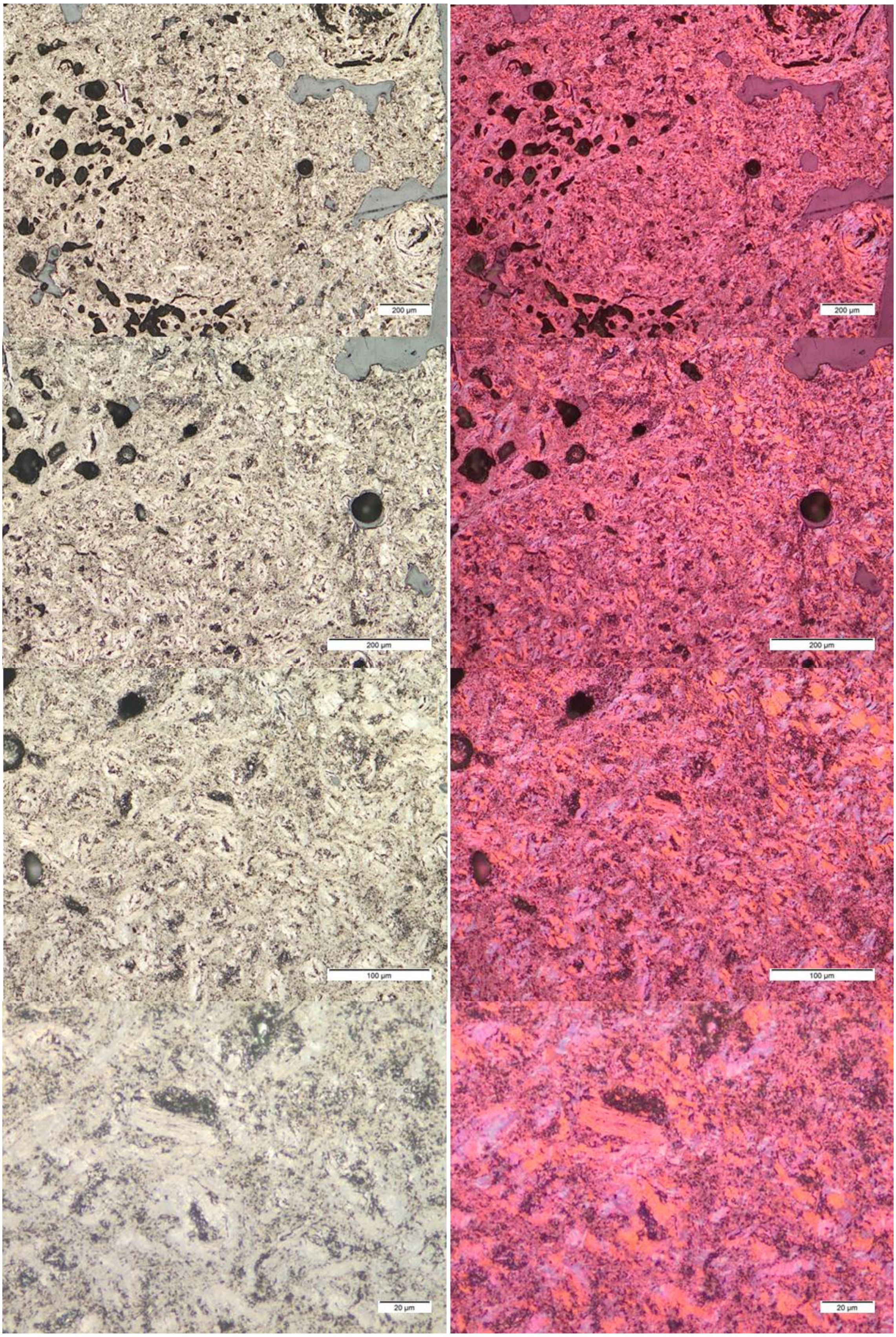

Figure 3-3 Bright field (left) and polarisation (right) micrographs of NBG-10 specimen 0S27

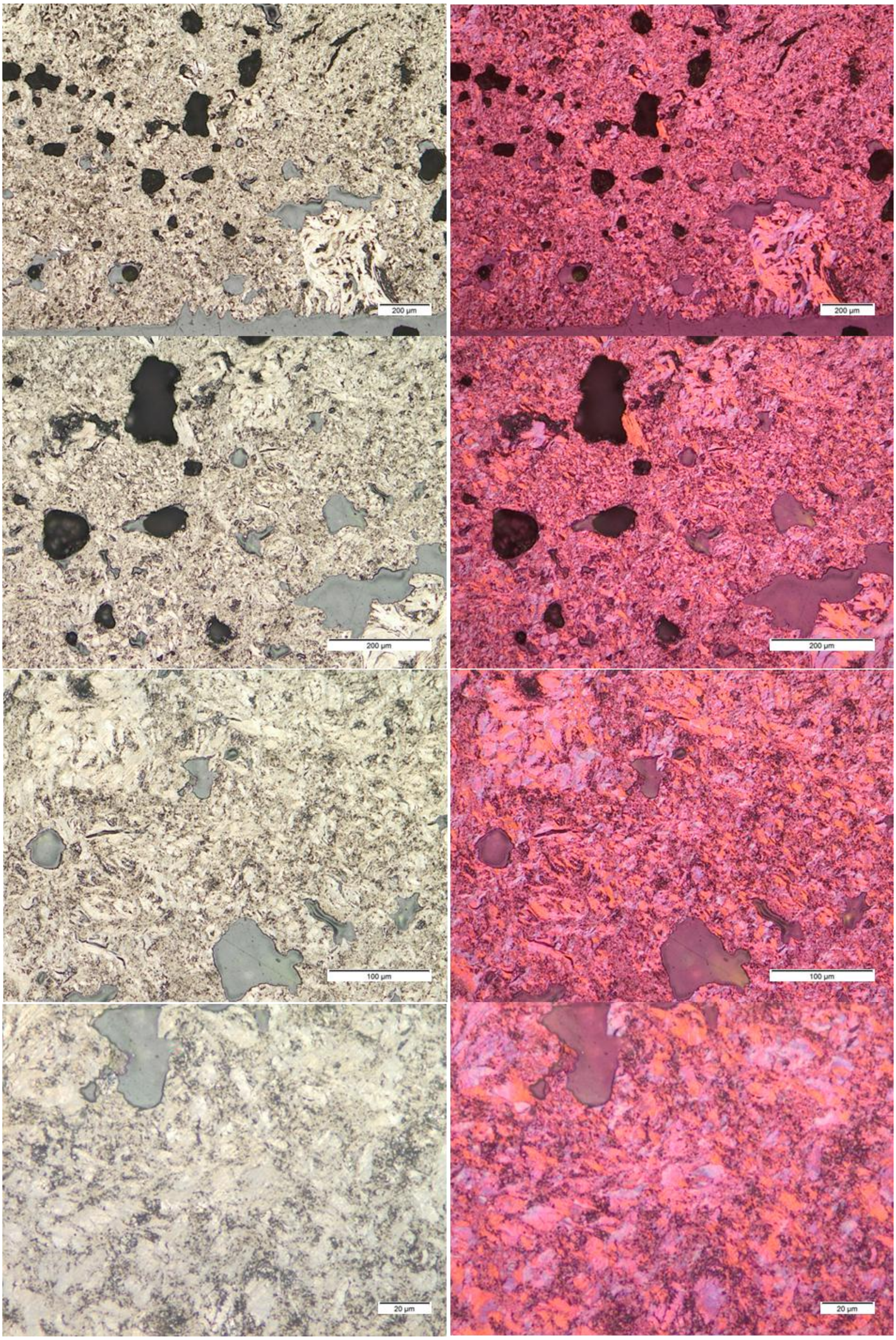

Figure 3-4 Bright field (left) and polarisation (right) micrographs of NBG-10 specimen 0S27

3.1.2 NBG-17 micrographs

Figure 3-5 to Figure 3-8 show the micrographs of irradiated NBG-17 sample 1S02 at 2.1 dpa.

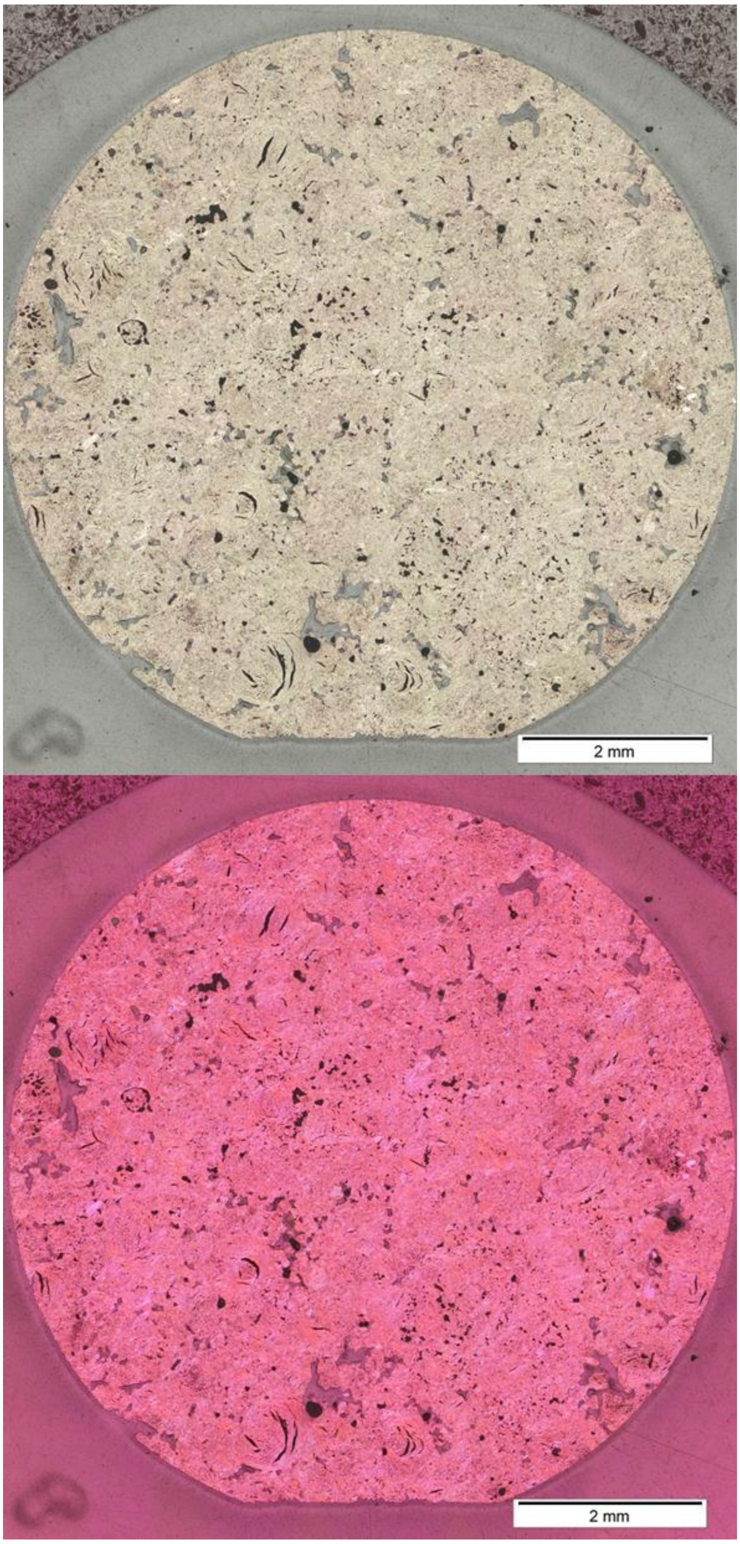

Figure 3-5 Bright field (top panel) and polarisation (bottom panel) overview micrographs of NBG-17 specimen 1S02 irradiated to 2.1.

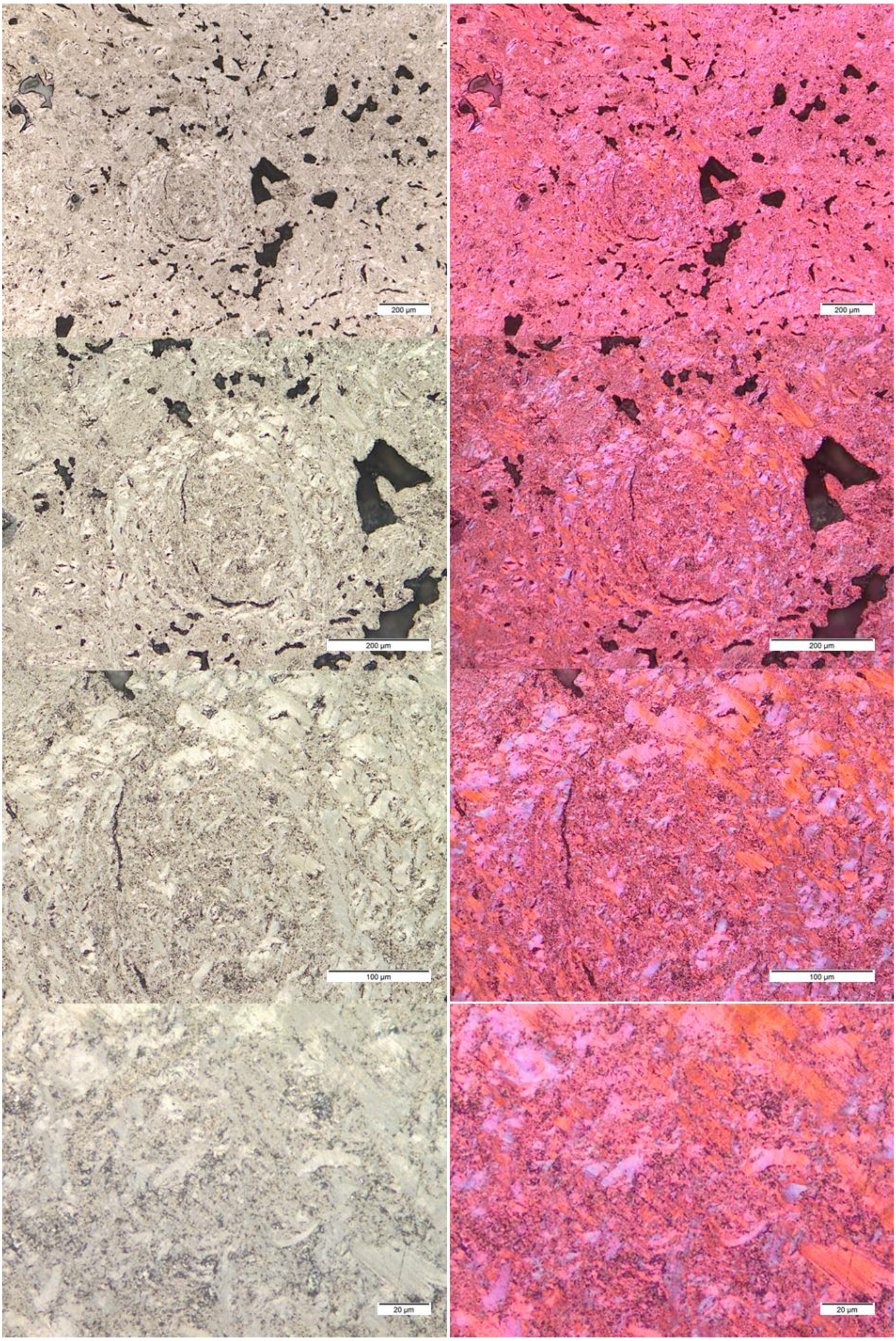

Figure 3-6 Bright field (left) and polarisation (right) micrographs of NBG-17 specimen 1S02

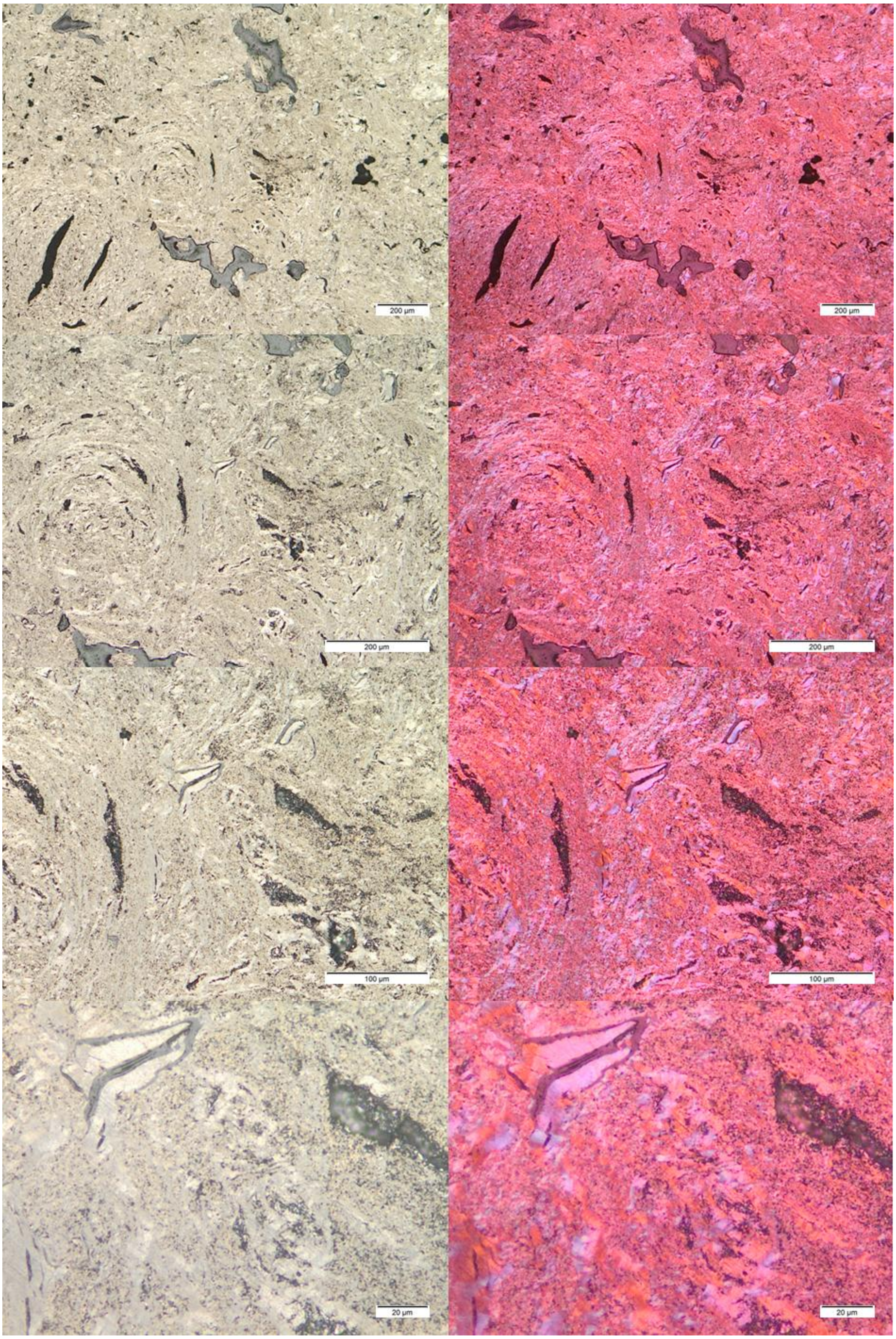

Figure 3-7 Bright field (left) and polarisation (right) micrographs of NBG-17 specimen 1S02

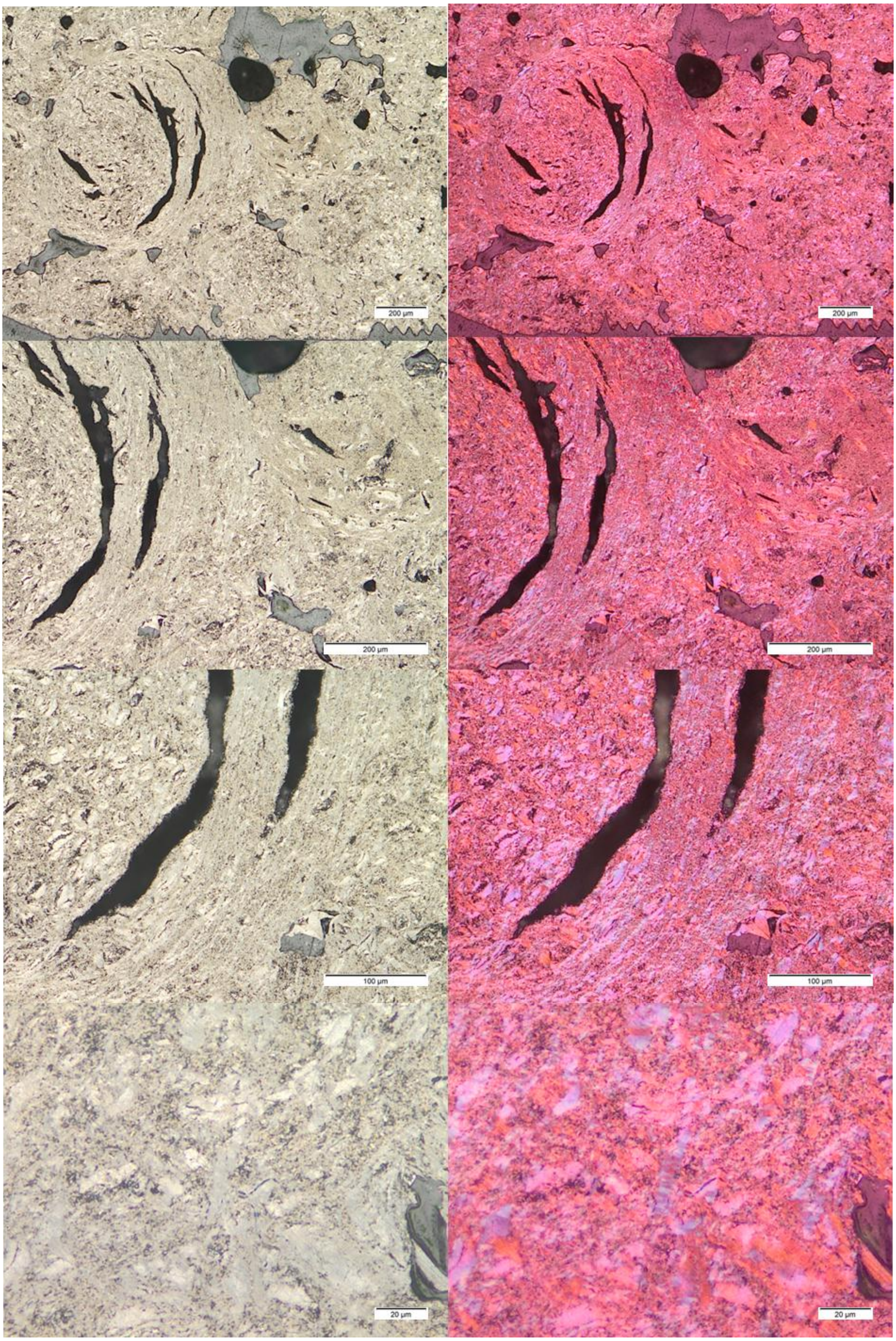

Figure 3-8 Bright field (left) and polarisation (right) micrographs of NBG-17 specimen 1S02

3.1.3 NBG-18 micrographs

Figure 3-9 to Figure 3-12 show the micrographs of irradiated NBG-18 sample 2S01 at 2.2 dpa.

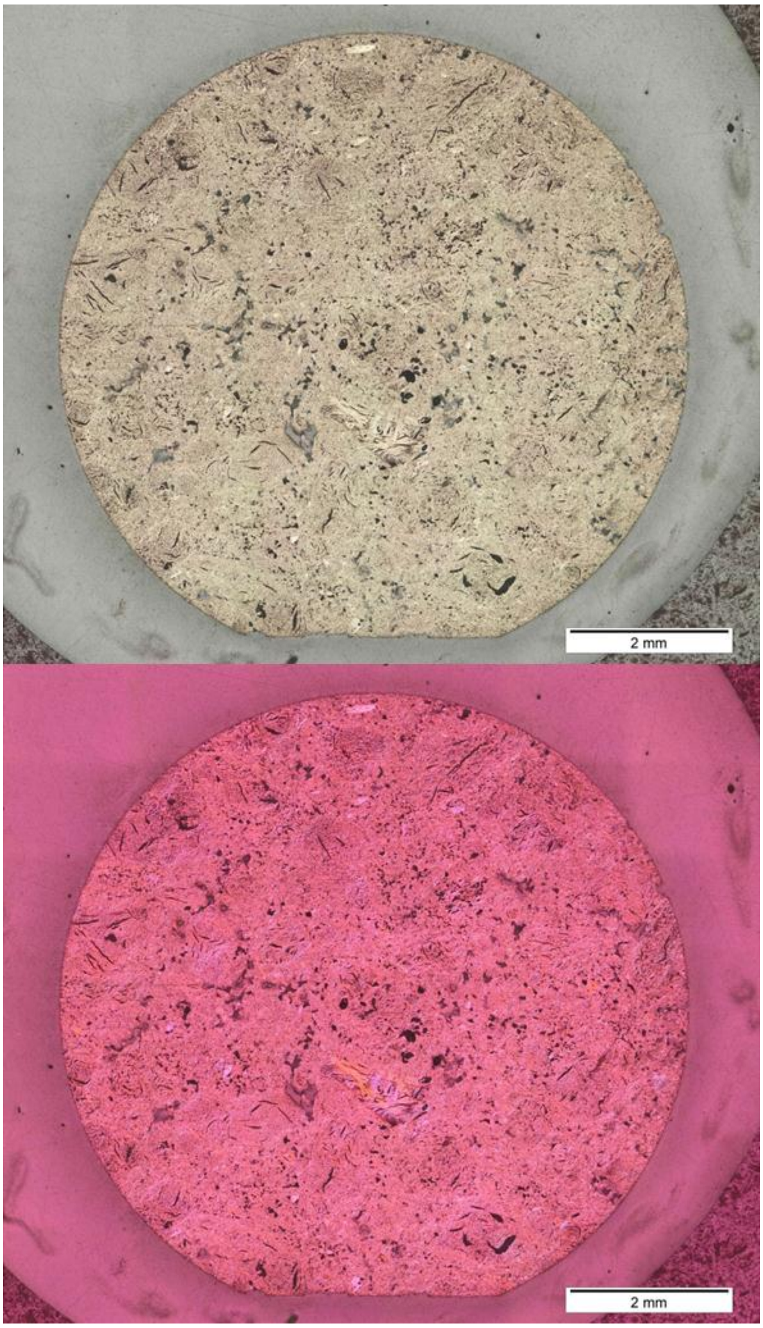

Figure 3-9 Bright field (top panel) and polarisation (bottom panel) overview micrographs of NBG-18 specimen 2S01 irradiated to 2.2

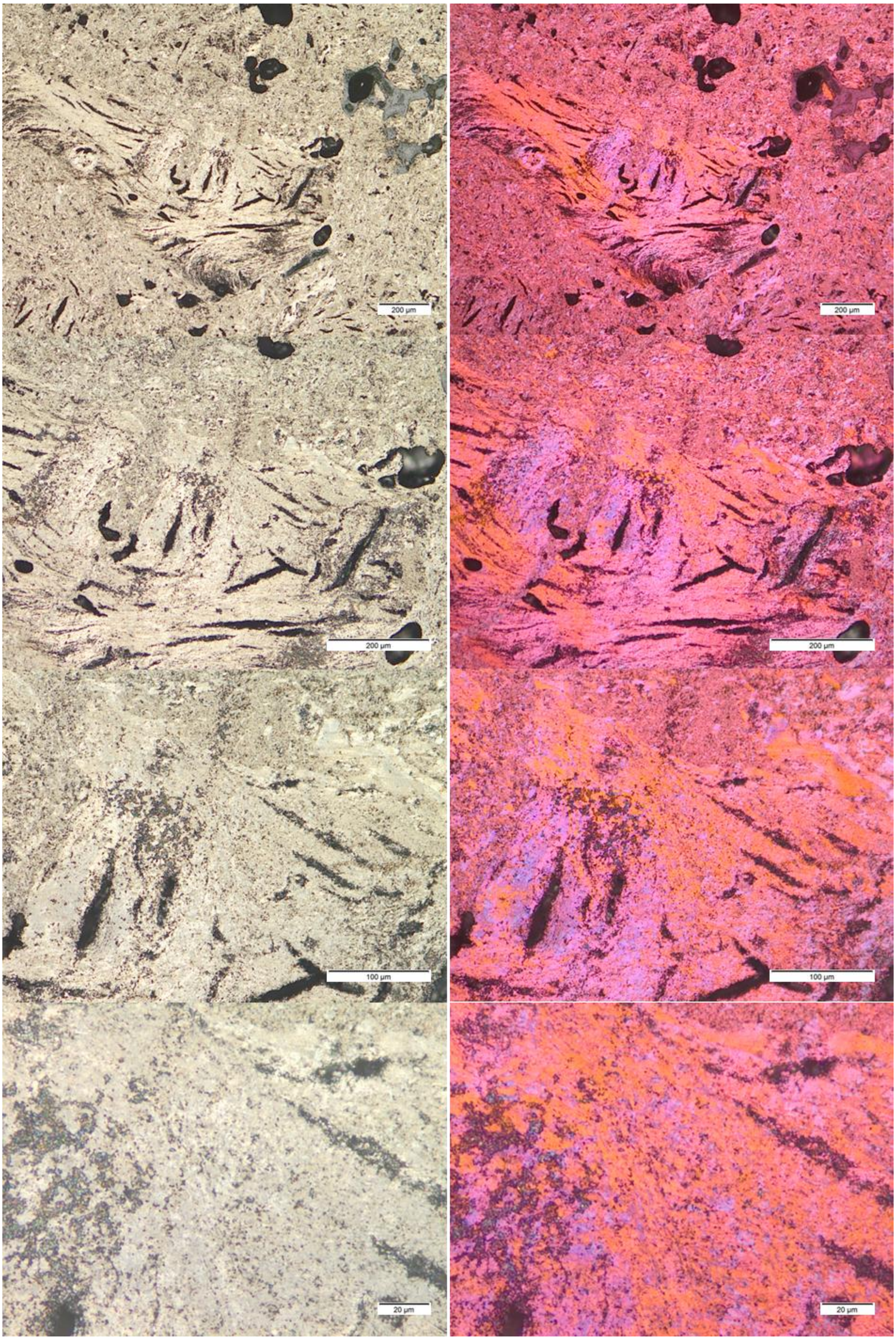

Figure 3-10 Bright field (left) and polarisation (right) micrographs of NBG-18 specimen 2S01

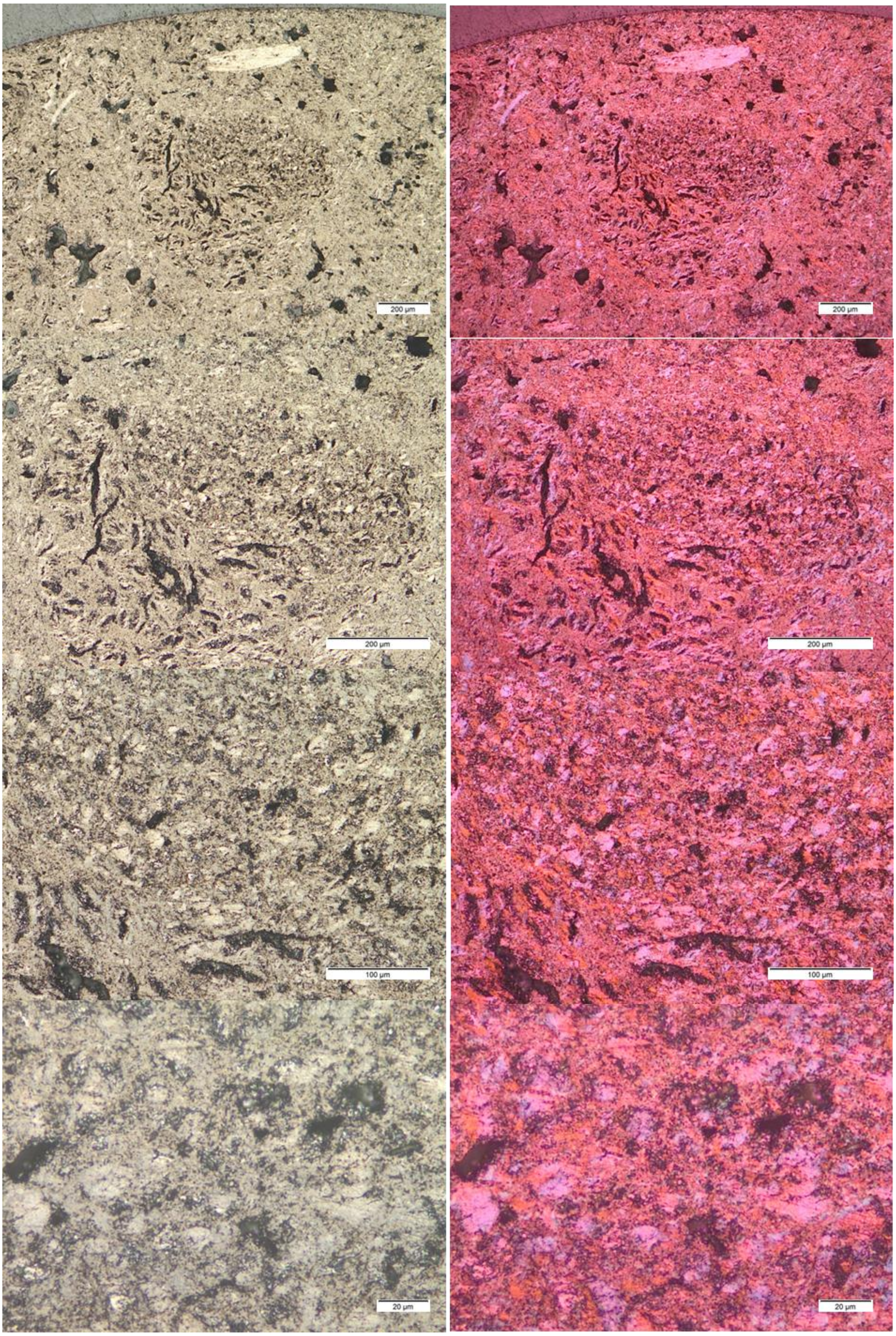

Figure 3-11 Bright field (left) and polarisation (right) micrographs of NBG-18 specimen 2S01

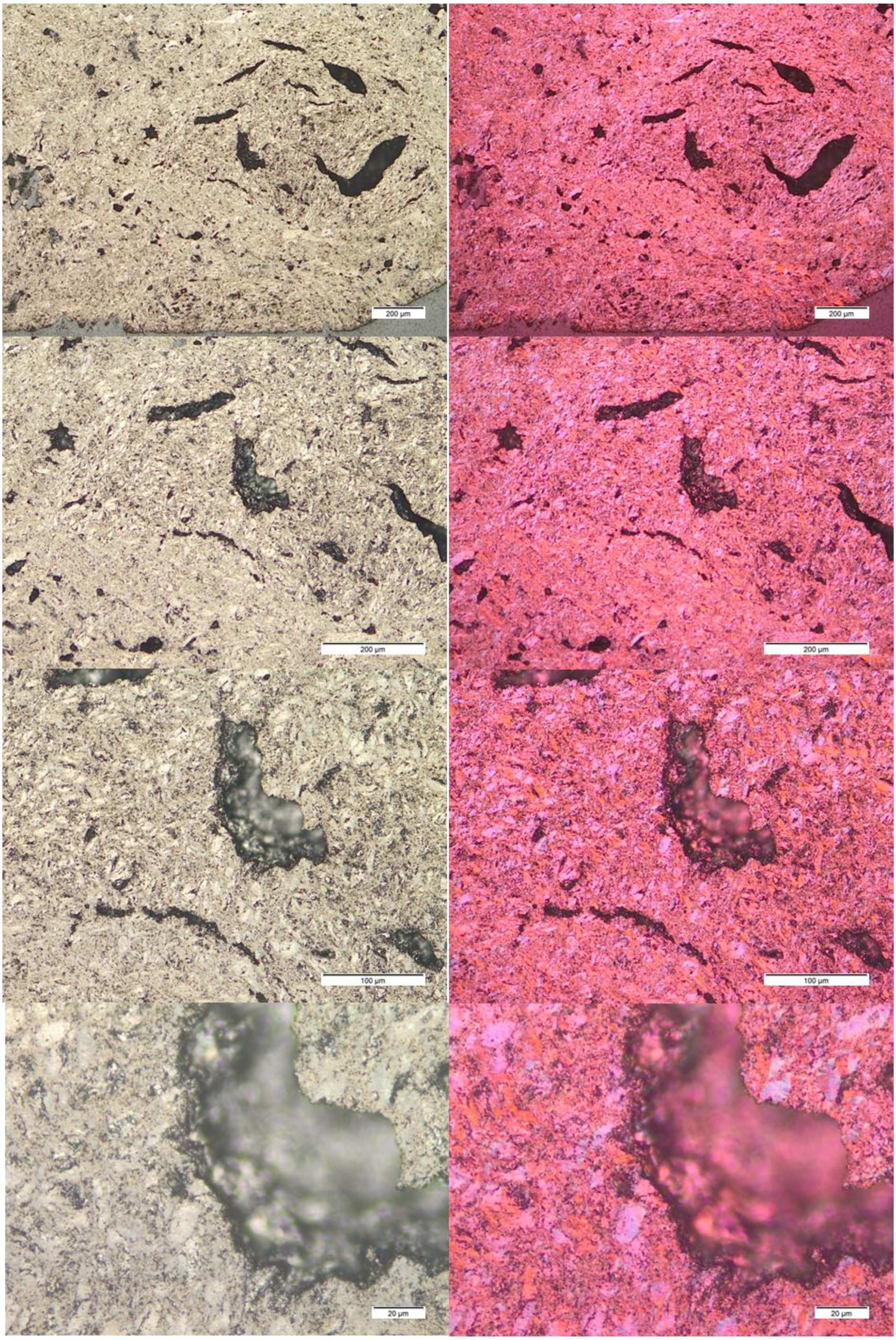

Figure 3-12 Bright field (left) and polarisation (right) micrographs of NBG-18 specimen 2S01

3.1.4 NBG-25 micrographs

Figure 3-13 to Figure 3-15 show the micrographs of irradiated NBG-25 sample 3S37 at 2.2 dpa.

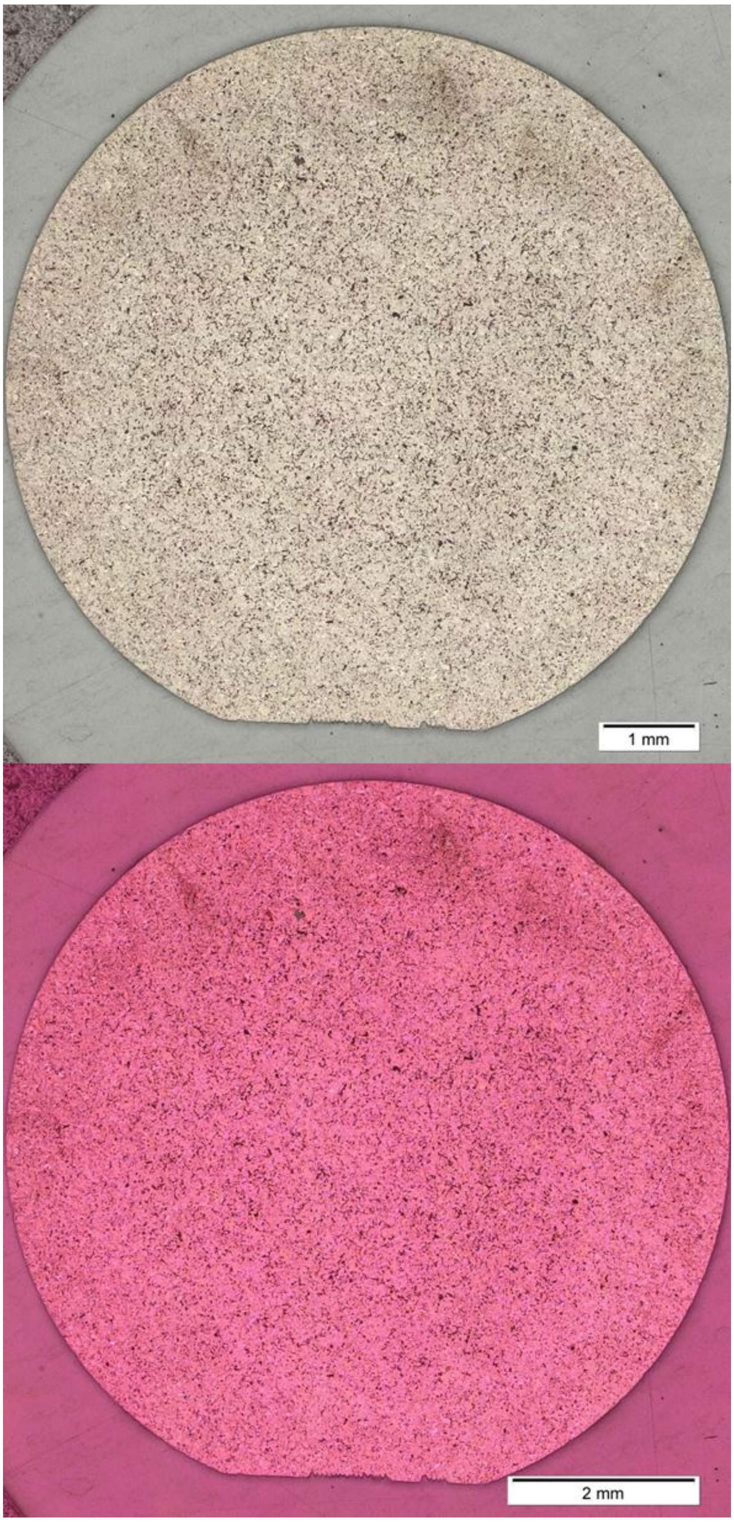

Figure 3-13 Bright field (top panel) and polarisation (bottom panel) overview micrographs of NBG-25 specimen 3S37 irradiated to 2.2

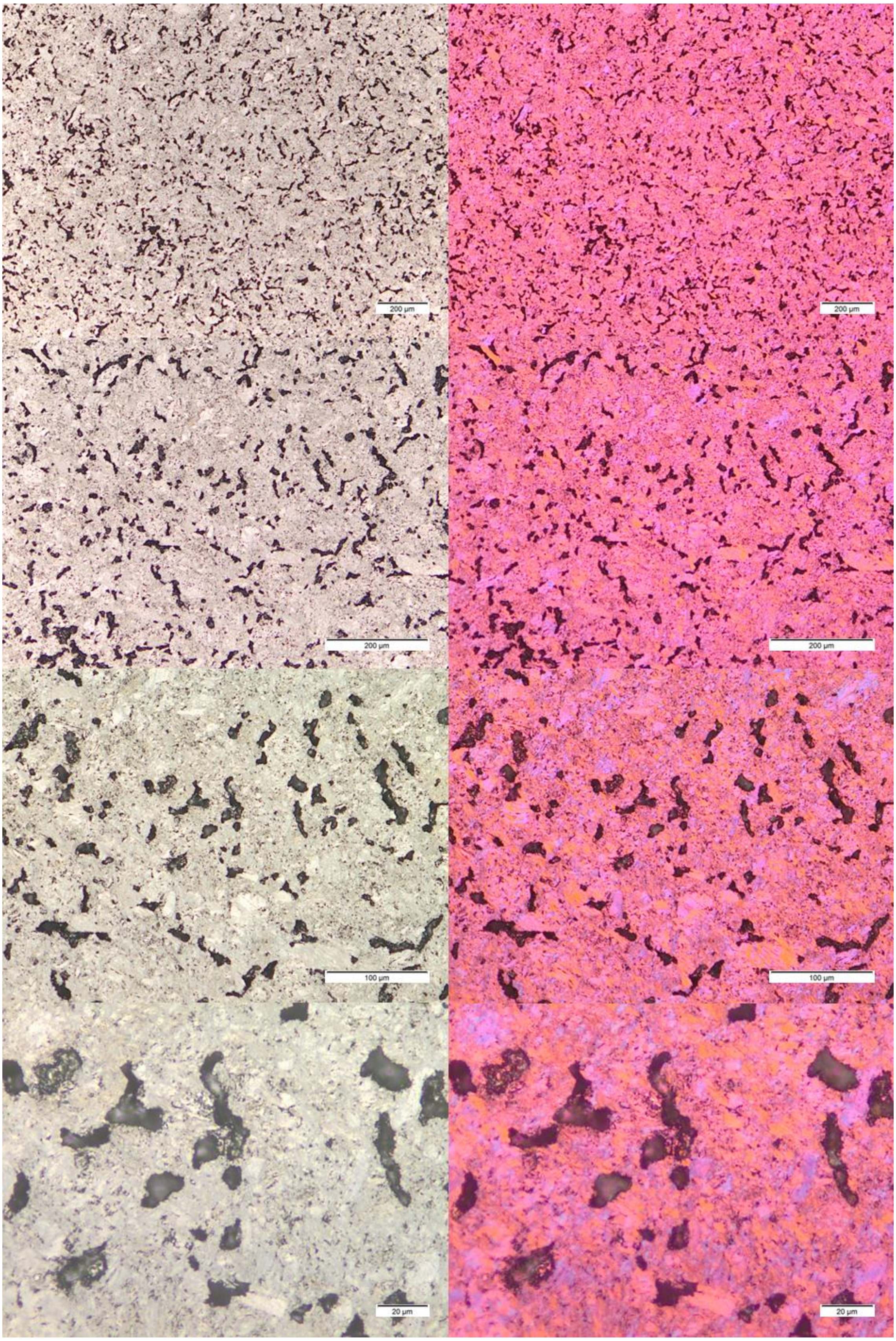

Figure 3-14 Bright field (left) and polarisation (right) micrographs of NBG-25 specimen 3S37

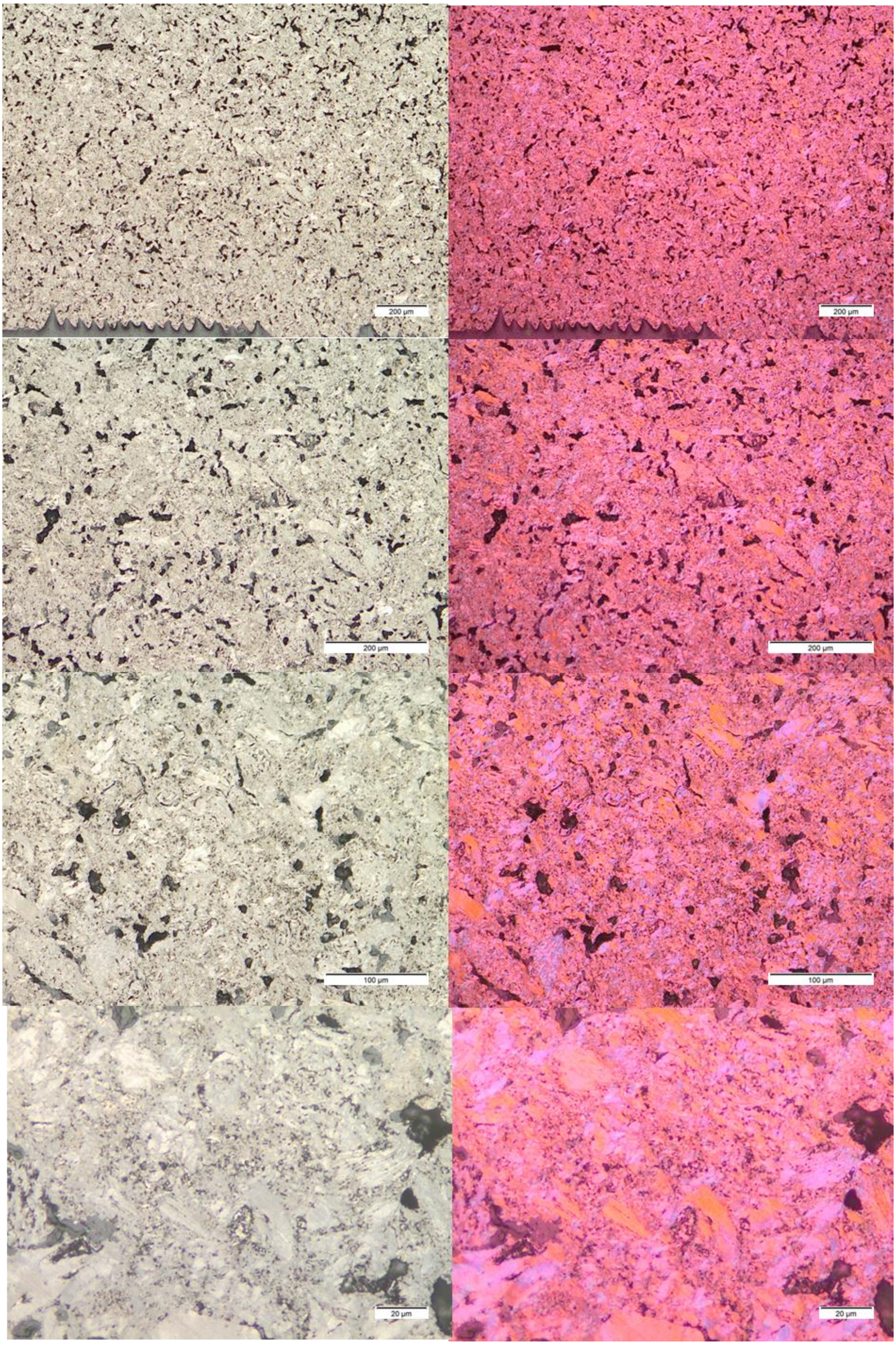

Figure 3-15 Bright field (left) and polarisation (right) micrographs of NBG-25 specimen 3S37

3.1.5 PCEA micrographs

Figure 3-16 to Figure 3-19 show the micrographs of irradiated PCEA sample 0U32 at 2.2 dpa.

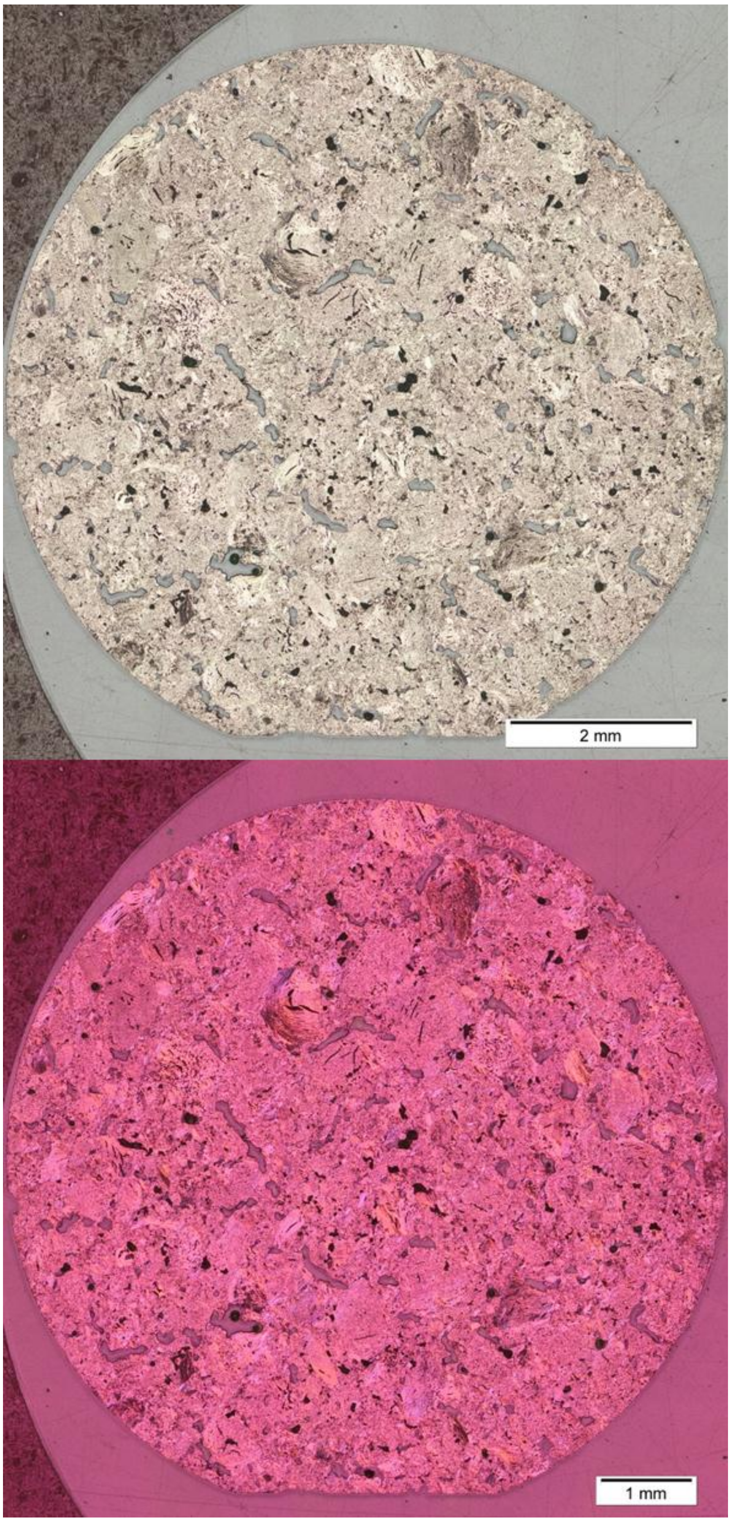

Figure 3-16 Bright field (top panel) and polarisation (bottom panel) overview micrographs of PCEA specimen 0U32 irradiated to 2.2 dpa

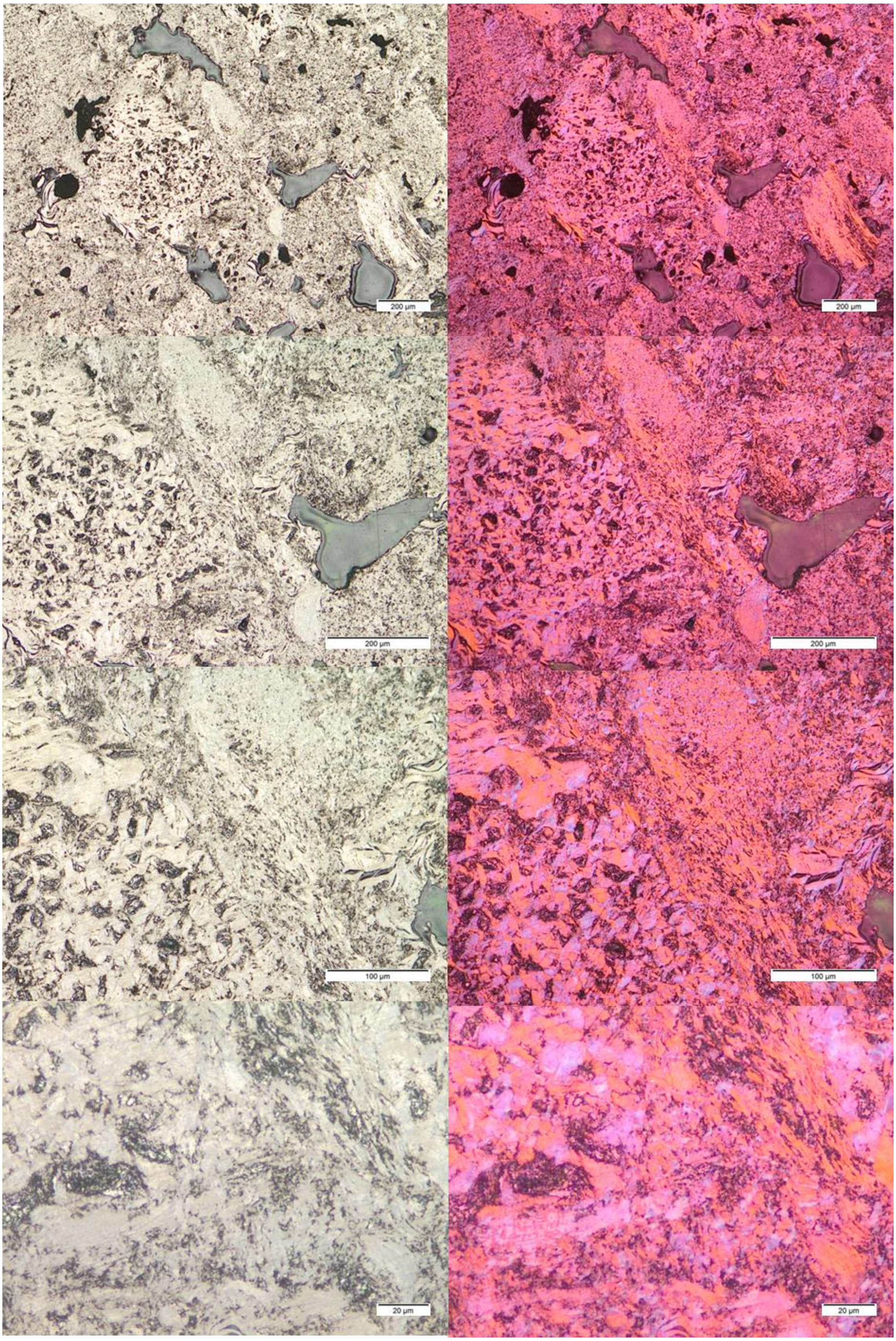

Figure 3-17 Bright field (left) and polarisation (right) micrographs of PCEA specimen 0U32

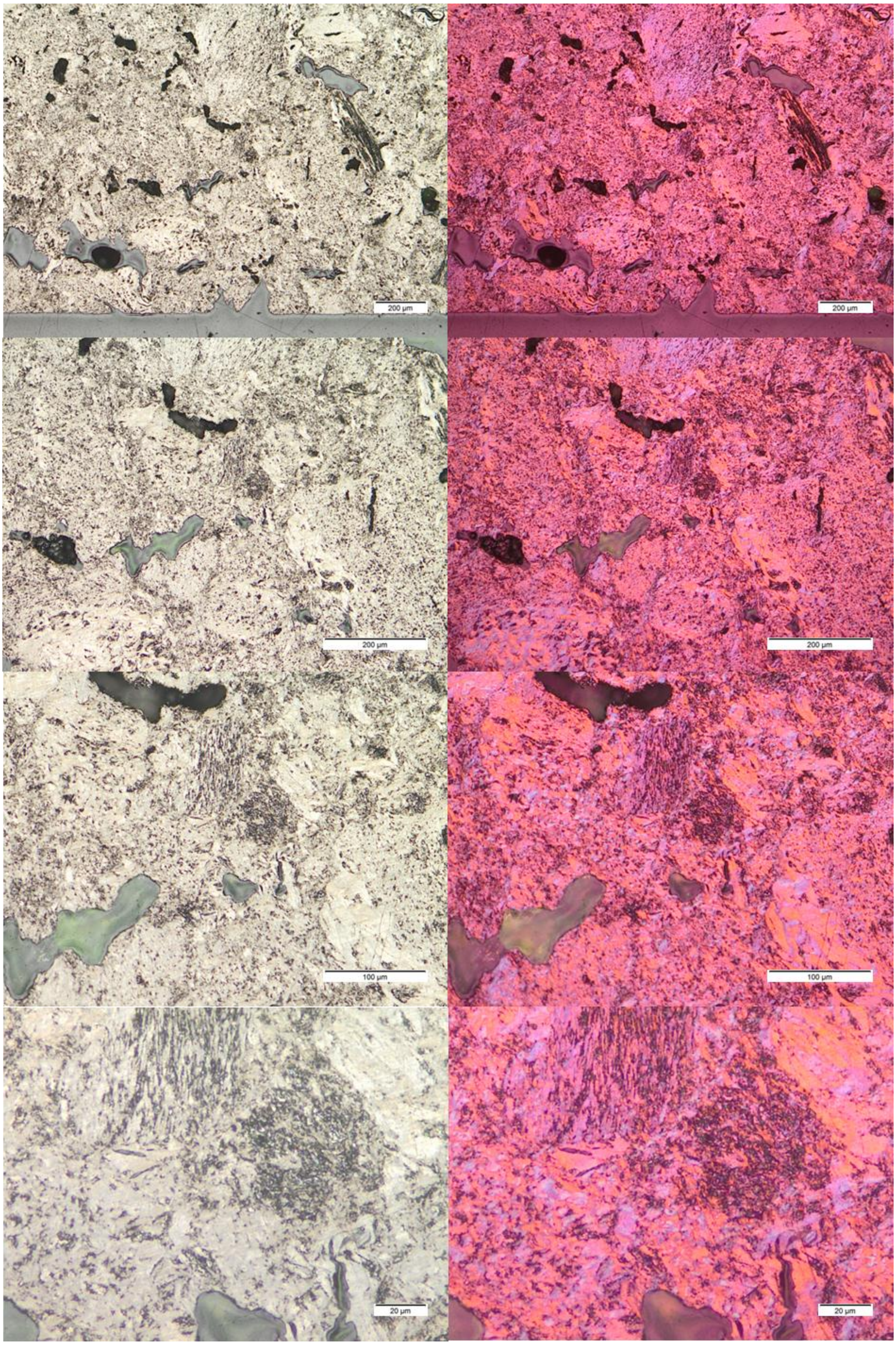

Figure 3-18 Bright field (left) and polarisation (right) micrographs of PCEA specimen 0U32

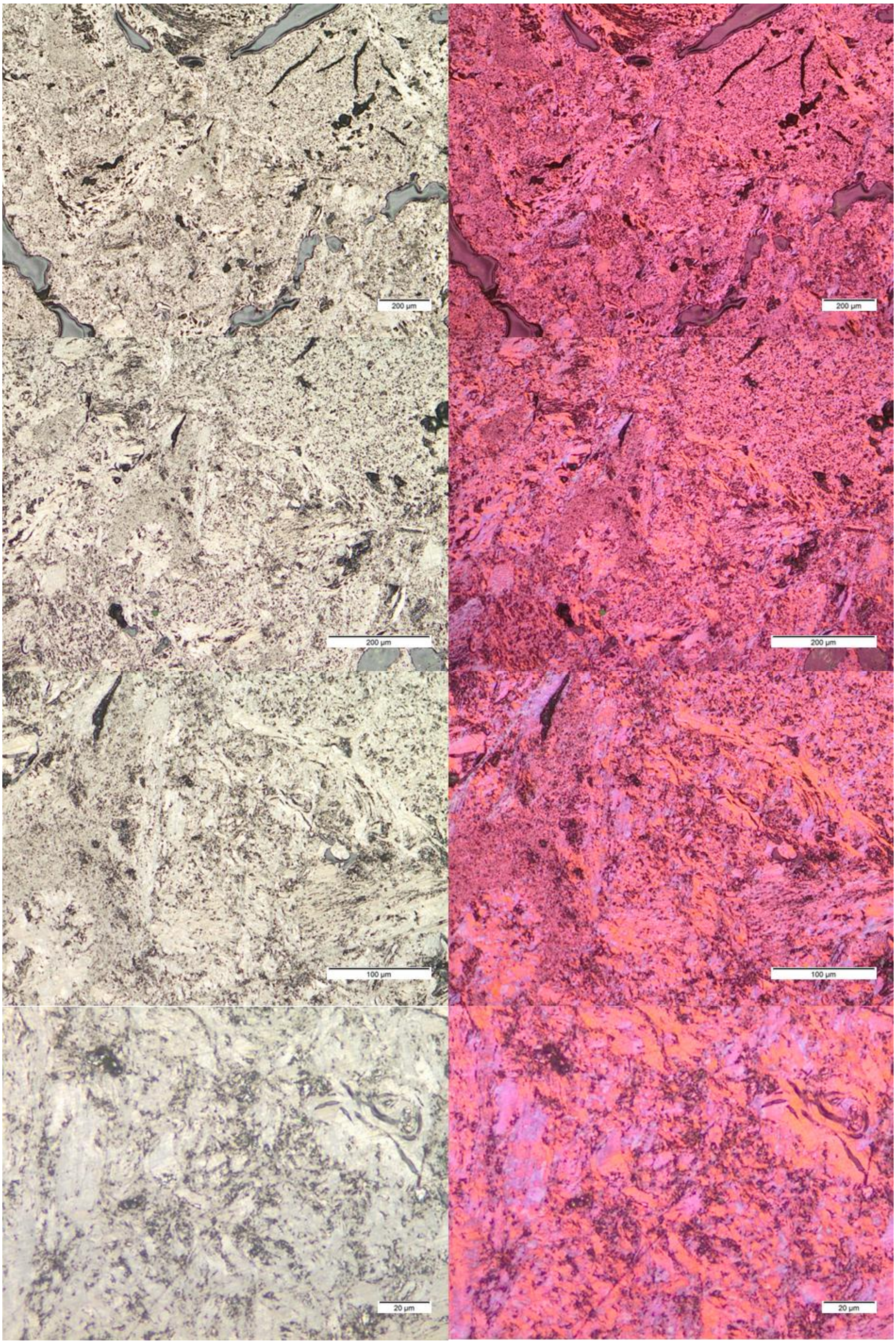

Figure 3-19 Bright field (left) and polarisation (right) micrographs of PCEA specimen 0U32

3.1.6 PCIB micrographs

Figure 3-20 to Figure 3-22 show the micrographs of irradiated PCIB sample 1U07 at 2.2 dpa.

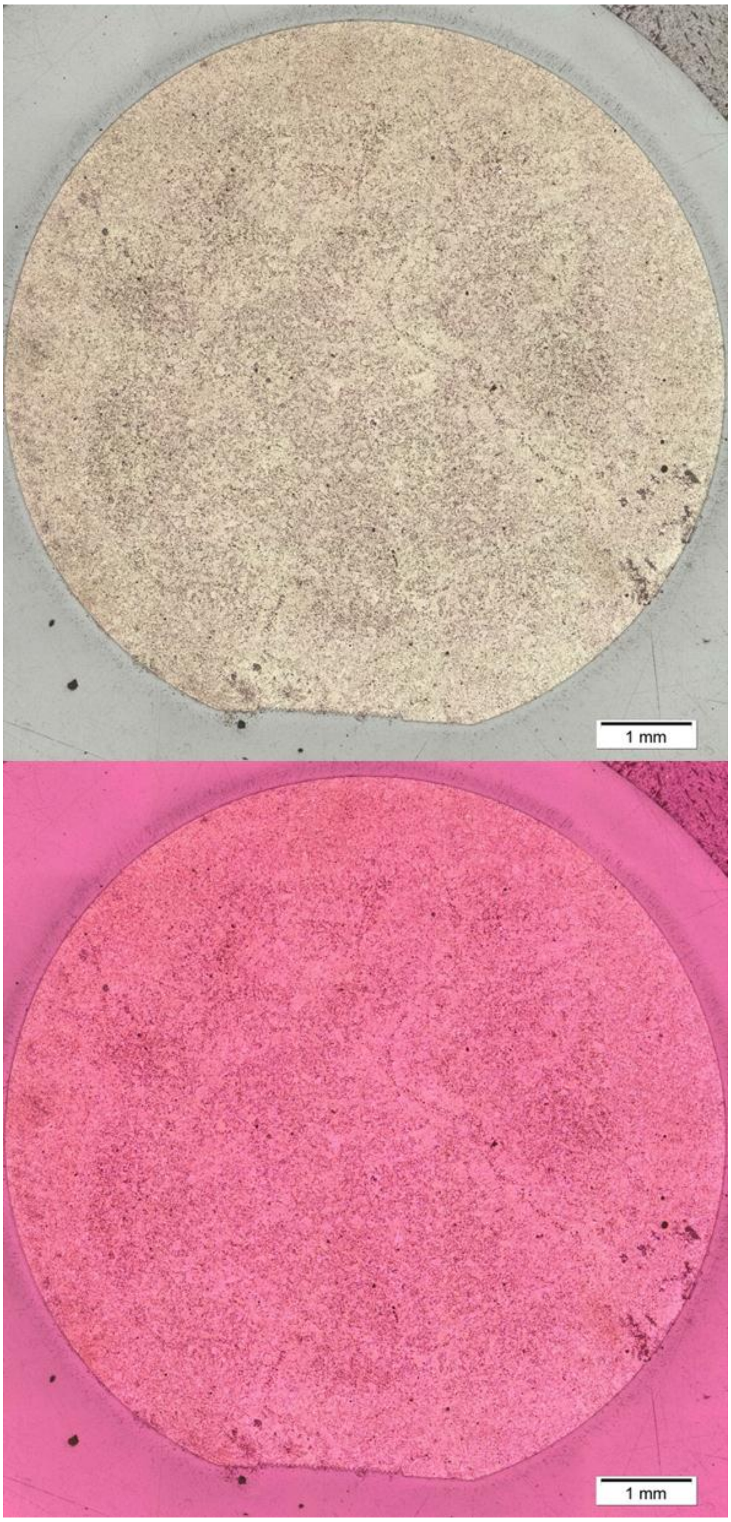

Figure 3-20 Bright field (top panel) and polarisation (bottom panel) overview micrographs of PCIB specimen 1U07 irradiated to 2.2 dpa

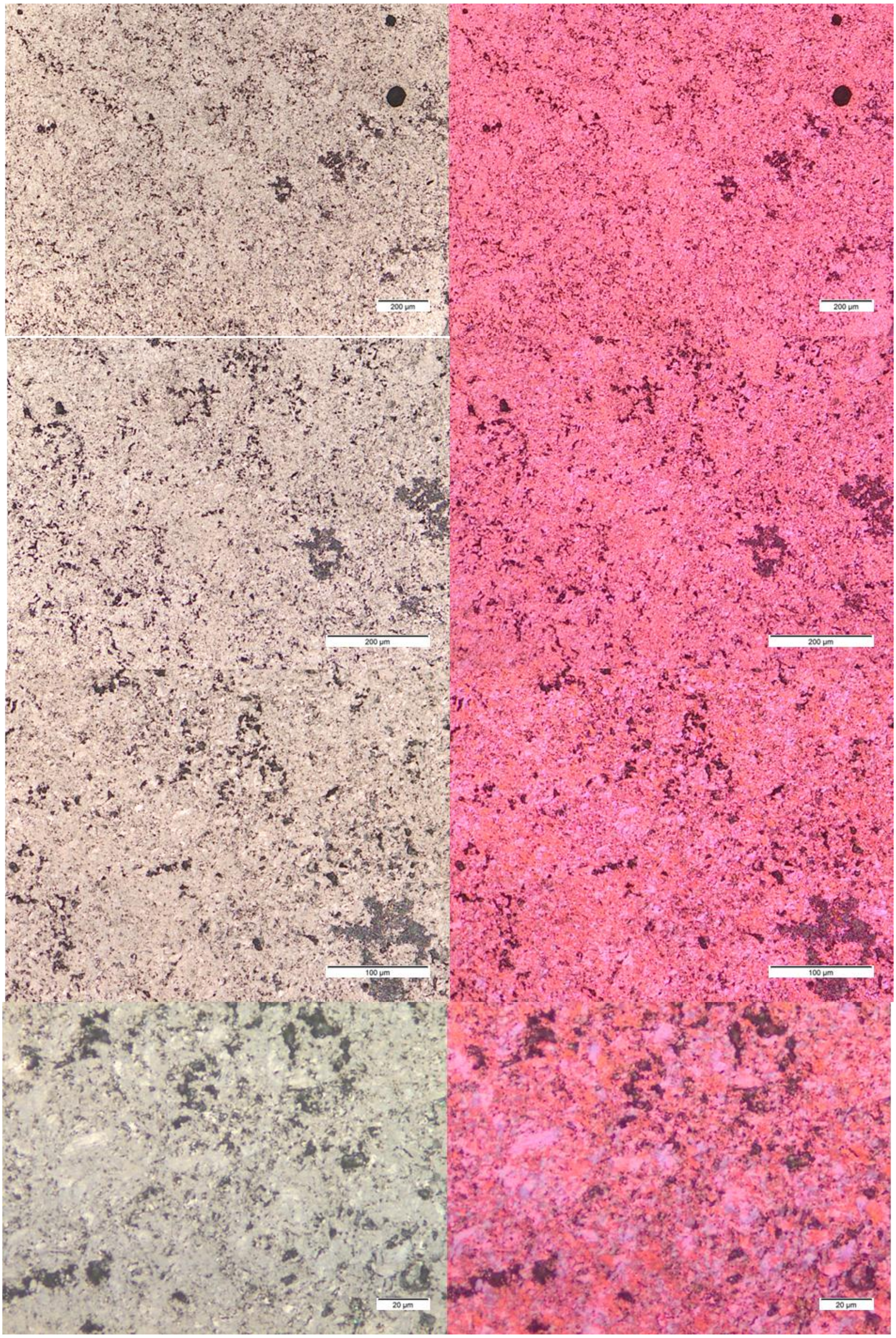

Figure 3-21 Bright field (left) and polarisation (right) micrographs of PCIB specimen 1U07

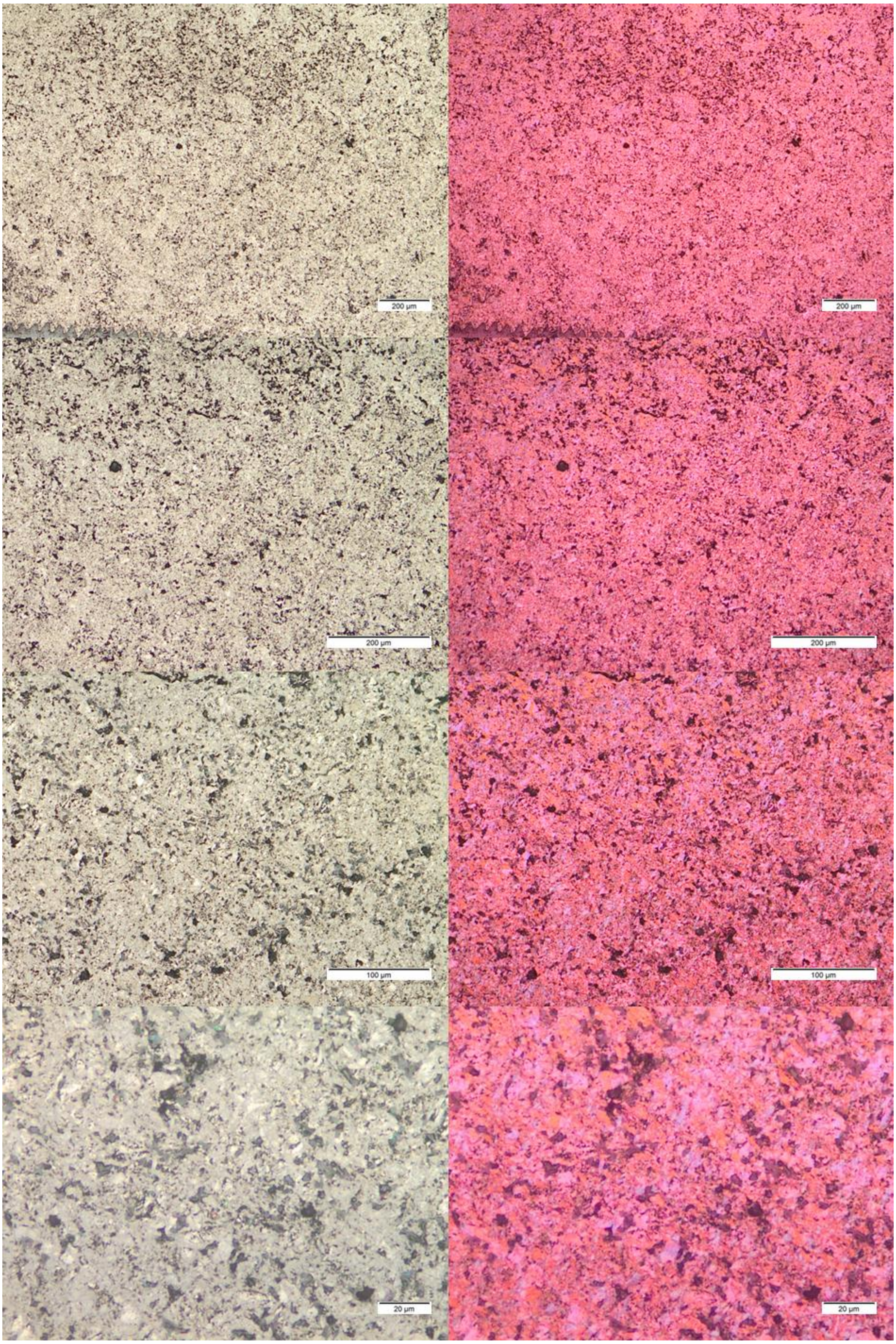

Figure 3-22 Bright field (left) and polarisation (right) micrographs of PCIB specimen 1U07

3.1.7 PPEA micrographs

Figure 3-23 to Figure 3-26 show the micrographs of irradiated PPEA sample 2U03 at 1.7 dpa.

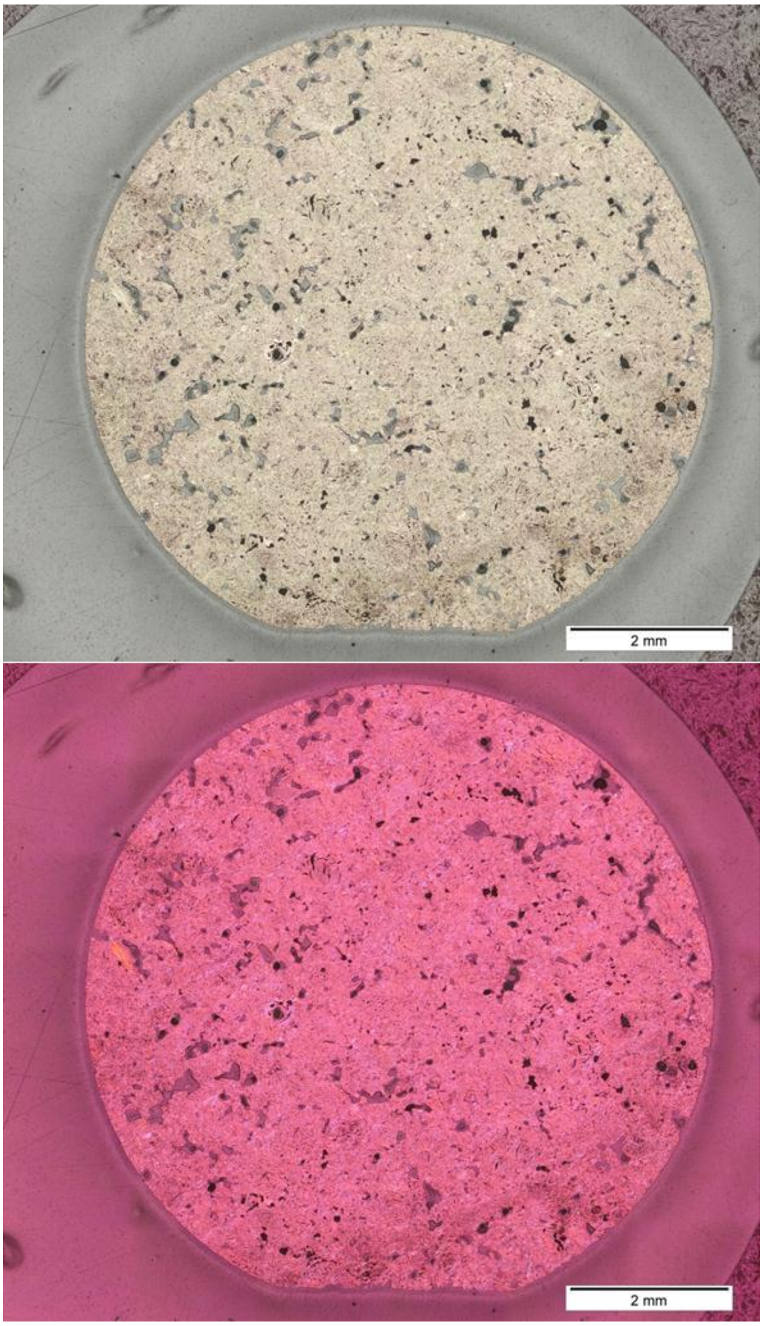

Figure 3-23 Bright field (top panel) and polarisation (bottom panel) overview micrographs of PPEA specimen 2U03 irradiated to 1.7 dpa

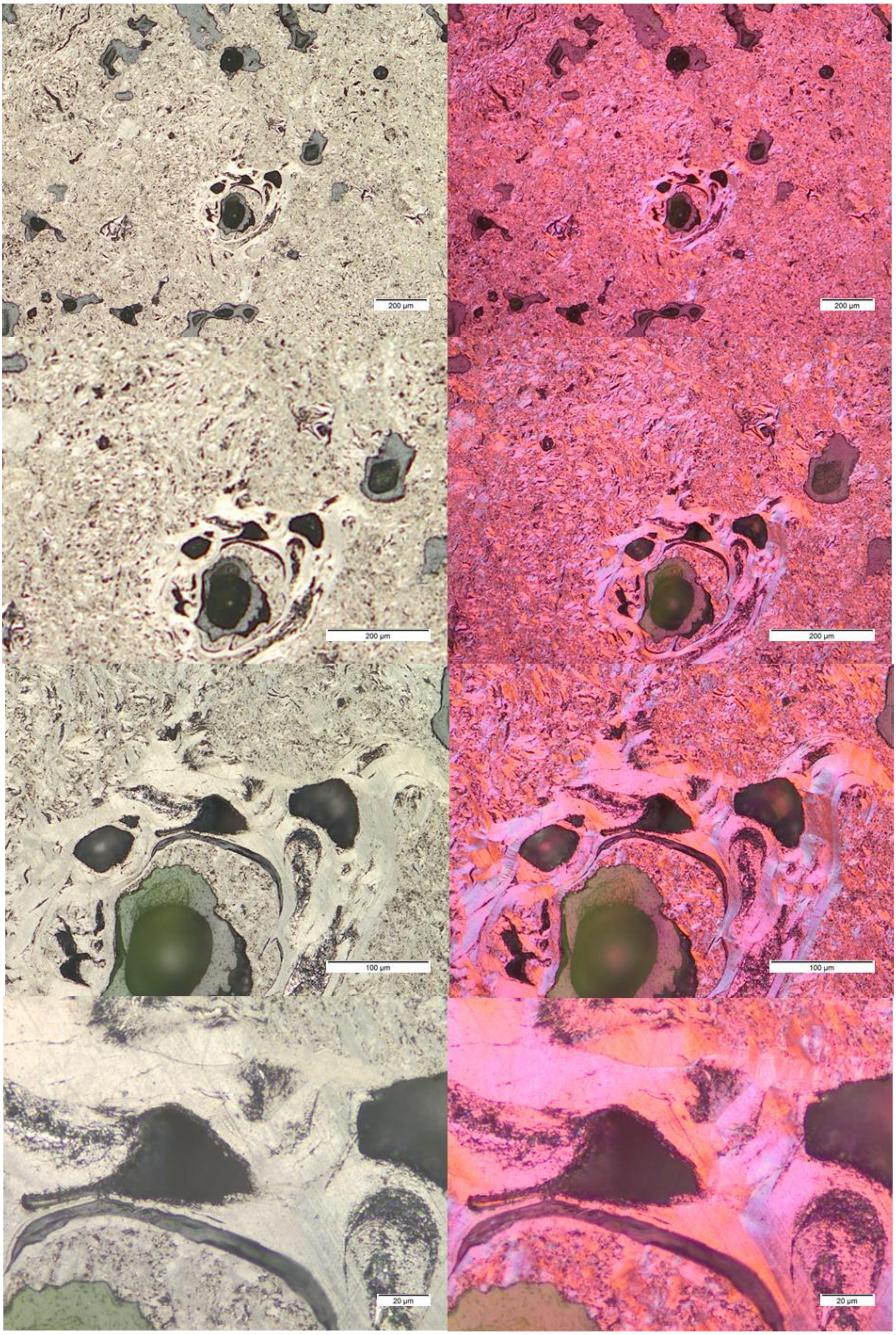

Figure 3-24 Bright field (left) and polarisation (right) micrographs of PPEA specimen 2U03

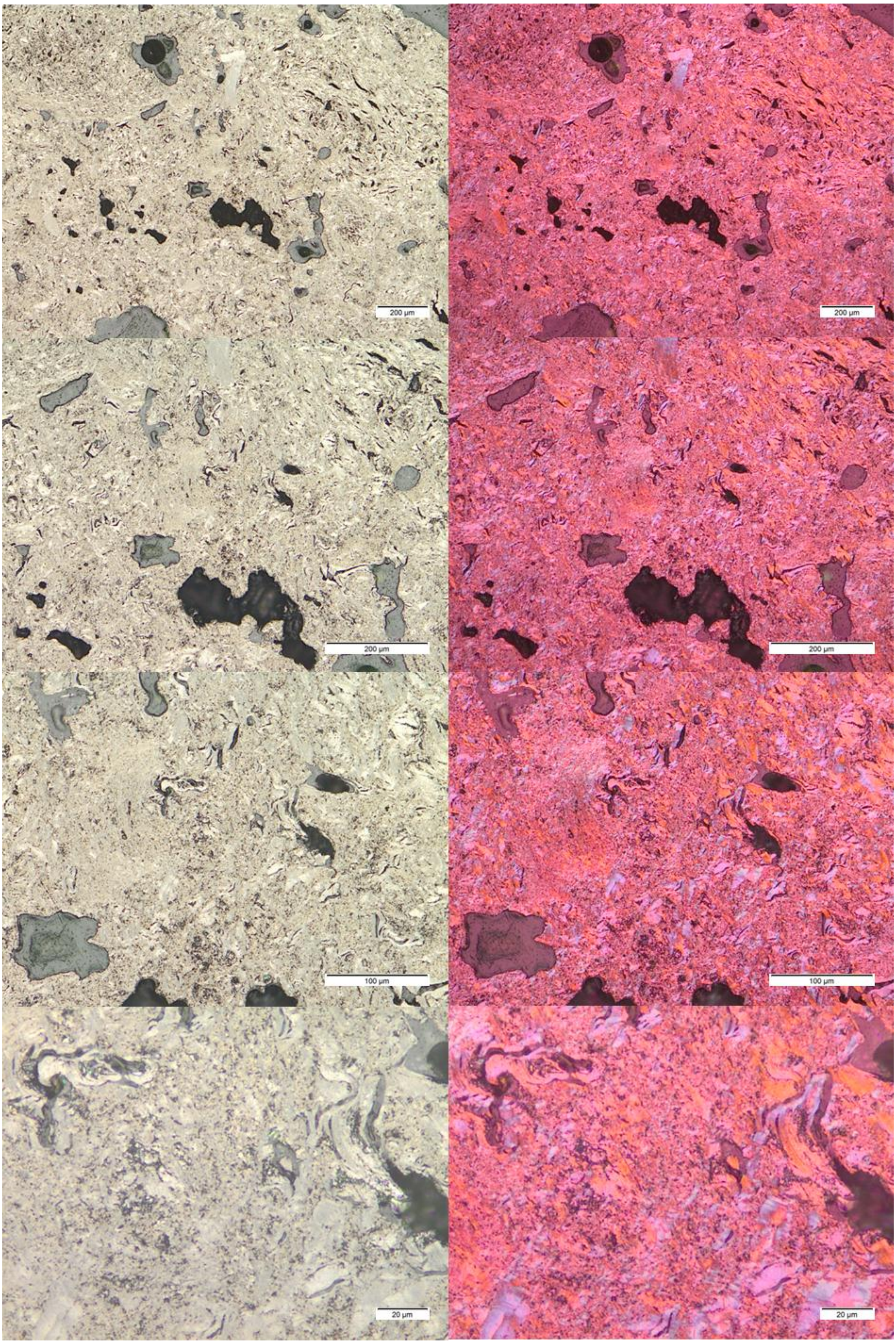

Figure 3-25 Bright field (left) and polarisation (right) micrographs of PPEA specimen 2U03

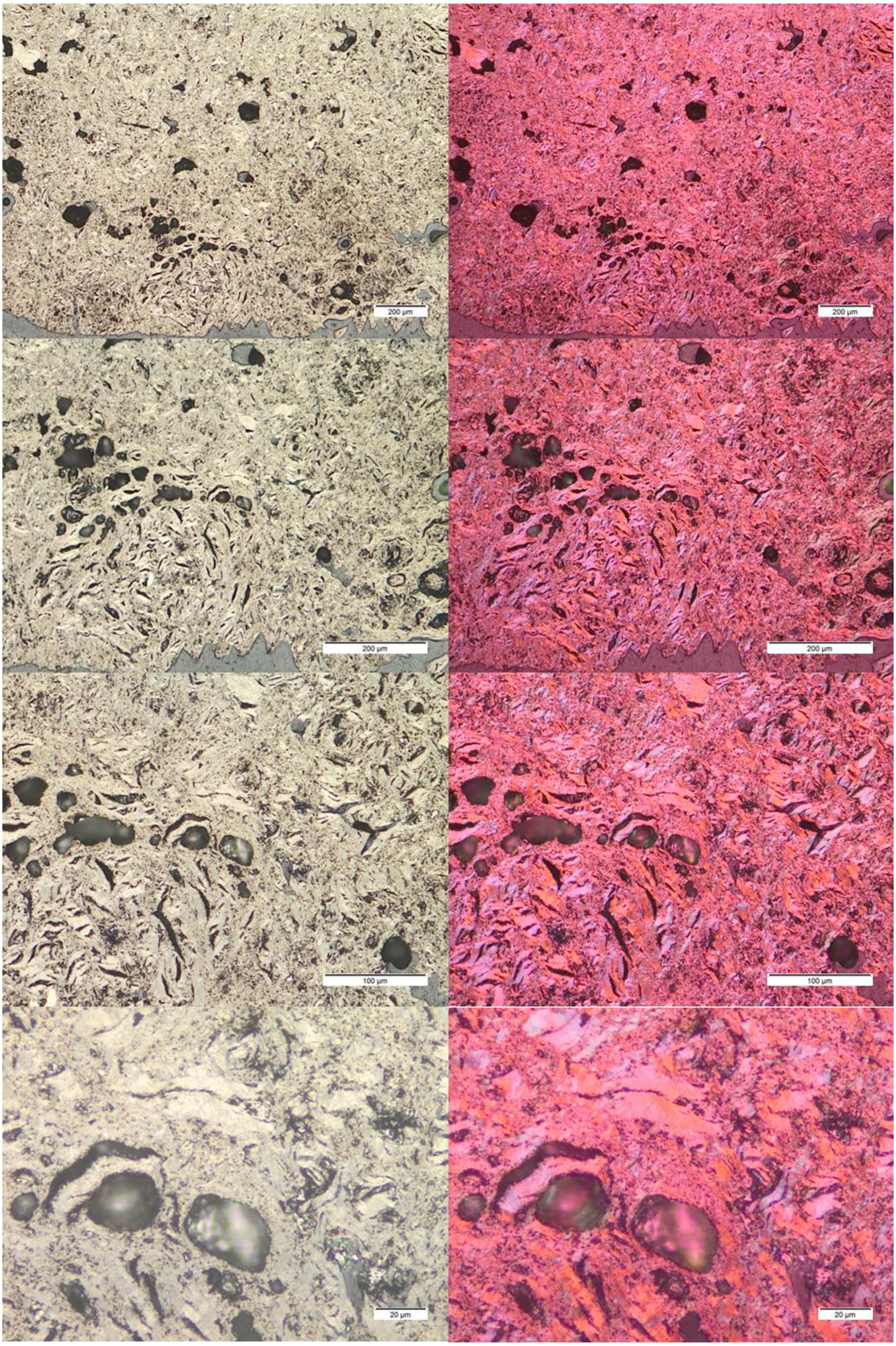

Figure 3-26 Bright field (left) and polarisation (right) micrographs of PPEA specimen 2U03

3.2 Scanning Electron Microscopy

The INNOGRAPH-1C specimens selected for optical microscopy were also used for Scanning Electron Microscopy (SEM). In paragraphs 3.2.1 to 3.2.7, the SEM results for these graphite specimens are presented. Images are taken at 3 kV and 20 kV, resulting in different appearances of the sample surface.

3.2.1 NBG-10

Figure 3-27 and Figure 3-28 show SEM images of NBG-10 sample 0S27 that has been irradiated to 2.2 dpa. The figures show two locations on the sample at various magnifications and voltages.

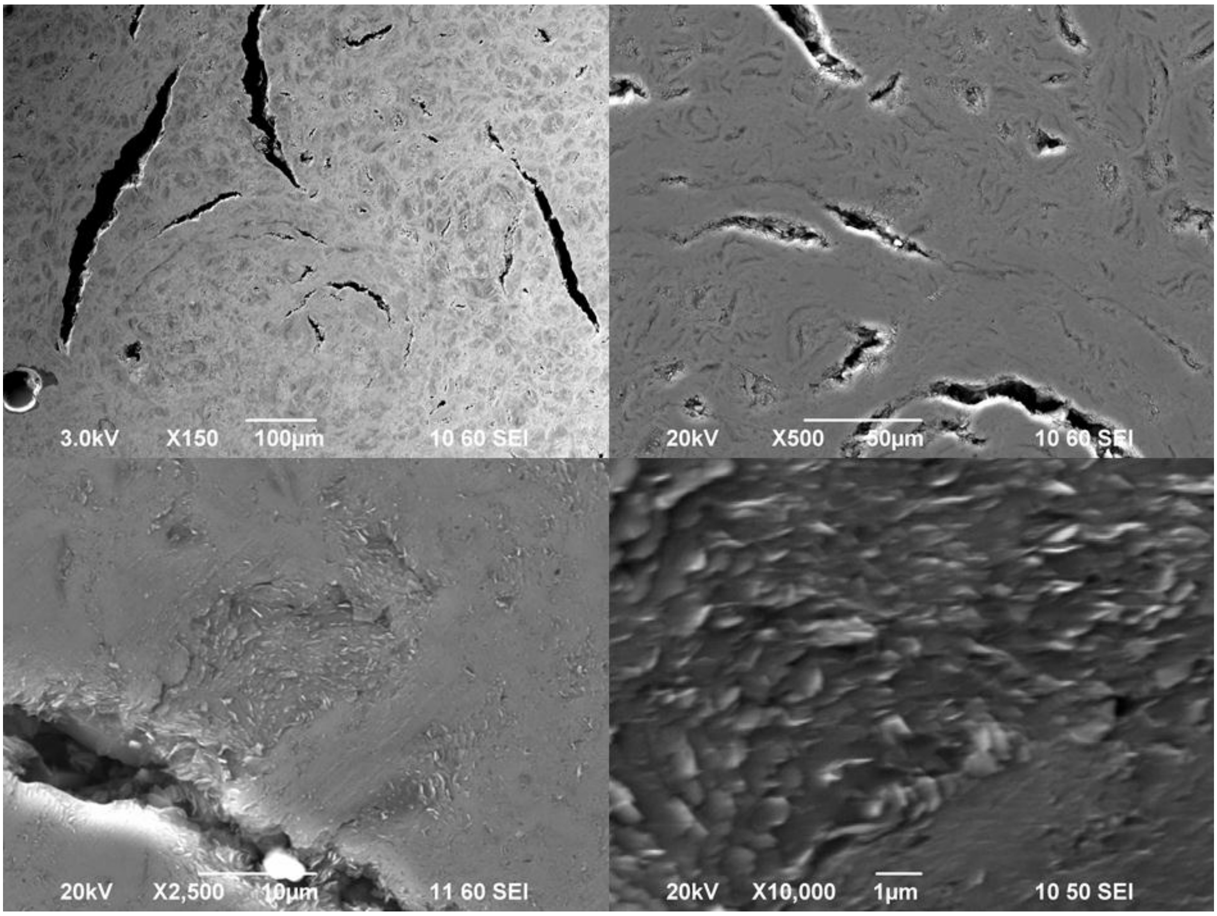

Figure 3-27 SEM images of NBG-10 sample 0S27, irradiated to 2.2 dpa

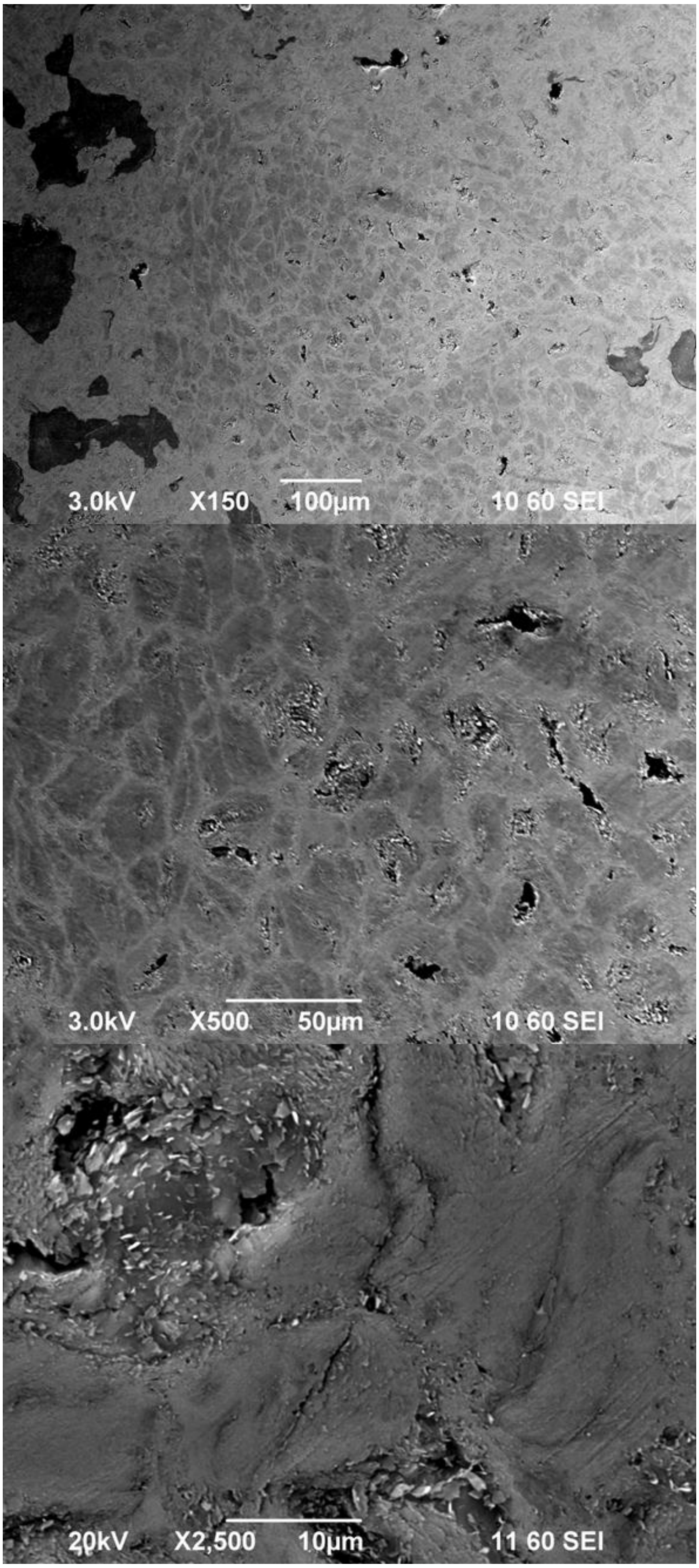

Figure 3-28 SEM images of NBG-10 sample 0S27, irradiated to 2.2 dpa

3.2.2 NBG-17

Figure 3-29 and Figure 3-30 show SEM images of NBG-17 sample 1S02 that has been irradiated to 2.1 dpa. The figures show two locations on the sample at various magnifications and voltages.

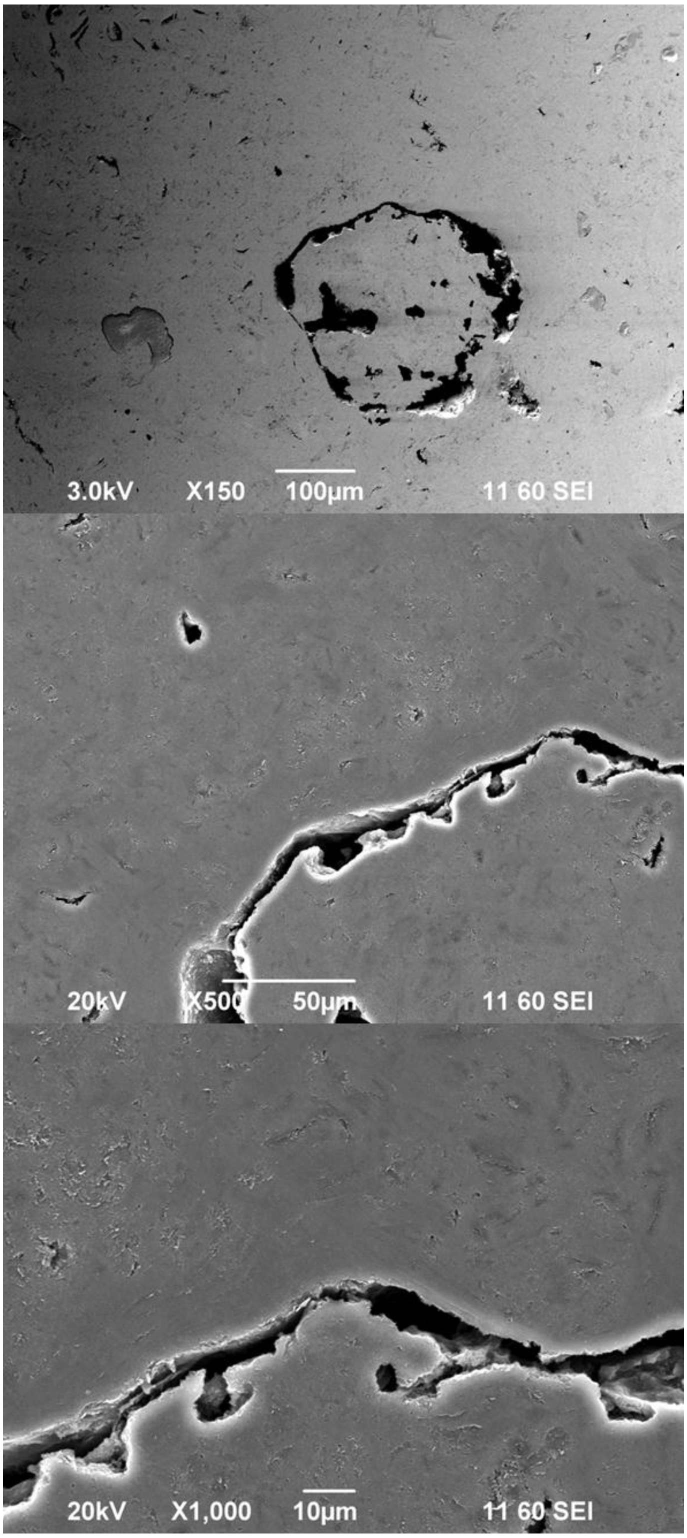

Figure 3-29 SEM images of NBG-17 sample 1S02, irradiated to 2.1 dpa

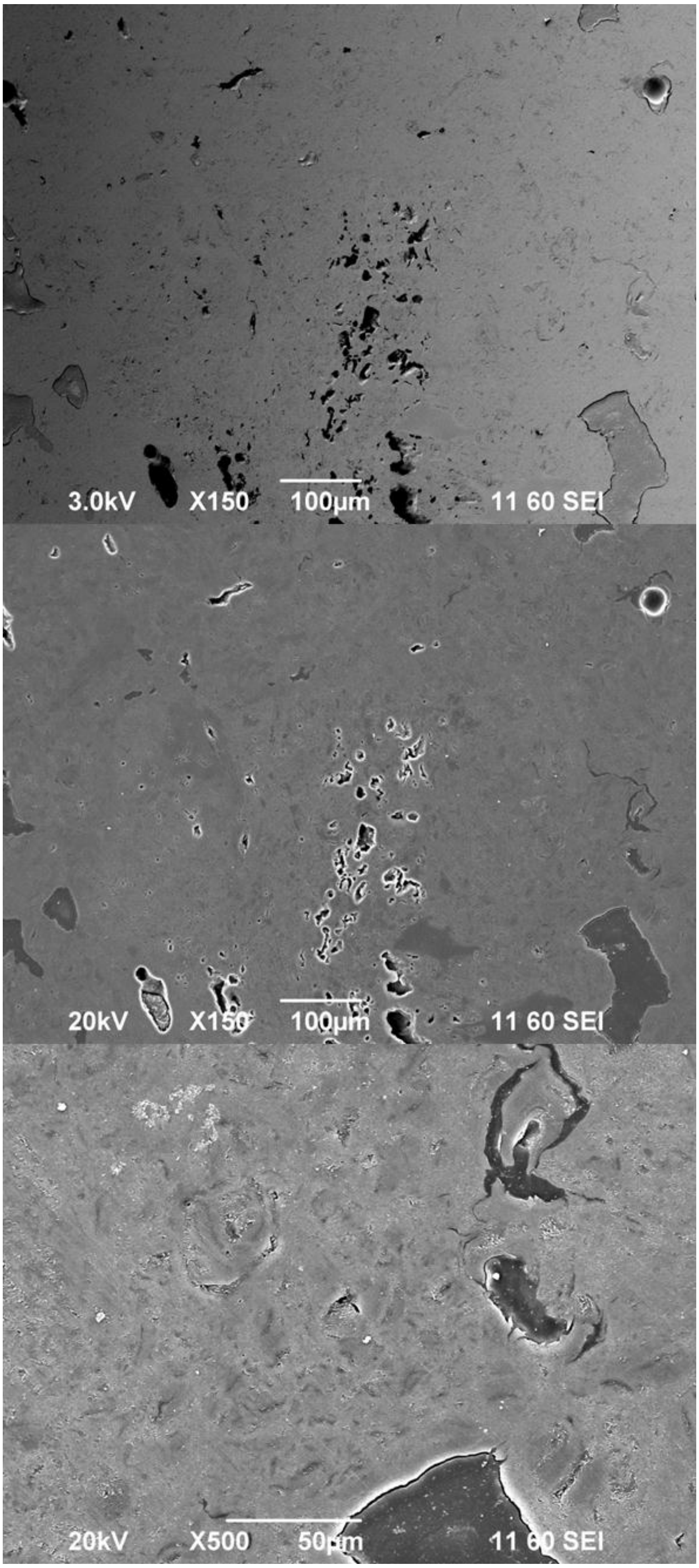

Figure 3-30 SEM images of NBG-17 sample 1S02, irradiated to 2.1 dpa

3.2.3 NBG-18

Figure 3-31 and Figure 3-32 show SEM images of NBG-18 sample 2S01 that has been irradiated to 2.2 dpa. The figures show two locations on the sample at various magnifications and voltages.

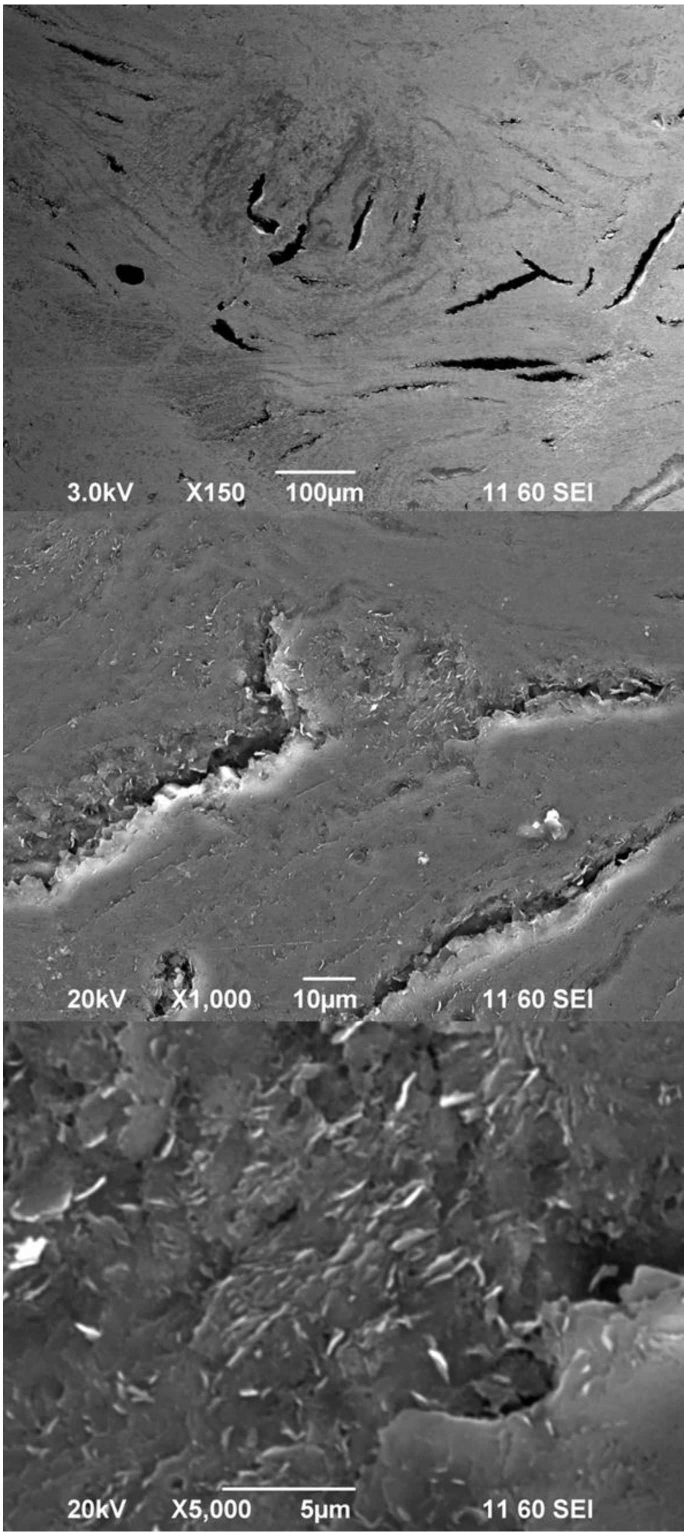

Figure 3-31 SEM images of NBG-18 sample 2S01, irradiated to 2.2 dpa

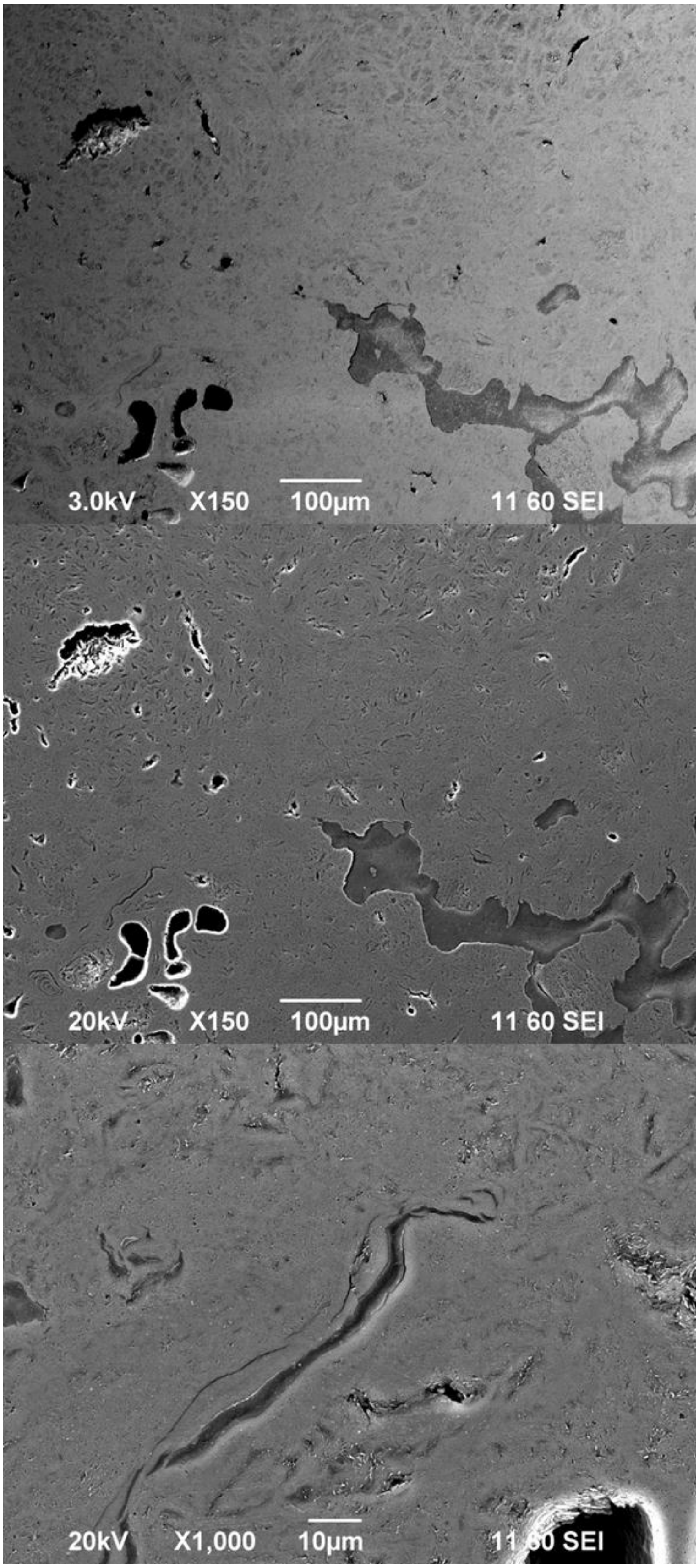

Figure 3-32 SEM images of NBG-18 sample 2S01, irradiated to 2.2 dpa

3.2.4 NBG-25

Figure 3-33 and Figure 3-34 show SEM images of NBG-25 sample 3S37 that has been irradiated to 2.2 dpa. The figures show two locations on the sample at various magnifications and voltages.

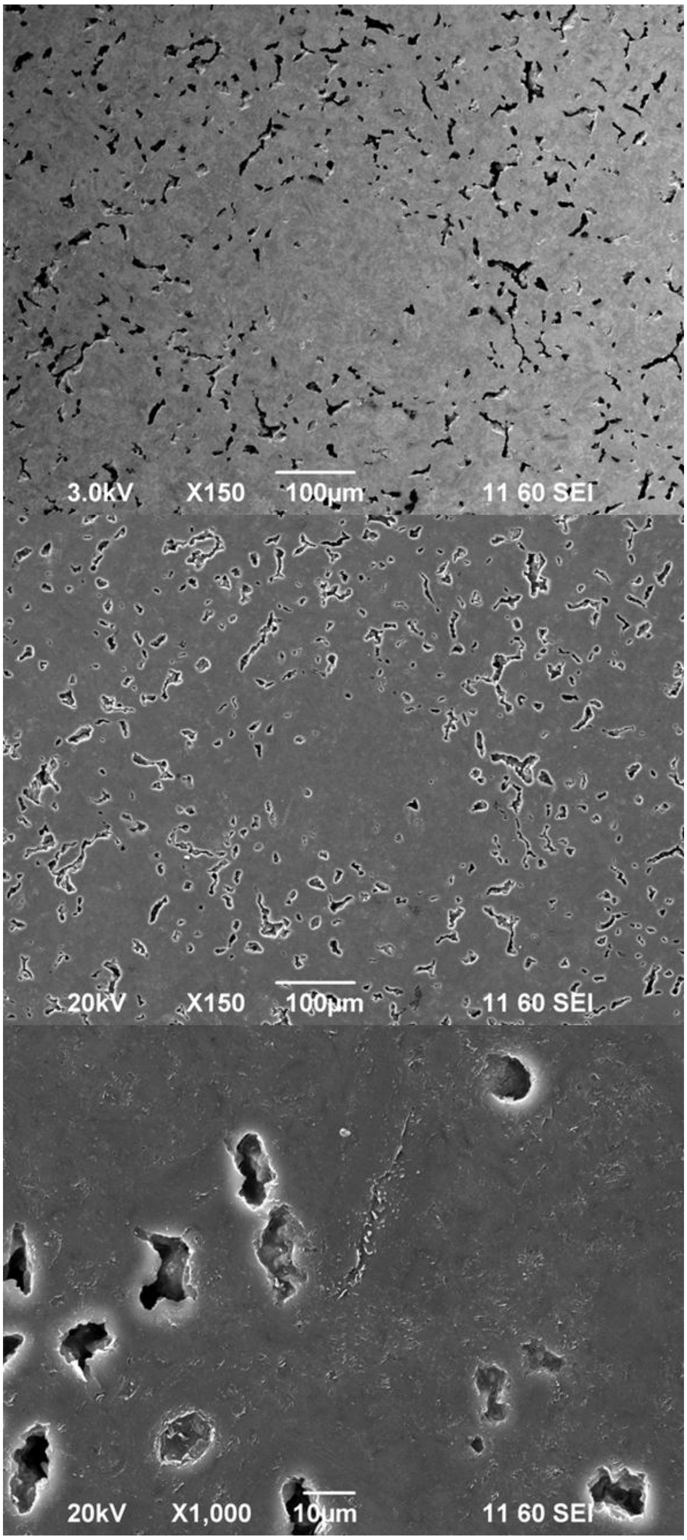

Figure 3-33 SEM images of NBG-25 sample 3S37, irradiated to 2.2 dpa

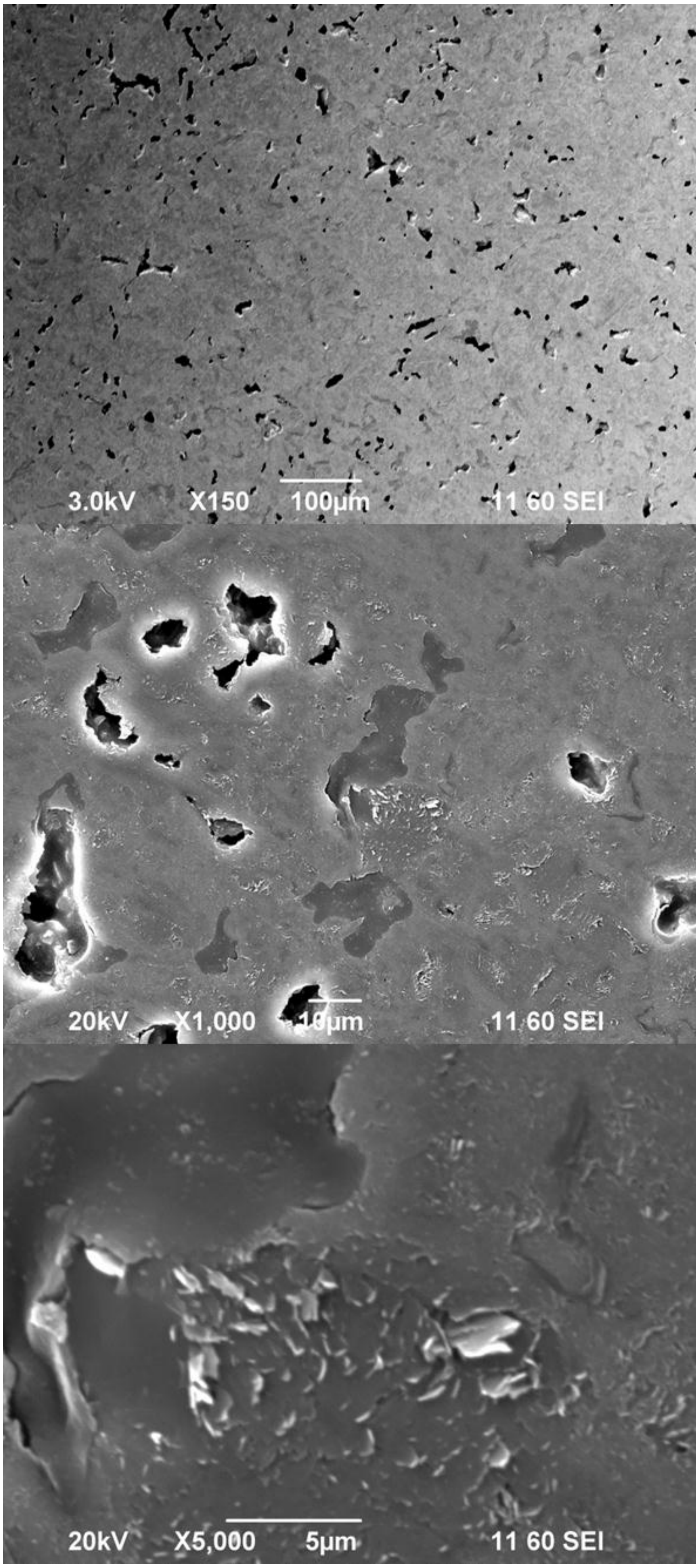

Figure 3-34 SEM images of NBG-25 sample 3S37, irradiated to 2.2 dpa

3.2.5 PCEA

Figure 3-35 to Figure 3-37 show SEM images of PCEA sample 0U32 that has been irradiated to 2.2 dpa. The figures show two locations on the sample at various magnifications and voltages.

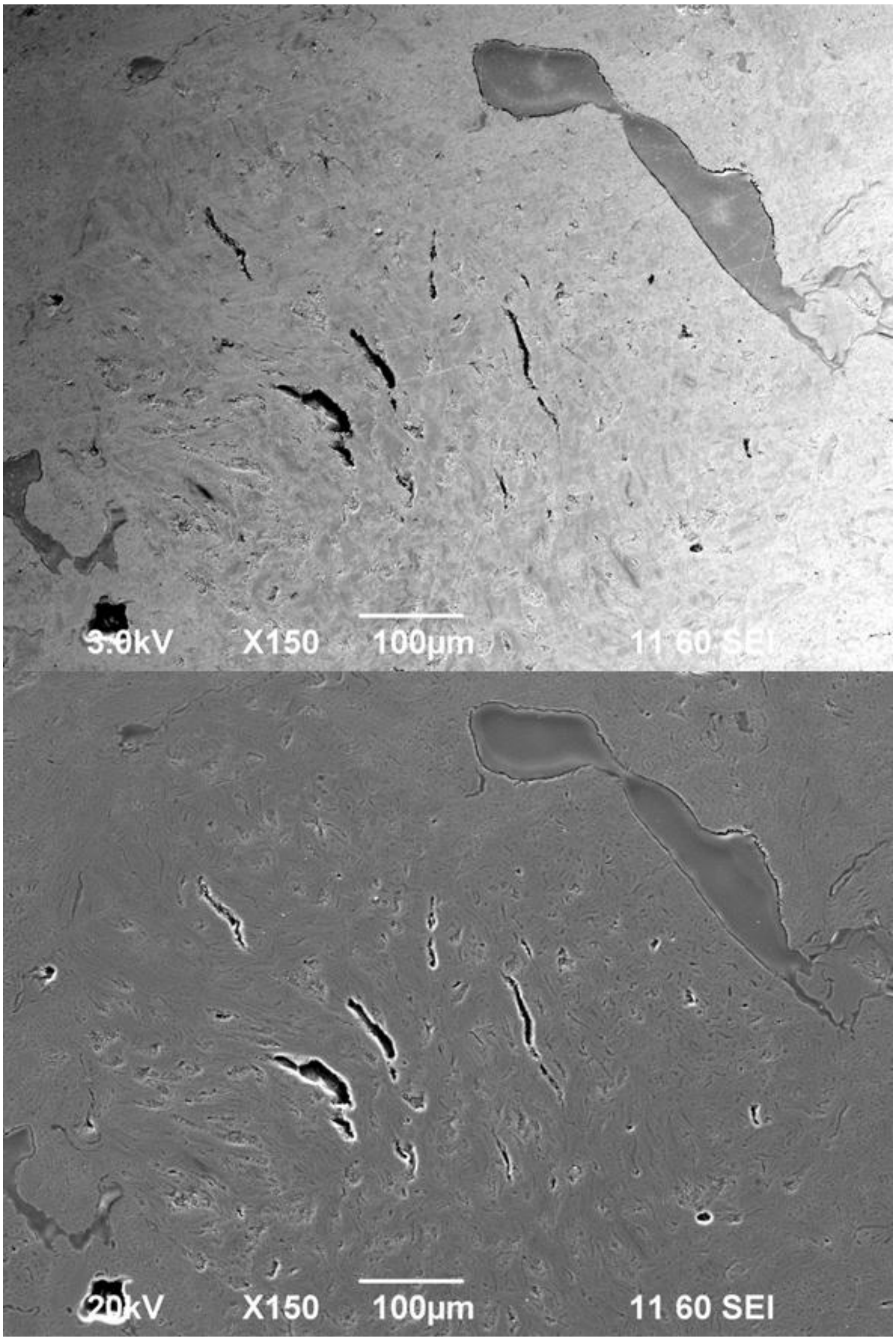

Figure 3-35 SEM images of PCEA sample 0U32, irradiated to 2.2 dpa

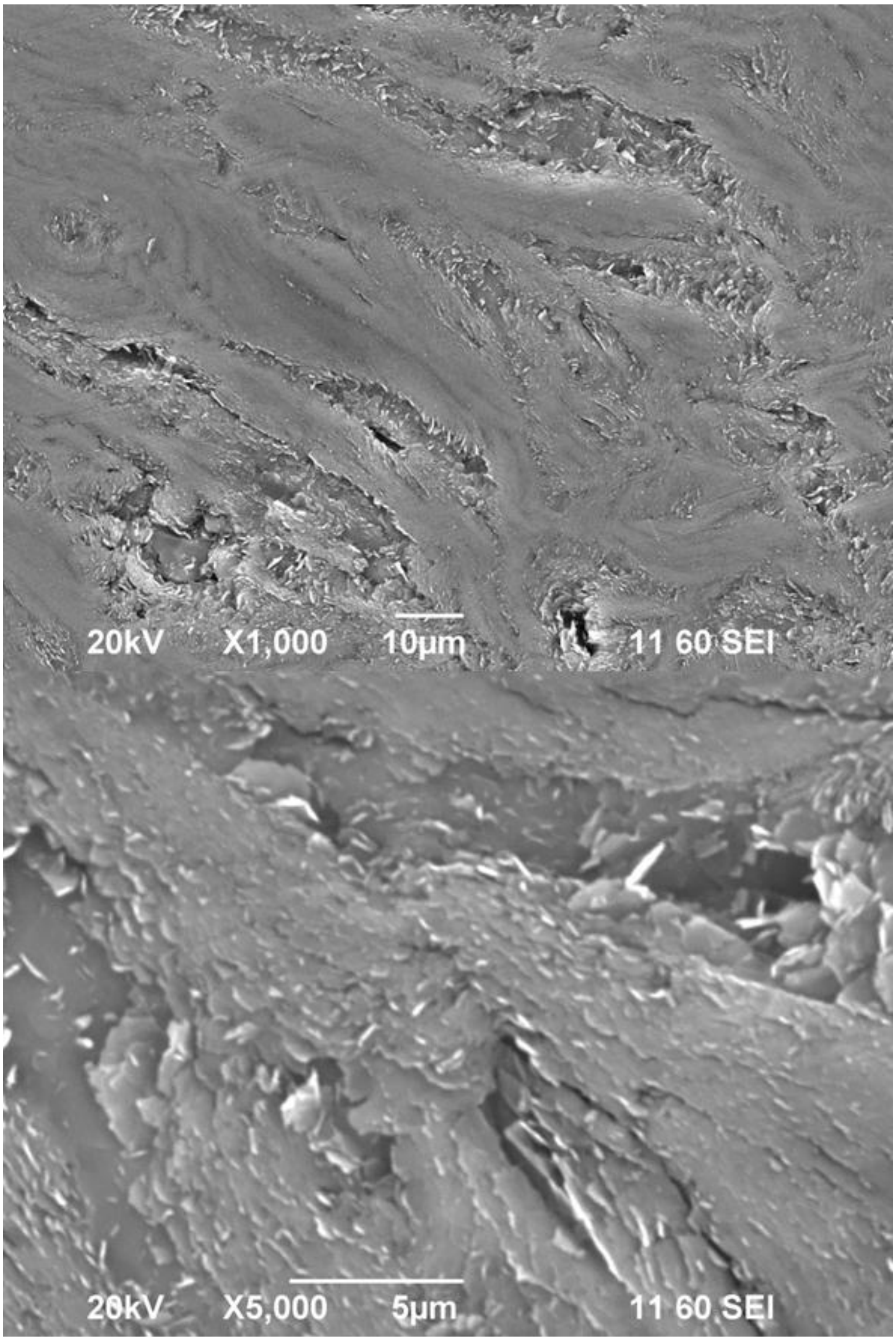

Figure 3-36 SEM images of PCEA sample 0U32, irradiated to 2.2 dpa; images are taken for the same location as Figure 3-35

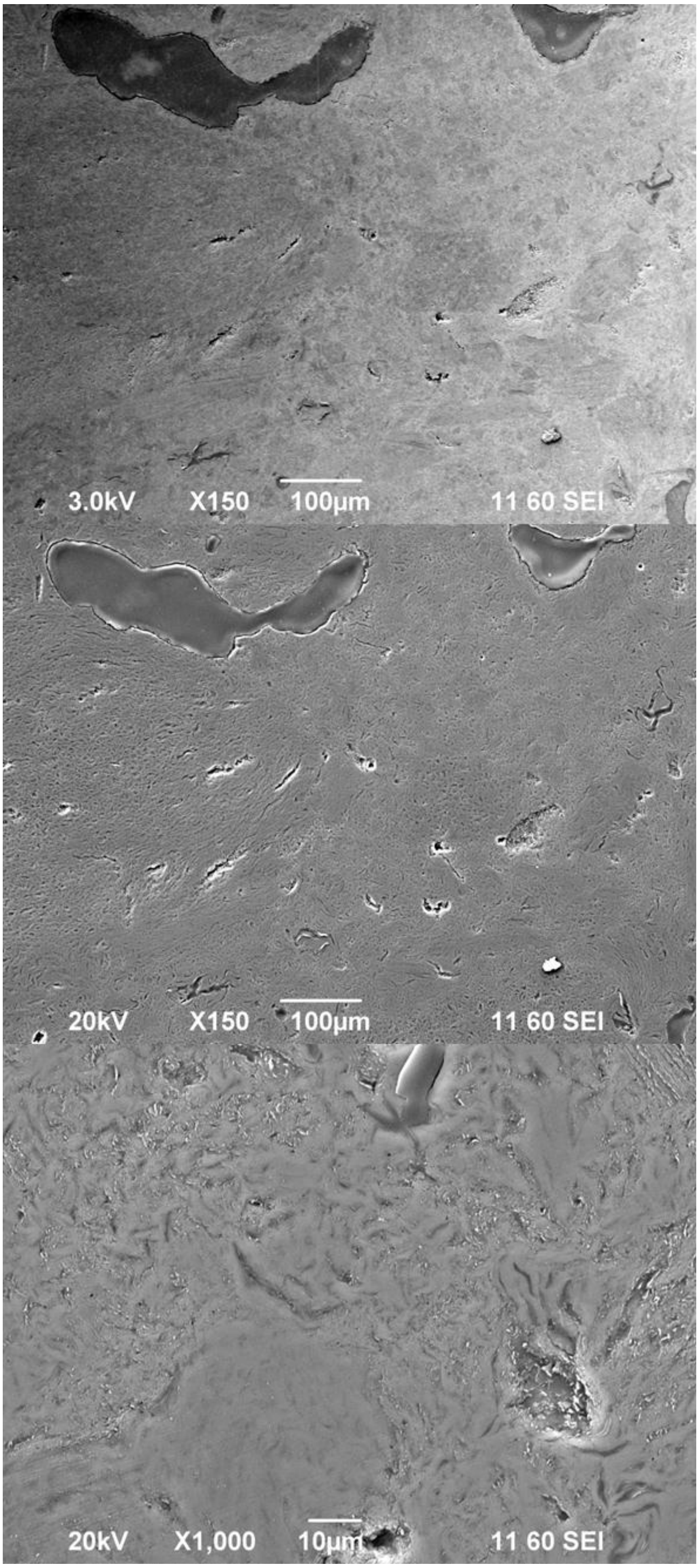

Figure 3-37 SEM images of PCEA sample 0U32, irradiated to 2.2 dpa

3.2.6 PCIB

Figure 3-38 and Figure 3-39 show SEM images of PCIB sample 1U07 that has been irradiated to 2.2 dpa. The figures show two locations on the sample at various magnifications and voltages.

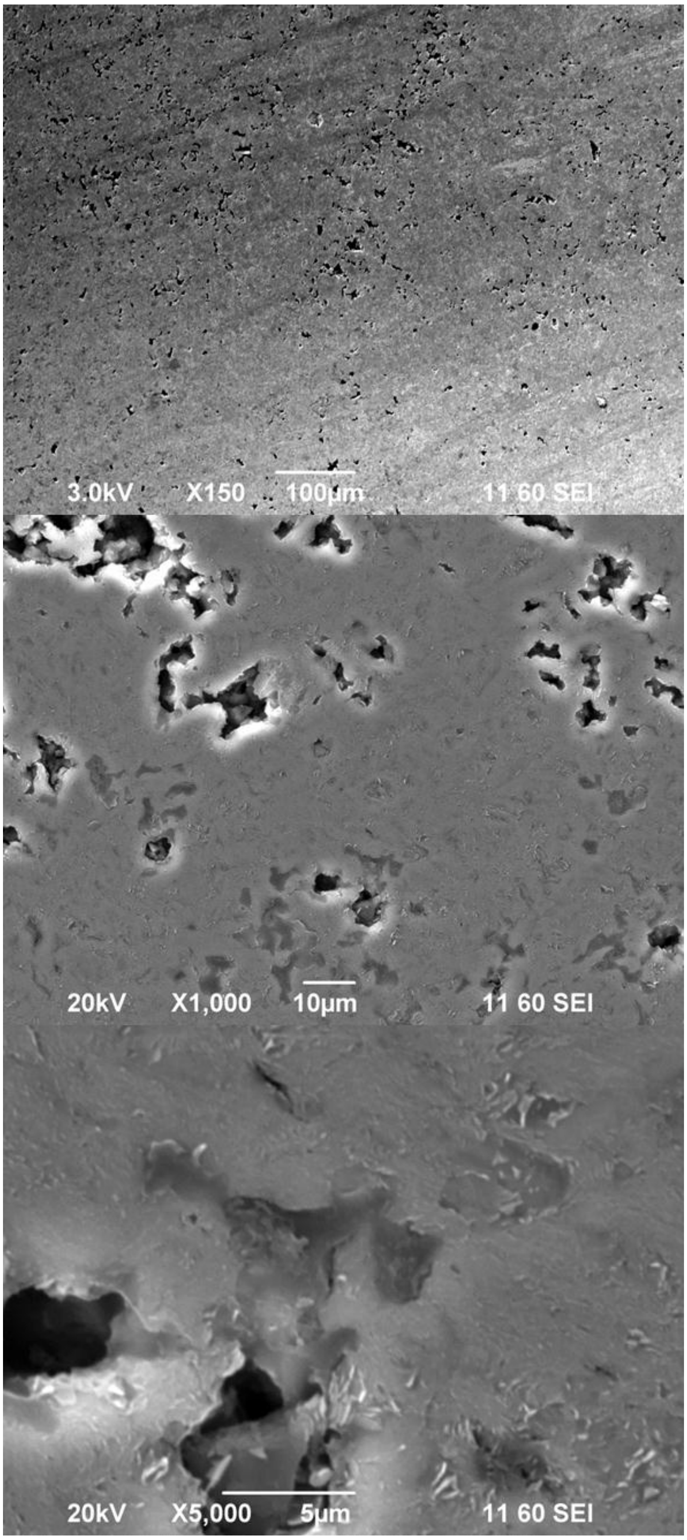

Figure 3-38 SEM images of PCIB sample 1U07, irradiated to 2.2 dpa

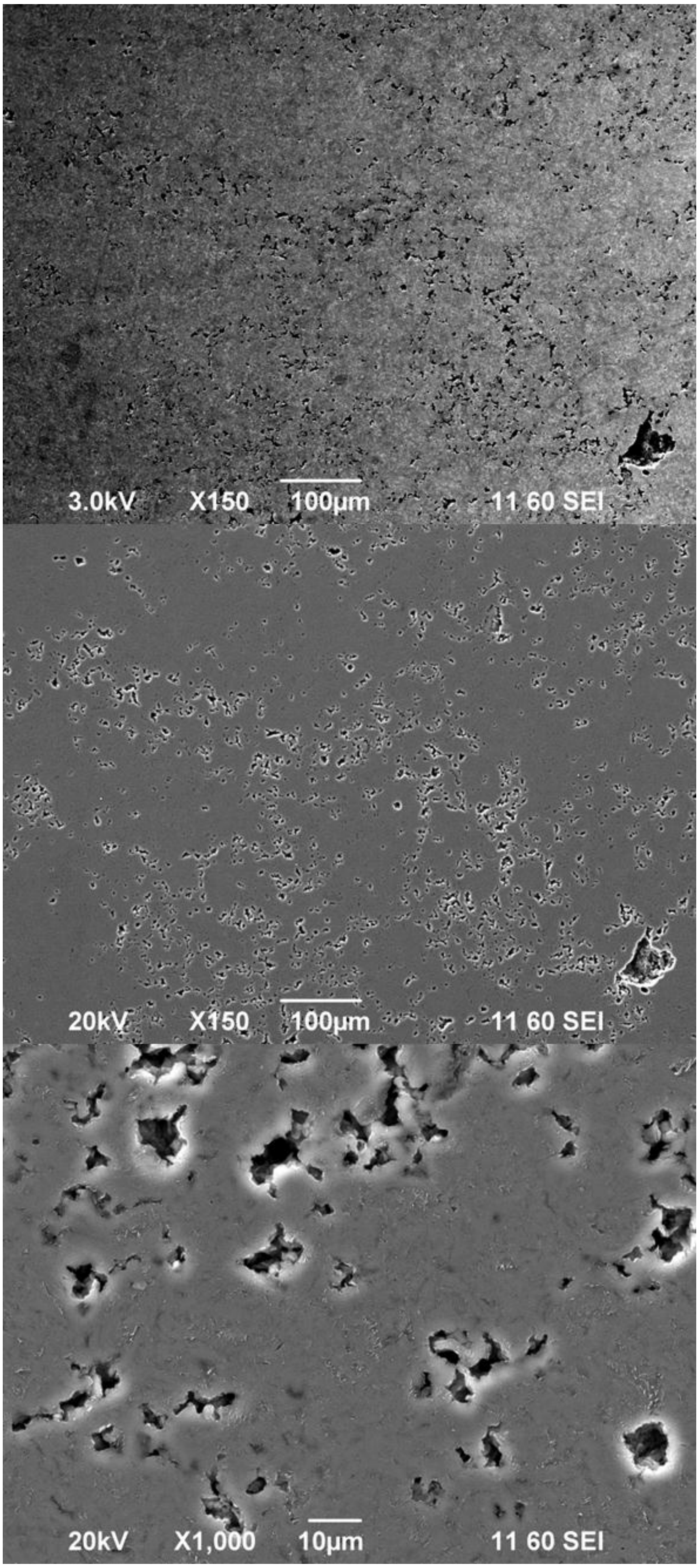

Figure 3-39 SEM images of PCIB sample 1U07, irradiated to 2.2 dpa

3.2.7 PPEA

Figure 3-40 and Figure 3-41 show SEM images of PPEA sample 2U03 that has been irradiated to 1.7 dpa. The figures show two locations on the sample at various magnifications and voltages.

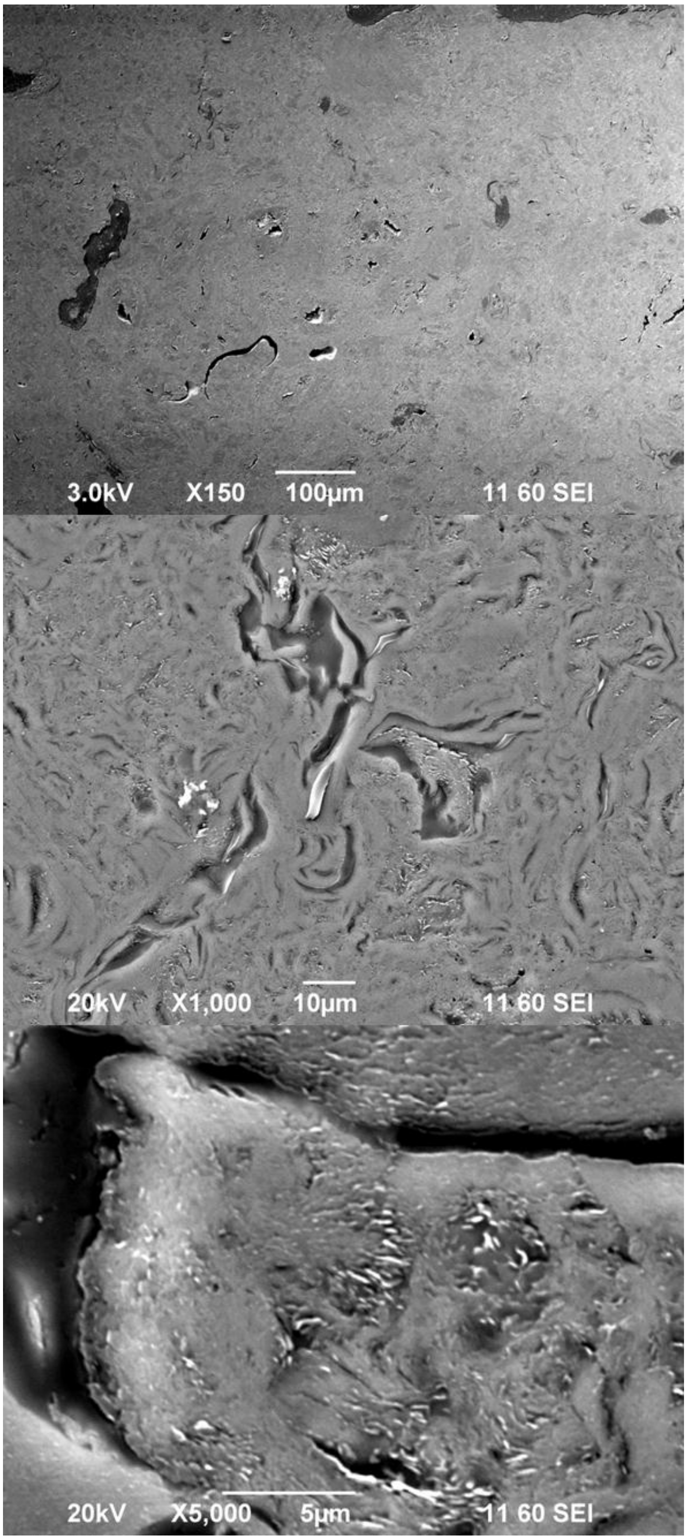

Figure 3-40 SEM images of PPEA sample 2U03, irradiated to 1.7 dpa

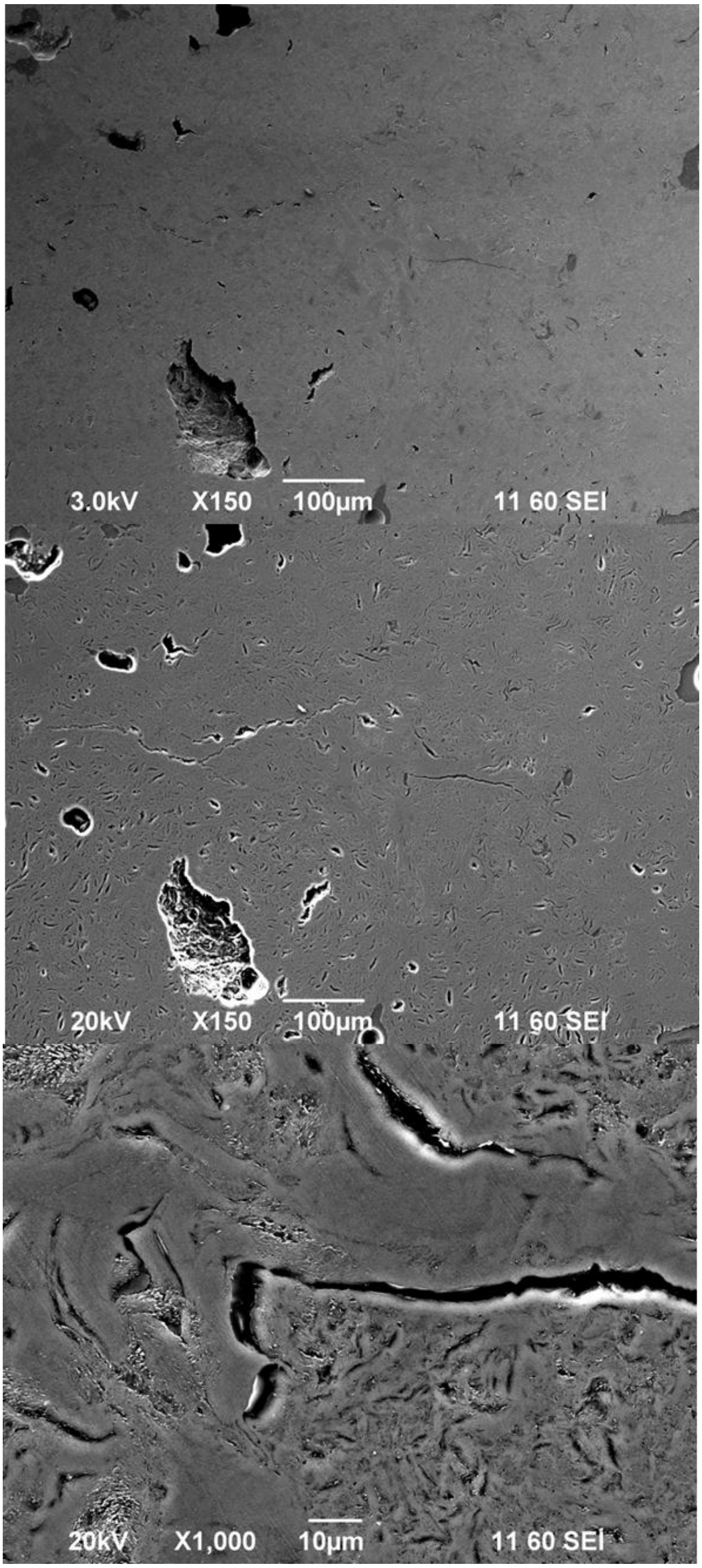

Figure 3-41 SEM images of PPEA sample 2U03, irradiated to 1.7 dpa

4 Conclusion

Seven graphite samples irradiated in the INNOGRAPH-1C irradiation rig were prepared for optical microscopy and scanning electron microscopy. Both optical microscopy and SEM were succesfully performed on these samples.

5 References

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