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Uporaba fokusiranega ionskega snopa pri karakterizaciji livnih Al-zlitin

Application of a focussed ion beam by characterization of casting Al-alloys

Povzetek

Mikrostrukture livnih aluminijevih zlitin vsebujejo številne mikrostrukturne sestavine mikro- ali nanovelikosti. Za metalografsko analizo največ uporabljamo standardne metode, kot so svetlobna mikroskopija, vrstična in presevna elektronska mikroskopija s številnimi analitičnimi metodami ter rentgenska difrakcija. Dodatne informacije o mikrostrukturi pa nam omogoča tudi metoda dvojnega snopa: fokusiranega ionskega snopa (FIB) in elektronskega snopa (SEM).

Fokusirani ionski snop (FIB; angl. Focussed Ion Beam) običajno galijevih ionov lahko pa tudi ioni drugih kovin nastane z ekstrakcijo v električnem polju in ima premer od nekaj nanometrov do mikrometrov. Pospešeni ioni ob trku z vzorcem izbijajo atome in tako odstranjujejo snov. Pri tem nastanejo različni signali, ki jih uporabimo za slikanje mikrostrukture. V kombinaciji s SEM lahko s FIB obdelujemo površino in jo hkrati s SEM upodobimo.

V tem članku je predstavljena uporaba metode (FIB-SEM) pri metalografskih preiskavah izbranih aluminijevih livnih zlitin. Prednost te metode so predvsem prečni prerezi na točno določenih mestih ter odkrivanje razporeditve in oblike mikrostrukturnih sestavin pod površino vzorca. Na kratko je predstavljen tudi postopek izdelave serijskih rezov in prostorska (3D) rekonstrukcija mikrostrukture.

Ključne besede: aluminijeva zlitina, litje, fokusiran ionski snop, karakterizacija, mikrostruktura

Abstract

Microstructures of cast aluminium alloys consist of numerous micro- and nanosized microstructural constituents. For metallographic investigations, several standard methods have been used: light microscopy (LM), scanning (SEM) and transmission (TEM) electron microscopies combined with several analytical techniques, and X-ray diffraction (XRD). Additional information regarding the microstructure can be obtained by a dual beam microscopy: focussed ion beam (FIB) and electron beam (SEM).

The focussed ion beam is a device, in which a gallium ion beam is produced with a diameter ranging from few nanometres up to few micrometres. Collisions of ions with a sample result in scattering of atoms and ions from its surface, causing formation of different signals. By the application of a dual beam, the FIB modifies the surface, while SEM is used for the imaging.

In this article, we present the use of the dual beam (FIB-SEM) for metallography of selected aluminium casting alloys. The advantage of this method is a possibility to create cross-sections at particular sites and reveal the shapes and distributions of microstructural constituents, which are below the surface of a sample. We briefly represent the procedure for making serial cuts and spatial (3D) reconstruction of microstructure.

Keywords: aluminium alloy, casting, focussed ion beam, characterization, microstructure

1 Uvod

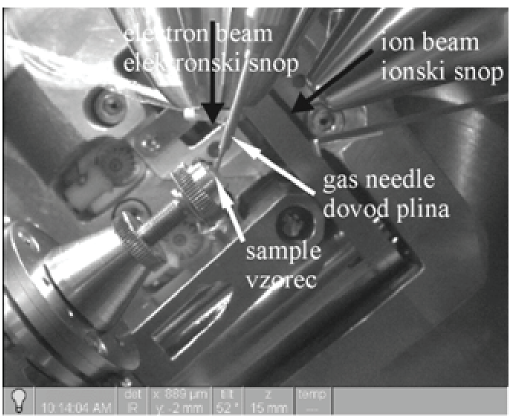
Fokusirani ionski snop (FIB) je naprava, v kateri nastane ionski curek s premerom nekaj nanometrov do nekaj mikrometrov [1]. Lahko se uporablja kot samostojna enota, vendar je večinoma dodatek vrstičnemu elektronskemu mikroskopu (SEM). V slednjem primeru govorimo o mikroskopu z dvojnim snopom (FIB-SEM) [2].

V fokusiranem ionskem snopu (FIB) so v večini primerov galijevi ioni. Poleg teh se v manjši meri še uporabljajo helijevi ali argonovi ioni [3]. Trki ionov s površino vzorca povzročijo izbijanje atomov s površine, kar imenujemo razprševanje. Hitrost razprševanje je sorazmerna razpršitvenemu koeficientu. FIB se lahko uporablja za odstranjevanje ali dodajanje snovi z natančnostjo nekaj desetin nanometre. Zaradi tega se veliko uporablja

1 Introduction

The focussed ion beam (FIB) is a device, in which an ion beam is produced with a diameter ranging from few nanometres up to few micrometres [1]. It is available as a standalone device; however, it is predominantly an attachment to a scanning electron microscope (SEM). In the latter case, it is usually referred to as a dual-beam microscope (FIB-SEM) [2]. The interior of the chamber is shown in Fig. 1.

There are predominantly gallium ions in the ion beam. Additionally, FIB with helium or argon ions is also used in a smaller extend [3]. Collisions of ions with a sample scatter atoms from its surface. The rate of scattering is proportional to the scattering coefficient. FIB can be utilised for the removing and addition of substance with a precision of few ten of nanometres.



Slika 1. Notranjost komore mikroskopa z dvojnim snopom (FIB-SEM) v mikroskopu Quanta 200 3D

Figure 1. The chamber interior of the dual-beam microscope (FIB-SEM) Quanta 200 3D

v elektroniki. Med trki ionov s površino nastajajo tudi izbiti (sekundarni) elektroni in ioni. Tako lahko FIB deluje tudi kot mikroskop. Pri tem moramo upoštevati, da se pri opazovanju površina spreminja zaradi sočasnega izbijanja površinskih atomov z ioni [4].

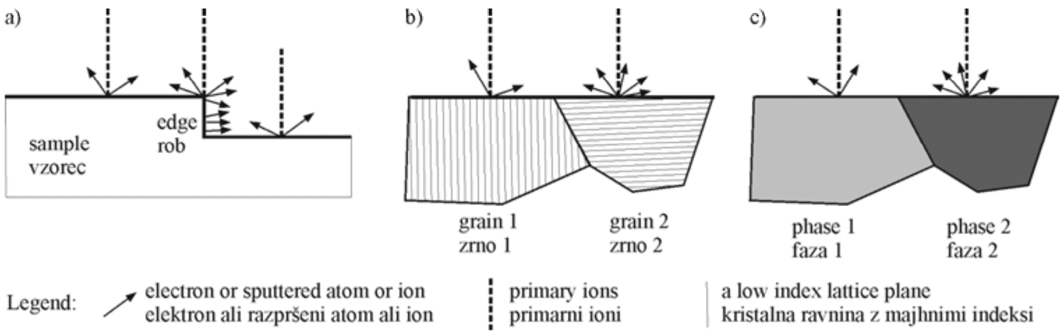
Nastanek slike omogočajo topografski, orientacijski in fazni kontrast. Dobimo lahko informacijo o hrapavosti površine (topografski kontrast), o orientaciji kristalnih zrn (orientacijski kontrast) in fazni sestavi (fazni kontrast), kar je shematično prikazano na sliki 2.

Fazni kontrast omogoča, da razlikujemo različne faze v zlitinah. Ker je hitrost odstranjevanja atomov z galijevimi atomi zelo majhna, je smiselno ugotavljati faze le v manjših območjih [5]. To so npr. meddendritna območja v počasi ohlajenih vzorcih. Po drugi strani se lahko uporabi za popolno karakterizacijo hitrostrjenih prahov ali trakov. Odstranjevanje snovi omogoča tudi pripravo prečnih prerezov, s katerimi odkrijemo mikrostrukturo na površini, ki je pravokotna na spolirano površino. Ta, tako imenovana 3D-mikroskopija, lahko odkrije

Therefore, it is instrumental in electronics. During collisions of ions with a surface, also secondary electrons and secondary ions can be formed. Thus, FIB can also work as a microscope. However, it has to be taken into account that during the microscopic investigation the surface of a sample is being changed simultaneously [4].

Imaging is possible due to the topographic, orientation and phase contrasts. One can obtain information regarding the surface roughness (topographic contrast), different orientation of crystal grains (orientation contrast), and the phase composition (phase contrast), which is schematically shown in Fig. 2.

A phase contrast enables distinctions between different phases in alloys. Since the removal rate by the use of gallium ions is rather low, it is worth to use for determination of phases in smaller regions [5]. Thus, it could be use for the determination of phases in the interdendritic regions of slowly cooling specimens. On the other hand, it can be used for the complete characterization of rapidly solidified powders or melt-spun ribbons. The removal of substance can also



Slika 2. Shematična predstavitev interakcije ionskega snopa s površino vzorca in nastanka različnih vrst kontrasta: a) topografski kontrast, b) orientacijski kontrast in c) fazni kontrast.

Figure 2. A schematic presentation of interactions of an ion beam with the surface of a specimen, and the formation of various types of contrast: a) topographic contrast, b) orientation contrast, and c) phase contrast

prostorsko razporeditev faz. S kombinacijo številnih zaporednih prečnih prerezov lahko naredimo prostorsko rekonstrukcijo faz, ki omogoča opredelitev njihove 3D-oblike [6].

V tem prispevku bomo prikazali uporabo metode FIB pri opredelitvi faz v nekaterih aluminijevih livnih zlitinah. Glavni cilj je prikazati uporabo FIB v metalografiji.

2 Eksperimentalno delo

Raziskali smo več aluminijevih zlitin. Predstavljamo rezultate zlitine A383 in poskusne zlitine Al-Mn-Be (preglednica 1). Zlitine so bile ulite v bakreno kokilo.

Zlitini smo preiskali v mikroskopu z dvojnim curkom Quanta 200 3D, ki uporablja galijev izvor ionov. Majhen tok (okoli 10 pA) smo uporabili za ionsko mikroskopijo in jedkanje površine. Tokove od 3 do 20 nA smo uporabili za grobo, tokove 0,5-1 nA pa za srednje grobo odvzemanje. Za poliranje površine so bili primerni tokovi od 0,1 do 0,5 nA. Tok 0,5 nA je bil uporabljen za nanašanje platine. Za opazovanje mikrostrukture smo uporabili sekundarne (SE) in izbite (BSE) elektrone, prav tako pa tudi sekundarne elektrone, ki so jih izbili ioni (II-SE).

3 Rezultati in diskusija

Pri klasični metalografiji pripravljamo vzorce z mehanskim brušenjem in poliranjem, čemur navadno sledi kemijsko jedkanje. S temi koraki odkrijemo mikrostrukturo

allow preparation of cross-sections, allowing the revealing of microstructure on a surface perpendicular to the polished surface. This, so-called, 3D-microscopy can reveal a 3D phase distribution. By a combination of many sequential cross-sections, a 3D-reconstruction of phases is possible, which allows determination of their exact 3D-shape [6].

In this article we used FIB for determination of phases in some Al-casting alloy. The main aim was to show the performance of FIB in metallography.

2 Experimental

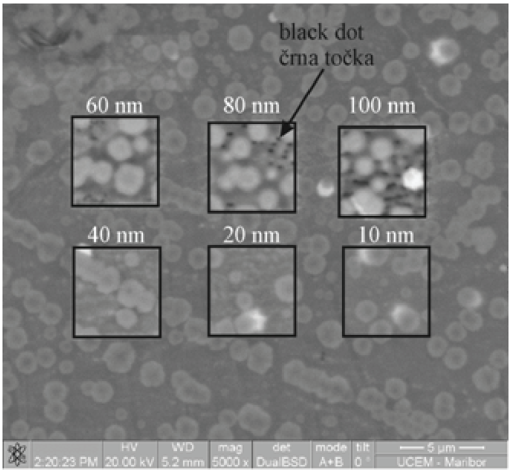
Several Al-alloys were investigated. In this article, we will present some results of the alloy A356 + Sc, A383 and an experimental Al-Mn-Be alloy (Table 1). The alloys were cast into a copper mould.

The alloys were investigated using a dual beam (ion and electron) scanning microscope Quanta 200 3D, which uses gallium ion source. A low ion current was used (usually 10 pA) for the ion microscopy and surface etching, 3-20 nA for the rough milling, 0.5-1 nA for the middle milling and 0.1-0.5 nA for the final polishing of cross-sections. The 0.5 nA current was used for the platinum deposition. The imaging was carried out using secondary (SE) and backscattered (BSE) electrons, as well as ion induced secondary electrons (II-SE).

Preglednica 1. Kemijska sestava raziskanih zlitin

Table 1. The chemical compositions of the investigated alloys

Alloy/Elements	Mg	Mn	Cu	Ti	Fe	Si	Sc	Be	Al
A383	0.35	0.24	2.61	0.04	0.69	10.72	-	-	Rest
Al-Mn-Be	-	11.18	-	-	-	-	-	1.90	Rest



Slika 3. Vpliv globine ionskega jedkanja za videz mikrostrukture zlitine Al-Mn-Be (slika z odbitimi elektroni)

Fig. 3. Effect of the ion etching depth on microstructure appearance of the alloy Al-Mn-Be (backscattered electron image, BSE)

vzorca, ki jo lahko opazujemo s svetlobnim mikroskopom. Ker sta ločljivost in globinska ostrina svetlobnega mikroskopa zelo omejeni, danes vzorce večinoma raziskujemo v vrstičnem elektronskem mikroskopu, ki ima ločljivost okoli 1 nm in odlično globinsko ostrino. Pri opazovanju z odbitimi elektroni se lahko pogosto izognemo jedkanju vzorca, torej lahko razločimo mikrostrukturo na spolirani površini. Za končno poliranje lahko uporabimo ¼ µm diamantno pasto ali suspenzijo SiO_2. Elektronski curek, ki ga uporabljamo za opazovanje, praktično ne poškoduje površine kovinskega vzorca. Po drugi strani ionski curek vselej odstranjuje atome s površine. Torej se površina nenehno spreminja. Nadzorovano odstranjevanje površinskih atomov lahko poveča kontrast med mikrostrukturnimi sestavinami, če mikrostrukturo upodobimo

3 Results and discussion

In the classical metallography, metallic samples are prepared by mechanical grinding and polishing, followed by chemical etching. These steps reveal the microstructure of the samples that can be observed by a light microscope.

Since the resolution and depth of field of light microscopes are limited, metallic samples are nowadays mainly investigated by scanning electron microscopes that have a resolution around 1 nm, and excellent depth of field. By imaging using backscattered electrons, one can even avoid sample etching, and can observe the samples in the as-polished condition. The final polishing can comprise polishing using a ¼ µm diamond paste or a silica suspension. An electron beam can hardly damage any metallic sample. On the other hand, an ion beam always causes scattering of atoms from the surface. Thus, the surface is being constantly modified. The controlled removal of the surface atoms can increase a contrast between the microstructural constituents when the microstructure is imaged by ions or electrons. This process is called ion etching.

Ion etching

Fig. 3 shows the microstructure of an Al-Mn-Be sample that was cast into a permanent mould. The microstructure outside the boxes corresponds to the initial as-polished conditions. The backscattered electron image reveals the α-Al matrix (Al-rich solid solution), with the quasicrystalline (slightly darker particles) and intermetallic compound $Al_{15}Mn_3Be_2$ particles (brighter particles). The numbers above the boxes indicate the thicknesses of the layers that were removed from the surface by the ion beam.

z ioni ali elektroni. Ta proces imenujemo ionsko jedkanje.

Ionsko jedkanje

Slika 3 prikazuje mikrostrukturo zlitine Al-Mn-Be, ki je bila ulita v bakreno kokilo. Mikrostruktura izven kvadratov ustreza začetnemu stanju po mehanskem poliranju. Mikroposnetek z odbitimi elektroni odkrije aluminijevo osnovo α-Al (aluminijeva trdna raztopina) s kvazikristalnimi delci (nekoliko temnejši delci) in delci intermetalne spojine $Al_{15}Mn_3Be_2$ (svetlejši delci). Številke nad kvadrati označujejo debelino plasti, ki je bila odstranjena s površine z ioni. Razvidno je, da se z večjo debelino odstranjene plasti poveča kontrast med mikrostrukturnimi sestavinami. Različne faze imajo različne razpršitvene koeficiente, zato se debeline odstranjenih plasti razlikujejo med fazami. Zato ionsko jedkanje poveča površinsko hrapavost, kar poveča topografski kontrast. Toda po odstranitvi več kot 60 nm snovi, se začnejo pojavljati črne točke, delci pa postajajo bolj zaobljeni. Ta primer kaže, da obstaja optimalna debelina odstranjene snovi. Ko je debelina večja, se začnejo pojavljati različni artefakti, prav tako pa se večajo poškodbe mikrostrukturnih sestavin. Znano je tudi, da se ob obstreljevanju galijevi atomi vgradijo v površino. To spremeni kemično sestavo površine in lahko vodi tudi do nastanka galijevih faz, ki povsem spremenijo mikrostrukturo.

Z ionskim jedkanjem lahko kontrastiramo površine, ki jih nismo metalografsko pripravili. To so npr. površine trakov, ki smo jih hitro strdili.

Z ionskim jedkanjem lahko povečamo kontrast, vendar dobimo še vedno samo 2D-sliko, podobno kot jo dobimo tudi pri svetlobni mikroskopiji ali vrstični elektronski mikroskopiji.

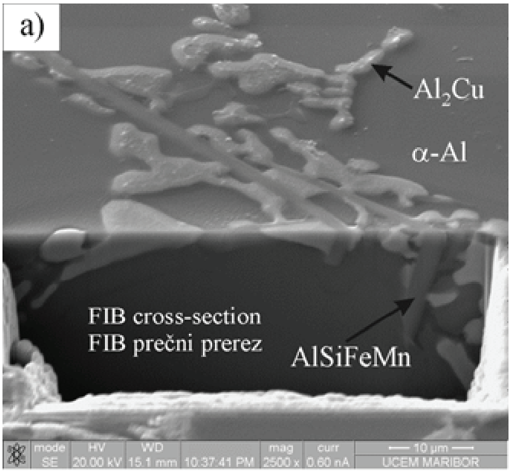
It is evident that by the increasing thickness of the removed layers, the contrast between microstructural constituents increases. Different phases possess different scattering factors, thus the thickness of the removed layer slightly differs between phases. Thus, the ion etching increases the surface roughness, which contributes to the topographic contrast. However, after removing of 60 nm and more substance, dark spots appear and particles become slightly more rounded. This example clearly shows that there exists an optimal thickness of the removed substance. When this thickness is exceeded, several artefacts can appear, and microstructural constituents become damaged. It is known, that incoming gallium atoms become implanted into the surface, which modifies the surface's chemical composition, can, in extreme cases, leads to the formation of Ga-rich compounds, which can totally change the microstructure.

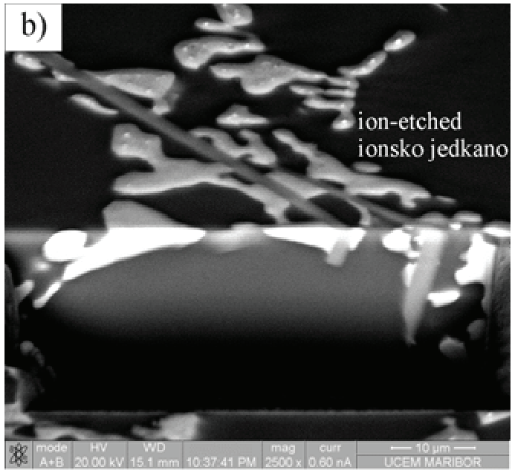
In addition to polished surfaces, the ion etching can be used for contrasting of the surfaces that was not prepared by metallography, e.g. the outer surfaces of casting or melt-spun ribbons.

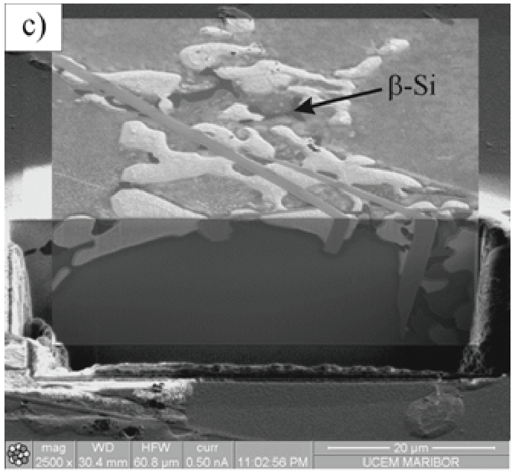
By ion etching, we can obtain an image with enhanced contrast, but it is still a 2D-image, similar as can be obtained by light and scanning electron microscopy.

3D-microscopy

With the ions, it is possible to remove material at selected places; the process is called ion milling. At this stage, higher ion current is applied, thus, all sides of the hole are rough. After that, one surface that is perpendicular to the initial mechanically polished surface is - smoothed by ion polishing. This surface is indicated by "FIB cross section" in Fig. 4a, and the initial mechanically surface is denoted by "ion etched" in Fig. 4b. This, so-







Slika 4. Prečni prerez narejen z ionskim snopom v meddendritnem območju zlitine A383, narejen s FIB: a) slika z izbitimi (sekundarnimi) elektroni, b) slika z odbitimi elektroni and c) slika z elektroni, ki so jih izbili ioni.

Figure 4. FIB cross-section of an interdendritic region in the alloy A383: a) SE-image, b) BSE-image and c) II-SE-image.

3D-mikroskopija

Z ioni lahko odstranimo snov na izbranih mestih. Proces imenujemo ionsko odvzemanje snovi. Pri tem uporabimo večje ionske tokove, zato so stranice nastale luknje precej hrapave. Zatem zgladimo eno od stranic luknje, ki je pravokotna na mehansko spolirano površino. Ta ploskev je označena kot "FIB prečni prerez" na sliki 4a, medtem ko je mehansko polirana površina označena z "ionsko jedkano" na sliki 4b. To je t.i. 3D-mikroskopija, ki odkrije 3D-obliko faz ter njihovo prostorsko razporeditev.

Slika 4 prikazuje isti prečni prerez s sekundarnimi elektroni (slika 4a), odbitimi elektroni (slika 4b) in s sekundarnimi elektroni, ki so jih izbili ioni (slika 4c). Vsaka slika nudi drugačno informacijo o

called, 3D-microscopy reveals 3D-shapes of phases and also indicates a spatial phase distribution.

Fig. 4 shows the same cross-section imaged by secondary (Fig. 4a) and backscattered (Fig. 4b) electrons, and by secondary electrons induced by ions (Fig. 4c). Each image carries different information. The silicon phase (β-Si) is hardly seen in SE- and BSE-images, while it is easily recognized in II-SE-image.

mikrostrukturi. Silicijeva faza (β-Si) se komaj opazi na slikah s sekundarnimi in odbitimi elektroni, medtem ko je izrazito vidna na sliki s sekundarnimi elektroni, ki so jih izbili ioni (II-SE).

3D-tomografija

Kadar združimo več zaporednih prečnih prerezov, dobimo 3D-rekonstrukcijo faz, kar omogoča natančno opredelitev njihove prostorske oblike. To je bilo narejeno že za več aluminijevih livnih zlitin [6]. Danes se 3D-tomografija pogosto kombinira z EDS (energijskodisperzijsko spektroskopijo), da dobimo prostorsko razporeditev elementov [8] in z EBSD (difrakcija odbitih elektronov), pri čemer dobimo prostorsko orientiranost faz [7].

4 Zaključki

Fokusiran ionski snop (FIB) je naprava, ki uporablja ionsko snop za odstranjevanje snovi s površine vzorcev, in tudi za mikroskopiranje. Nadzorovano odstranjevanje snovi s površine lahko povzroči ionsko jedkanje, ki poveča kontrast med mikrostrukturnimi sestavinami. Ionsko jedkanje lahko povzroči lastne artefakte, zato je za vsak primer posebej potrebno opredeliti optimalne parametre.

Zaradi sorazmerno počasnega odstranjevanje snovi, lahko odkrijemo mikrostrukturo le v manjših območjih. Uporaba FIB omogoča 3D-mikroskopijo in 3D-tomografijo, ki dodata nove vidike klasični metalografiji, ki temelji na raziskavi 2D-prečnih prerezov. Tako lahko metoda FIB predstavlja pomembno komplementarno tehniko drugi tehnikam karakterizacije.

3D-tomography

By a combination of many sequential cross-sections, a 3D-reconstruction of phases is possible, which allows determination of their exact 3D-shape. This has already been done for some Al-casting alloys, e.g. in Ref. [6]. In addition, 3D tomography is often combined with EDS (Energy Dispersive Spectroscopy) to obtain 3D chemical maps [8], and with EBSD (Electron Back-Scattered Diffration) to obtain 3D orientation maps [7].

4 Conclusions

The focussed ion beam (FIB) is a device, in which an ion beam is used for removing of a material from the surface from samples, as well as from imaging. Controlled removal of the material from the surface can produce ion etching. This increases the contrast between microstructural constituents. However, ion etching can also produce its own artefacts, thus the optimal conditions have to be found.

Due to the relatively small removal rates, the microstructure can be revealed at smaller regions. The application of FIB allows 3D-microscopy and 3D-tomography, which adds additional dimensions to classical metallography, which relied on 2D-cross sections. Thus, FIB can represent an important complementary method to other characterisation techniques.

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