<http://www.diva-portal.org>

Postprint

This is the accepted version of a paper published in *Journal of Alloys and Compounds*. This paper has been peer-reviewed but does not include the final publisher proof-corrections or journal pagination.

Citation for the original published paper (version of record):

Ghassemali, E., Sonkusare, R., Biswas, K., Gurao, N P. (2017)

In-situ study of crack initiation and propagation in a dual phase AlCoCrFeNi high entropy alloy.

Journal of Alloys and Compounds, 710: 539-546

<https://doi.org/10.1016/j.jallcom.2017.03.307>

Access to the published version may require subscription.

N.B. When citing this work, cite the original published paper.

Permanent link to this version:

<http://urn.kb.se/resolve?urn=urn:nbn:se:hj:diva-35281>

In-situ study of crack initiation and propagation in a dual phase AlCoCrFeNi high entropy alloy

Ehsan Ghassemali^{a,*}, Reshma Sonkusare^b, Krishanu Biswas^b, Nilesh P. Gurao^b

^a*School of Engineering, Jönköping University, Box 1026, 551 11 Jönköping, Sweden*

^b*Department of Materials Science and Engineering, Indian Institute of Technology, Kanpur-208016, India*

* Corresponding author: ehsan.ghassemali@ju.se; +46 36 10 1692

Abstract

This study reports the effect of phase distribution on crack propagation in a dual phase AlCoCrFeNi high entropy alloy under tensile loading. The alloy is characterized by the presence of a brittle disordered BCC phase that can be toughened by precipitation of a ductile FCC phase during homogenization heat treatment. The stress and strain partitioning between the two phases is of paramount importance to determine the mechanical response of this alloy. The as-cast alloy was subjected to homogenization at 1273 K for 6 hr to prevent the formation of detrimental sigma phases and to precipitate the ductile FCC phase. In-situ tensile test in a scanning electron microscope with an electron backscatter diffraction facility was carried out to understand the micro-mechanisms of deformation of the alloy. Precipitation of the FCC phase at the BCC grain boundaries reflected the effect of the FCC phase on crack deflection and branching during propagation. The strain partitioning between the two phases and the evolution of misorientation distribution was investigated. It is observed that the presence of ductile FCC high entropy phase can impart good room temperature ductility to the brittle BCC phase. The investigation on the dual phase HEA is very few. Therefore, proper microstructural design can be utilized to toughen the brittle HEAs.

Keywords: Dual phase; HEA; EBSD; Crack propagation; In-situ.

1. Introduction

High entropy alloys (HEAs) is a field of vigorous research activity now-a-days. HEAs are multiprincipal multicomponent alloys having either single or multiple FCC or BCC phases in the microstructure. These alloys normally include super saturated solid solutions yielding high strength, superior thermal stability, outstanding wear and corrosion resistant, etc. [1, 2].

Mechanical properties of these alloys are of paramount interest, because of the fact that deciphering micromechanisms of deformation will shed new light on the deformation of these novel alloys, as well as expand the potential applications. In this connection, lots of efforts have been directed to investigate the deformation behavior of these alloys. However, research works reported in the literature are mostly focused on single phase alloys. The rationale behind the single-phase HEA concept was that normally the

second phases are intermetallic particles or other phases that could embrittle the alloy. While this may not be always the case, as in superalloys [3]. Thus, precipitation of secondary phases in the microstructure could improve the properties of such alloys even further and seems to be worth investigating.

Among these novel alloys, the $\text{Al}_x\text{CoCrCuFeNi}$ is one of the most extensively investigated [4-8], which generally exhibit outstanding elevated temperature strength and good wear resistance. Tong et al. [9] reported that changing the Al content alters the fraction of the BCC and FCC solid solution phases.

Although the alloy with sole FCC phase microstructure presents 20-60% ductility, further addition of Al can increase the strength by a factor of 300% (BCC promoter), while the ductility can decrease to less than 5% [10]. Besides, due to segregation of Cu into the interdendritic region or grain boundaries, this alloy seemed to be not much suitable for high temperature applications [11].

As an alternative to overcome such challenges, Cu has been removed from the alloy composition. This still keeps the alloy, being a 5-component system, within the category of high entropy alloys ($\Delta S > 1.5R$).

Like the previously mentioned alloy, by altering the atomic ratio of Al in the $\text{Al}_x\text{CoCrFeNi}$ system, the fractional phase distribution can be altered [12], which in return is expected to affect the mechanical properties of the alloy as presented in Table 1.

Table 1. The effect of Al content on the phase fraction and mechanical properties (compression test) of $\text{Al}_x\text{CoCrFeNi}$ [13-16].

x (Al atomic fraction)	0-0.4	0.3-0.9	0.9-2
As-cast phase/s	FCC	BCC + FCC	BCC
Hardness (GPa)	1-2	3-5	5-6
Yield strength (GPa)	-	-	1.3-1.5
Uniform ductility (%)	-	25-40	10-24

Most of studies on the mechanical behavior of HEAs have been done using compression test. This is because compared to tensile test, the compression test is less sensitive to brittleness of the material and/or voids and porosities in the microstructure. In tension, the propagating crack tends to orientate perpendicularly to the loading direction; whereas in compression, the identical crack tends to align parallel to the applied compression direction [17]. It is proven that the stress require for crack propagation in compression test is inversely related to the Poisson's ratio of the material ($\sigma_{\text{Compression}} = \frac{\sigma_{\text{Tensile}}}{\text{Poisson's ratio}}$) [18]. In isotropic materials the Poisson's ratio is equal to 0.3; thus in isotropic materials, the stress required to break the specimen in compression test can be up to 3 times higher than that in tension.

As a continuation of the work by Wang et al. [19], who studied the compressive strength of the as-cast AlCoCrFeNi system, Munitz et al. [15] altered the fraction of the phases by heat treatment, and studied its effect on the mechanical behavior of the alloy under compressive loading. No phase transformation was reported by heating up to 923 K. Traces of the FCC phase as well as a brittle tetragonal σ phase started to appear after heat treatment above 1123 K. The detrimental σ phase can be avoided after heating above 1248 K. Thus, by heat treatment above 1248 K, the microstructure is composed of mainly BCC phase

with traces of ordered B2 phase and FCC phase. The fraction of the FCC phase can be decreased by heat treatment at higher temperatures. These results were in consistent with the report by Mohanty et al. [20]. After the compression test, Munitz et al. [15] found out that cracks mostly initiated and propagated in the inter-dendritic region, which was enriched in Co, Cr and Fe. It was also reported that cracks terminated upon reaching to the relatively ductile dendritic region.

In another study, Tang et al. [21] investigated the effect of microstructural features on tensile behavior of AlCoCrFeNi manufactured under different processing routes. It was reported that due to brittleness of the alloy in the as-cast condition, most of cracks initiated at grain boundaries and triple-junctions, which would lead to intergranular fracture. Although, the tensile properties of the AlCoCrFeNi alloy was studied in the mentioned report, the fracture behavior of the dual HEA, especially in-situ EBSD of the fractured region seems intriguing to investigate, which can provide more details on the fracture behavior of such alloys under tensile loading. To further develop such an interesting study, it seems rather important to investigate the effect of various phases and their orientation on the tensile properties of the alloy.

The aim of the present study is to analyze the influence of individual BCC and FCC phases on the crack initiation and propagation in the heat treated AlCoCrFeNi alloy. In-situ electron backscattered diffraction (EBSD) under tensile loading was employed to evaluate the effect of phase distribution on crack initiation and propagation.

2. Materials and methods

2.1. Arc melting

The AlCoCrFeNi equiatomic high entropy alloy was prepared by vacuum arc melting and subsequent suction casting process. Inert atmosphere was maintained using argon gas, during both the processes. Water-cooled copper mould was used with non-consumable tungsten electrode for melting and casting. High purity individual elemental pieces with purities more than 99.95% (Alfa Aesar, USA) were used to prepare the alloy buttons, which were melted for at least 5 times in two different directions to ensure the chemical homogeneity of the alloy. The buttons were subsequently suction-casted, in vacuum arc cum suction casting facility, to form a rod with square cross-section, having dimensions of 55 x 10 x 10 mm. The actual composition of the alloy in the as-cast state was measured using the Energy Dispersive Spectrometry (EDS), which fell within the equiatomic proportion of the elements (± 1.5 at.%).

2.2. Heat treatment

The as-cast rod was subsequently heat treated at 1273 K for 6 hr and then quenched in water to homogenize the microstructure. According to previous studies [15], this should lead to a microstructure composed of mainly BCC phase with precipitates of the FCC phase, without the detrimental σ phase. Argon was used for protection during heat treatment minimizing the possible oxidization on the sample surface.

2.3. Microstructural analysis and in-situ test

After heat treatment, the rod was cut by the waterjet method into bone-shape samples as schematically shown in Fig. 1a and then put into the tilted stage as shown in Fig. 1b.

Next, the samples were mounted and abraded by SiC papers up to 4000 grit. The preparation was followed by diamond polishing down to 0.1 μm particles and subsequent polishing with colloidal silica solution (OPS + 10% H_2O_2 (30%)). Samples were then taken out of the mount by careful cutting and breakage of the epoxy. The microstructure was analyzed using a high-resolution scanning electron microscope (LYRA3, TESCAN) equipped with EDS and EBSD detectors (EDAX). The EDS analysis was carried out with ZAF correction to minimize possible errors (accuracy of around 0.4 at.%). The EBSD and EDS data was collected using 20 kV accelerating voltage and 7 nA electron beam. Using the TESCAN electron column design, such a condition can provide a spot size of around 70 nm at working distance of 18 mm. The step size of 0.5 μm was chosen to balance the data accuracy and collection time for each EBSD mapping. The successful EBSD indexing rate was higher than 85% for all runs.

Figure 1. a) Schematic of the in-situ tensile sample and b) the in-situ tensile stage.

The in-situ tensile test was carried out at the pulling rate of $3 \mu\text{m.s}^{-1}$ using a Kammrath and Weiss instrument (Max. load: 5kN) in a pre-tilted mode suitable for EBSD data collection. The loadcell was aligned before the test using a stiff sample to ensure uni-axial loading conditions. The elastic deformation of the in-situ stage was also calibrated using an extensometer. This data was used to correct the obtained load-displacement curve. The tensile test was interrupted in different steps for EBSD data collection.

3. Results and discussion

3.1. Heat treated microstructure prior to deformation

EDS mapping of a random area in the heat treated sample showed a reasonable elemental distribution (Fig. 2). A slight phase separation was seen, specifically in the EDS map of Al and Fe. This is due to the presence of two phases under the heat treated conditions, which actually show slightly different chemical

composition as reported before [22]; specifically, Al (and a maybe Ni) is a BCC promoter, Cr and Fe are FCC promoter and Co and Ni are relatively evenly distributed in both phases, as represented in Table 2, including the chemical analysis of both phases.

Table 2. Chemical composition (wt.%) of the constituent phases in the heat-treated alloy.

	Al	Co	Cr	Fe	Ni
FCC	12.3 ± 2.5	20.3 ± 1.6	28.4 ± 1.8	22.3 ± 2.1	19.7 ± 1.7
BCC	26.5 ± 0.9	20.5 ± 3.0	23.5 ± 1.9	18.8 ± 2.0	24.6 ± 1.5

EBSD analysis revealed an interesting distribution of the FCC phase within the microstructure including particle-shape precipitates inside the BCC grains and mostly continuous FCC precipitates along the BCC grain boundaries (Fig. 3). The BCC phase showed a narrow Gaussian distribution of grain size (average $52 \pm 4 \mu\text{m}$, measured from 5 different regions using the ASTM E112-13 standard). As also examined in larger EBSD phase maps, the area fraction of the FCC phase was around 21%. The study by Munitz et al. [15], showed that the FCC area fraction is the maximum that can be achieved in this composition after a heat treatment above 1248 K. Figure 3 shows the detailed microstructural characterization of the heat treated equiatomic AlCoCrFeNi alloy revealing precipitate of FCC phase in the BCC grains. The precipitates are found to be distributed uniformly within the grains of BCC phase. The microstructure consists of precipitates in BCC grains, typically modulated basket-weave morphology. The pole figures (PFs) of the individual phases illustrated a random orientation distribution of both phases after the heat treatment.

Figure 2. Random scanning electron microscope (SEM) micrograph as well as EDS elemental mapping of the heat treated AlCoCrFeNi alloy.

Figure 3. a) SEM micrograph; b) inverse pole figure (IPF) map; c) phase map of a random area on the heat treated equiatomic AlCoCrFeNi alloy.

3.2. During in-situ tensile

The sample is found to undergo abrupt fracture. Several attempts were made to study the crack initiation and propagation. We shall report here the salient results. The EBSD maps around the fractured area illustrated valuable information about the crack initiation and propagation. As highlighted by circles in Fig. 4, most of cracks have initiated at the triple junction of the BCC grains. It is worth noting that some of the triple junctions (in 2 dimensions) could be the junction point of more than 3 BCC grains in 3-dimensions (3D - subsurface grains), which makes them the candidate region as the weak points for crack initiation. This can be further investigated using 3D tomography. Tang et al. [21] observed the same trend and suggested the same reason as mentioned here. They also suggested formation of voids at grain boundaries perpendicular to the principle stress to be another possible reason for crack initiation in such a manner.

As illustrated by arrows in Fig. 4, the cracks rather rapidly propagated throughout the BCC grain. All cracks propagated trans-granularly (Fig. 4c), which shows the strengthening effect of the FCC precipitates at the BCC grain boundaries. The FCC phase located at the grain boundary acts as an obstacle for the crack propagation. As a result, most of cracks either branched into new cracks, redirected or even stopped at the BCC grain boundaries (See Fig. 4&5). This fact is also schematically shown in Fig. 5d.

Munitz et al. [15] reported that in such an alloy, heat treated below 1273 K, the cracks propagated in the interdendritic area, that contained the FCC and the σ phase. They reported that cracks mostly terminated at the dendritic core regions, where the BCC matrix is the major phase. It was remarked that heat treatment above 1248 K would improve the mechanical properties, but no evidences was shown in the mentioned study.

It is worth mentioning that no particular texture evolution was observed during deformation. This could be due to the dominant brittle behavior of the alloy at room temperature that restricts the amount of lattice rotation.

Figure 4. a) SEM micrograph and b) the IPF map (over imposed to the image quality map) of the same region near to the fractured area after the in-situ test. The circles show the possible regions for crack initiation and arrows illustrate the effect of the BCC grain boundaries (FCC phase) on deflection and/or stopping crack propagation. The micrograph c) illustrates a low magnification EBSD map of a relatively long transgranular crack propagation.

Figure 5. a) SEM micrograph, b) IPF map, c) phase map of a region near the fractured area after the in-situ test. The circles show the locations where the crack has been redirected or branched (BCC grain boundaries, where the continuous FCC phase was located). d) schematic of the crack initiation and propagation in the dual phase (BCC+FCC) HEA.

The histogram of the Kernel Average Misorientation (KAM) as a function of stress are shown for both phases in Fig. 6. KAM is used as a qualitative measure of local orientation changes, that is related to geometrically necessary dislocations (GNDs) [23]. Especially for the BCC phase, the KAM distribution shifted to higher values by increasing the deformation. This indicates the raise in the density of GNDs and accumulation of plastic strain in the BCC phase. Due to higher mobility of dislocations in the relatively more deformable FCC phase, less plastic strain accumulation was observed for this phase, as depicted in Fig. 6b. It is to be mentioned here that the BCC phase is harder than the FCC phase and it is expected that the harder phase should bear more stress and the softer phase should carry more strain. Nevertheless, phase contiguity seems to play an important role from the KAM distribution as the contiguous hard BCC phase shows higher KAM indication higher dislocation density and hence more microscopic strain than the non-contiguous soft FCC phase. It is worth noting that, as shown in Fig. 4 and 5, mostly the continuous FCC precipitates at the BCC grains boundaries are those delaying the crack propagation. Consequently, increasing the amount of continuous FCC precipitates at the grain BCC boundaries (by altering the Al content in the composition [14] and heat treatment [15]) may result in more ductility without much reduction in the strength of the alloy. This fact requires further investigations.

Figure 6. KAM profile of the a) BCC and b) FCC phases as a function of stress during in-situ tensile deformation.

As depicted in Fig. 7, the misorientation profile for the FCC phase shows a relatively large peak at around 60°. This peak is attributed to annealing twins that could occur in the FCC phase. After deformation at room temperature, there is only slip dominated deformation in the alloy. The decrease in twin boundary fraction is due to the interaction of dislocations with the twin boundary, which contributes to change in character of the twin boundary. The absence of deformation twinning in low stacking fault energy (SFE) FCC phase is attributed to the smaller grain size of the FCC phase in the alloy. There seems not to be any deformation twinning mechanism activated for this alloy, since the peak height decreased by deformation. The misorientation distribution of BCC phase shows increase in fraction of low angle grain boundaries with stress similar to the FCC phase indicating that the BCC phase also undergoes plastic deformation by slip.

Figure 7. Misorientation profile of a) BCC phase, b) FCC phase.

3.3. After fracture

As depicted in Fig. 8, the tensile sample fractured at 935 MPa and around 1% total strain (Elastic (Young's) modulus of 162.5 GPa), which can be considered as a brittle fracture.

Figure 8. Engineering stress-strain curve obtained from the in-situ tensile test of the alloy. The intermittent points are when the test was paused to look at the microstructure.

The fractography examination showed a typical brittle fracture surface, as depicted in Fig. 9 a-c, which was expected from a material containing around 80% brittle BCC phase.

Only a couple of relatively small porosities were seen at the fracture surface, which is represented in Fig. 9d. The phase separation is clearly shown in Fig. 9e. This can imply the fact that the FCC phase has been precipitated separately as a post-solidification phenomenon during heat treatment.

Figure 9. Fracture surface of the heat treated AlCoCrFeNi alloy after in-situ tensile test; a) brittle fracture surface with river marks; b) brittle fracture surface in higher magnification, showing the underneath phase distribution; c) high magnification micrograph of the fracture surface showing the cleavage planes; d) a porosity seen at the fracture surface; e) EDS map of the porosity.

4. Conclusions

The influence of secondary phase precipitation on the crack initiation and propagation behavior of the AlCoCrFeNi alloy was investigated using in-situ tensile test at room temperature inside a high-resolution SEM. The following results were obtained:

- After heat treatment of the AlCoCrFeNi alloy at 1273 K and subsequent quenching, a dual phase microstructure consisting around 79% BCC (Al and Ni rich) and 21% FCC (Cr and Fe rich) phase was obtained. The FCC precipitates are mostly located at the BCC grain boundaries. Co seemed to be distributed relatively evenly between the two phases.
- Crack propagation mode in the heat-treated alloy was transgranular, which showed the toughening impact of the FCC phase at the grain boundaries. Such a phase distribution worked as a 3D net of obstacles for crack propagation, leading to crack stop or redirection or branching at the BCC grain boundaries.

- Evolution of the misorientation profile during tensile deformation showed an accumulation of plastic strain in the BCC phase; whereas in the FCC phase, due to noncontiguous nature the evolution of the density of GNDs was marginal.

Crack initiation sites could be further analyzed using 3D microscopy at the BCC triple junction boundaries. Besides, there was a very marginal increase in the fraction of the FCC phase in the field of view during deformation. Such a phenomenon could be activated at elevated temperature, which is under investigation. In addition, to quantify the effect of phase fractions on the cracking behavior of the alloy, it is worth investigating the other end of the phase fractions in the microstructure of similar alloy with different amount of Al, consisting a major FCC phase and a minor BCC phase.

References

- [1] B.S. Murty, J.W. Yeh, S. Ranganathan, High Entropy Alloys, Elsevier, UK, 2014.
- [2] A. Takeuchi, K. Amiya, T. Wada, K. Yubuta, W. Zhang, A. Makino, Entropies in Alloy Design for High-Entropy and Bulk Glassy Alloys, *Entropy*, 15 (2013) 3810-3821.
- [3] D.B. Miracle, O.N. Senkov, A critical review of high entropy alloys and related concepts, *Acta Materialia*, 122 (2017) 448-511.
- [4] S. Singh, N. Wanderka, B.S. Murty, U. Glatzel, J. Banhart, Decomposition in multi-component AlCoCrCuFeNi high-entropy alloy, *Acta Materialia*, 59 (2011) 182-190.
- [5] H. Zhang, Y.Z. He, Y. Pan, S. Guo, Thermally stable laser cladded CoCrCuFeNi high-entropy alloy coating with low stacking fault energy, *Journal of Alloys and Compounds*, 600 (2014) 210-214.
- [6] Z. Tang, T. Yuan, C.W. Tsai, J.W. Yeh, C.D. Lundin, P.K. Liaw, Fatigue behavior of a wrought Al_{0.5}CoCrCuFeNi two-phase high-entropy alloy, *Acta Materialia*, 99 (2015) 247-258.
- [7] X.D. Xu, P. Liu, S. Guo, A. Hirata, T. Fujita, T.G. Nieh, C.T. Liu, M.W. Chen, Nanoscale phase separation in a fcc-based CoCrCuFeNiAl_{0.5} high-entropy alloy, *Acta Materialia*, 84 (2015) 145-152.
- [8] G. Li, D. Xiao, P. Yu, L. Zhang, P.K. Liaw, Y. Li, R. Liu, Equation of State of an AlCoCrCuFeNi High-Entropy Alloy, *Jom*, 67 (2015) 2310-2313.
- [9] C.J. Tong, M.R. Chen, J.W. Yeh, S.J. Lin, S.K. Chen, T.T. Shun, S.Y. Chang, Mechanical performance of the Al_xCoCrCuFeNi high-entropy alloy system with multiprincipal elements, *Metallurgical and Materials Transactions A*, 36 (2005) 9.
- [10] C.W. Tsai, M.H. Tsai, J.W. Yeh, C.C. Yang, Effect of temperature on mechanical properties of Al_{0.5}CoCrCuFeNi wrought alloy, *Journal of Alloys and Compounds*, 490 (2010) 160-165.
- [11] M.H. Tsai, J.W. Yeh, High-Entropy Alloys: A Critical Review, *Materials Research Letters*, 2 (2014) 107-123.
- [12] W.R. Wang, W.L. Wang, J.W. Yeh, Phases, microstructure and mechanical properties of Al_xCoCrFeNi high-entropy alloys at elevated temperatures, *Journal of Alloys and Compounds*, 589 (2014) 143-152.
- [13] S.G. Ma, Y. Zhang, Effect of Nb addition on the microstructure and properties of AlCoCrFeNi high-entropy alloy, *Materials Science and Engineering: A*, 532 (2012) 480-486.
- [14] C. Li, J.C. Li, M. Zhao, Q. Jiang, Effect of aluminum contents on microstructure and properties of Al_xCoCrFeNi alloys, *Journal of Alloys and Compounds*, 504 (2010) S515-S518.
- [15] A. Munitz, S. Salhov, S. Hayun, N. Frage, Heat treatment impacts the micro-structure and mechanical properties of AlCoCrFeNi high entropy alloy, *Journal of Alloys and Compounds*, 683 (2016) 221-230.

- [16] Y. Zhang, T.T. Zuo, Z. Tang, M.C. Gao, K.A. Dahmen, P.K. Liaw, Z.P. Lu, Microstructures and properties of high-entropy alloys, *Progress in Materials Science*, 61 (2014) 1-93.
- [17] C. Brooks, A. Choudhury, *Failure Analysis of Engineering Materials*, McGraw-Hill Education, 2002.
- [18] P.N.B. Anongba, J. Bonneville, A. Joulain, Brittle cracks under compression: introducing Poisson effect, *Revue Ivoirienne des Sciences et Technologie*, 17 (2011) 17.
- [19] Y.P. Wang, B.S. Li, M.X. Ren, C. Yang, H.Z. Fu, Microstructure and compressive properties of AlCrFeCoNi high entropy alloy, *Materials Science and Engineering: A*, 491 (2008) 154-158.
- [20] S. Mohanty, T.N. Maity, S. Mukhopadhyay, S. Sarkar, N.P. Gurao, S. Bhowmick, K. Biswas, Powder metallurgical processing of equiatomic AlCoCrFeNi high entropy alloy: Microstructure and mechanical properties, *Materials Science and Engineering: A*, 679 (2017) 299-313.
- [21] Z. Tang, O.N. Senkov, C.M. Parish, C. Zhang, F. Zhang, L.J. Santodonato, G. Wang, G. Zhao, F. Yang, P.K. Liaw, Tensile ductility of an AlCoCrFeNi multi-phase high-entropy alloy through hot isostatic pressing (HIP) and homogenization, *Materials Science and Engineering: A*, 647 (2015) 229-240.
- [22] T. Yang, S. Xia, S. Liu, C. Wang, S. Liu, Y. Zhang, J. Xue, S. Yan, Y. Wang, Effects of AL addition on microstructure and mechanical properties of Al_xCoCrFeNi High-entropy alloy, *Materials Science and Engineering: A*, 648 (2015) 15-22.
- [23] A.J. Schwartz, M. Kumar, B.L. Adams, D.P. Field, *Electron Backscatter Diffraction in Materials Science*, Springer US, 2009.