UDK 620.18:001.891:669.715
Original scientific article/Izvirni znanstveni članek

ISSN 1580-2949
MTAEC9, 55(4)541(2021)

METALLOGRAPHIC METHODS FOR DETERMINING THE QUALITY OF ALUMINIUM ALLOYS

METALOGRAFSKE METODE ZA DOLOČEVANJE KAKOVOSTI ALUMINIJEVIH ZLITIN

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Prejem rokopisa – received: 2021-04-19; sprejem za objavo – accepted for publication: 2021-05-18

doi:10.17222/mit.2021.143

Metallographic investigations are critical in research and materials' quality control. Therefore, these methods are very present in scientific and industrial activities. In particular for quality control, metallographic analyses have an indirect correlation with quality, time and economic aspect in the metallurgical industry. Most of the metallographic methods used for aluminium alloys' quality determination are non-standard and are required by customers. The suitability and effectiveness of these metallographic control methods are best determined by interlaboratory investigations. In this investigation, two metallographic laboratories with seven different operators using five different light microscopes with associated software were involved. The interlaboratory analyses covered a determination of the rim zone, dendritic arm space, intermetallic phases and non-metallic inclusions. From the calculated measurement uncertainty, where in the worst case results differ by 4.7 %, it is clear that all four metallographic methods are suitable for implementation in industrial quality laboratories, when correctly and accurately defined (with easy-to-follow instructions).

Keywords: metallography, non-standard control methods, measurement uncertainty, aluminium alloys

Metalografske preiskave so nepogrešljivo sredstvo za izvajanje raziskovalnih kot tudi kontrolnih analiz. Tako so tovrstne metode močno prisotne pri znanstveno raziskovalnih, kot tudi v industrijskih dejavnostih. Zlasti pri slednjih je izvajanje metalografskih analiz neposredno povezano z delovanjem celotnega metalurškega obrata tako na kakovostnem, časovnem in ekonomskem nivoju. Velika večina metalografskih metod, ki se izvajajo za aluminijeve zlitine je nestandardnih in so pogosto določene s strani naročnika oz. kupca. Primernost in uspešnost tovrstnih preiskav je bila smiselno določena z medlaboratorijskimi preiskavami. Pri tej raziskavi sta sodelovala dva metalografska laboratorija s sedmimi različnimi operaterji, ki so uporabili pet svetlobnih mikroskopov s pripadajočo programsko opremo. Medlaboratorijske analize so zajemale izvajanje meritev obrobne cone, meddendritne razdalje, velikosti intermetalnih faz in nekovinskih vključkov. Iz podatkov izračunane merilne negotovosti, kjer je v najslabšem primeru prišlo do zgolj 4,7 % odstopanja, je razvidno, da so vse štiri izvajane metalografske metode ob predhodno dobro definiranih pogojih in navodilih, primerne in smiselne za izvajanje.

Ključne besede: metalografija, nestandardne kontrolne metode, merilna negotovost, aluminijeve zlitine

1 INTRODUCTION

Metallography is a group of basic investigation methods, primarily for metals and alloys.[1] Besides the different mechanical and chemical properties, the control reports of final or semi-final products must also contain the results of metallographic tests.[2] It is important to emphasize that metallography, before observational techniques such as light microscopy (LM), in the initial stages also covers the preparation of the samples. This usually consists of cutting, mounting, grinding, polishing and etching.[3] The majority of ISO and ASTM standards, related to metallography, are dedicated and written for steels.[4] Therefore, most metallographic laboratories have created their own metallographic techniques for aluminium and aluminium alloys, which are often customized in accordance with the client's or customer's requirements. Non-standard metallographic methods present more challenges in the field of validation, repeatability, deviation and uncertainty of the techniques and/or measurements. A very effective way to analyse and obtain information about certain metallographic methods characteristics are interlaboratory tests.

In general, a direct correlation between the crystal grain size and the mechanical properties (tensile strength, hardness) is the reason that the determination of the crystal grain characteristics (average diameter $\overline{d}$, average grain area $\overline{A}$, grain size number G) in the microstructure are very often the result of scientific research as well as industrial control reports.[5,6] Besides the grain size, as a metallographic characteristic, the rim zone, dendrite arm spacing (DAS), intermetallic phases and non-metallic inclusions are very important. The reverse segregations formed on the rim of the cast semi-products aggravate further mechanical processing.[7,8] That way, the distance from the edge to the interior of the sample, where the segregations end, and the normal microstructure continue, is measured and presented as the rim zone. Specific phenomenon of the non-equilibrium solidification of metals and alloys is the appearance of den-

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drites,[9] which during the recrystallization present the origin for the subsequent formation of crystal grains. The ratio between the length and the number of dendrite arms is one possible type of DAS determination.[10] The same as the grain size, the DAS also has an important correlation with the mechanical properties.[11] The formation of intermetallic phases is essential, especially in the case of industrial alloys' production, where a large number of additional elements are applied.[12] Intermetallic phase analyses have focus on length, type and their frequencies/density in the sample. Intermetallic phases can improve the mechanical properties of the material. However, too long, specific types or too frequently obtained intermetallic phases can increase the notch effect in the matrix,[13] which decreases the homogeneity and consequently impairs the mechanical properties. Like for the intermetallic phases there is also a determination of the non-metallic inclusions, also known as defects.[13,14] They are determined by quantification (number) and measurements of their length. The defects have a negative impact and decrease the material's homogeneity and purity, and consequently decrease the mechanical properties of the material.[15,16] In the control methods for the intermetallic phases and non-metallic inclusions, the critical length needs to be determined. This is changing and it depends on the investigated alloys, the samples' condition and the customers' requirements.

The aim of this study is focused on the suitability, accuracy, reliability and repeatability of four different non-standard metallographic methods for aluminium alloys. For the verification of the rim zone, DAS, intermetallic phases and non-metallic inclusions, different measurement techniques were applied. The verifications were performed thorough the number of operators, using different LMs, investigating the same selected samples, repeating the measurements and statistical calculations. Regarding the results of the minimum and maximum measured differences, the standard deviation, the confidence index and the relative accuracy, all four metallographic methods are suitable to use by following the recommended instructions and guidelines.

2 MATERIALS AND METHODS

2.1 Sample preparation

Fourteen samples were selected in the aluminium industry from different metallurgical steps of production (cast, formed, heat treated). The samples were first cut out from industrial semi-products, followed by standard metallographic sample preparation for aluminium. First, the samples were grinded with a paper roughness of P320. The metallographic sample preparation continued with grinding with lower roughness papers, which was followed by a combination of grinding and polishing with a 9-µm suspension on a cloth. Further, a 3-µm suspension on the cloth was used for next polishing step. The last step of surface preparation for most of the samples was a combination of polishing and etching with an oxide polishing slurry (OPS). The tree samples marked with * in **Table 1** were additionally electrolytic etched with Barker reagent (50 mL HBF_4 + 950 mL H_2O, 90 s, 25 V). For the measurements of the rim zone, DAS and the largest intermetallic phase, three samples for each presented technique, were investigated. One representative (untypical) sample was used for the determination of the number of non-metallic inclusions. Four samples were observed for the measurement of the non-metallic inclusions' size (longest length). In **Table 1** are all the investigated samples for the individually introduced metallographic methods.

Table 1: Sample names and metallographic methods

Sample name	Metallographic methods				
	Rim zone	Dendrite arm spacing	Inter-metallic phases	Non-metallic inclusions	
				Number	Size
RZ 1	X				
RZ 2	X				
RZ 3	X				
DAS 1*		X			
DAS 2*		X			
DAS 3*		X			
IP 1			X		
IP 2			X		
IP 3			X		
NMI N				X	
NMI S 1					X
NMI S 2					X
NMI S 3					X
NMI S 4					X

* etched samples

2.2 Microscopes and software

The metallographic techniques and measurements were performed by seven different operators. They used five different LMs and the associated software. Two used microscopes were from Zeiss, the others were from Olympus, Leica and Nikon. All the used microscopes with the associated software are listed in **Table 2**.

Table 2: The light microscopes and software used for metallographic analyses

Light microscope type	Software version
Zeiss Axio Observer 7	ZEN core v2.4
Zeiss Axio Imager.Z2m	ZEN core v2.7
Leica MEF4 M	Imagic IMS v14q3
Olympus BX61	Stream Motion 2.3.3
Nikon MICROPHOT-FXA	Stream Motion 1.9.1

2.3 Procedures and calculations

The four performed and evaluated metallographic techniques/procedures are not standardised. The operators need to perform the measurements in accordance with specific rules, which are distinguished for the individual methods and techniques.[17,18]

2.3.1 Rim zone

Rim-zone measurements were performed on polished cross-sections of the samples. The whole sample must be evaluated for the rim zone by LM. On areas where a larger rim zone is recognized, it is recommended to take an image. The length of the rim zone is measured on images using the LM software. Attention needs to be paid to shadows, which can unconsciously extend the rim zone. The rim zone length must be measured perpendicular to the surface (edge) of the sample to the point where the segregations appear the deepest in the interior of the sample.

2.3.2 Dendrite arm spacing (DAS)

DAS measurements must be performed on etched samples.[18] For the observation with LM, polarised light or a bright-field stage can be used. For each sample, five micrographs with a minimum of 6 dendrites for the evaluation are recommended. It is necessary to select the area of the sample where the dendrites are easily seen. First, the overall length L of a separate dendrite is measured, and then the number of the dendrite arms n of this dendrite is counted. The *DAS* is finally calculated with the Equation (1):

$$DAS = \frac{L}{n} \quad (1)$$

2.3.3 Intermetallic phases

The same as for the rim-zone measurements, also for the length of intermetallic phases, the sample's surface needs to be polished. The whole sample area must be overviewed by LM. It is recommended that during observation, the numbers of micrographs are taken at the positions with larger intermetallic phases. From among them, the largest intermetallic phase is determined. The intermetallic phase length must be measured precisely from the beginning to the end of one continuous intermetallic phase.[19] The discontinued or joined intermetallic phases must not be taken into account. The control requirements of customers often also define the critical type (different colours) and critical length of intermetallic phases.

2.3.4 Non-metallic inclusions

The metallographic technique for non-metallic inclusions' evaluation contains two methods. The first is focused on the number of non-metallic inclusions and the second on the size (length) measurement of the detected non-metallic inclusions. For both methods the whole sample must be observed. Typical inclusions for aluminium alloys are oxide films, oxides (MgO), titanium borides as grain-refiner nets (AlTi5B1), spinels and salts. If there is a distance between the two non-metallic inclusions of less than 20 µm, that inclusion needs to be treated as one inclusion. In contrast, if the distance between the two inclusions is more than 20 µm, the inclusions need to be treated and measured separately. The rules considering the distance between non-metallic inclusions are mostly defined by the control requirements of the customers. At the same time, the customers define the critical number and critical size (length) of non-metallic inclusions.

2.3.5 Overall evaluation

The evaluation of the results and the final validation of the four performed metallographic methods for aluminium alloys were performed with the calculations of certain statistical quantities. As a result of the comparison between the operator's measurements, the minimum and maximum differences in the results for the measured values were calculated. Furthermore, the standard deviation s of all the results was calculated. With a combination of s, the number of operators n and the factor t, which is in accordance with the number of operator-observed fields, the confidence index in range of 95 % (95 % *CI*) was calculated with the Equation (2)[18]:

$$95\ \%\ CI = \pm \frac{t \cdot s}{\sqrt{n}} \quad (2)$$

The 95 % *CI* was divided with the average value of measurements $\overline{P}p$ to calculate the relative accuracy % *RA*, as presented with the equation[18]:

$$\%\ RA = \frac{95\ \%\ CI}{\overline{P}p} \quad (3)$$

3 RESULTS AND DISCUSSION

3.1 Rim zone

Measurements of the rim zone are very important in a correlation with the selection of further mechanical treatments and the processing of the materials and products.[20,21] The largest value of the rim zone is more important than the average value of the rim zone. With the proper determination of the average value, the rim zone with reverse segregations and possible defects[22] can be mostly mechanically removed (i.e., milled). In our case of three samples, the largest rim-zone values were from 264.0 µm to 386.9 µm. The rim-zone distance is influenced by the chemical composition, material or product condition as well as a suitable sample preparation. Operators must pay extra attention to choosing the end point or the line of the rim zone, which is, in some samples, clearly visible (**Figure 1a**), and in the others (**Figure 1b**), very difficult to identify. In **Figure 2** it is clear that the operators' measurement of the rim zone does not differ greatly for individual samples and that the length of the rim zone has no important impact on the operator's accuracy of the measurements. This is also confirmed with the statistical evaluation (**Table 3**), where it is clear that there are at least two operators who measured and determine the same size of the rim zone. The

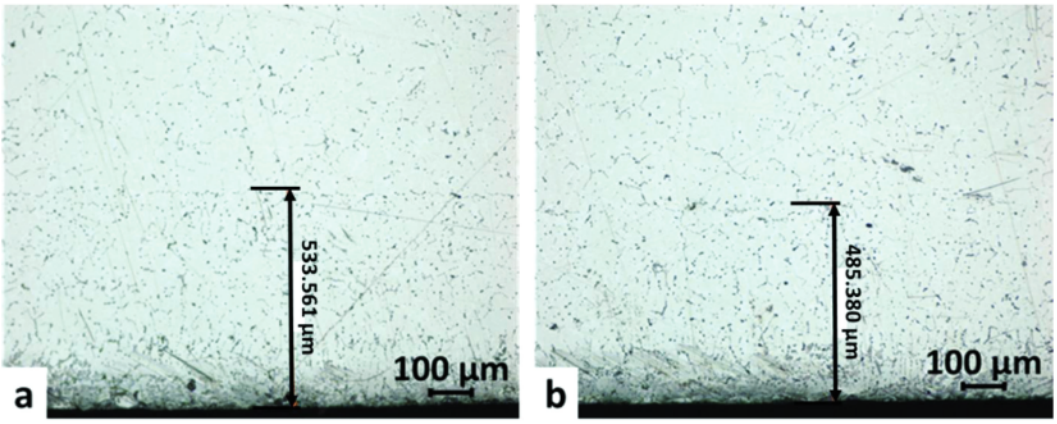

Figure 1: Rim-zone measurements: a) example of clearly visible end of the rim zone; b) example of difficult identification of the rim-zone end

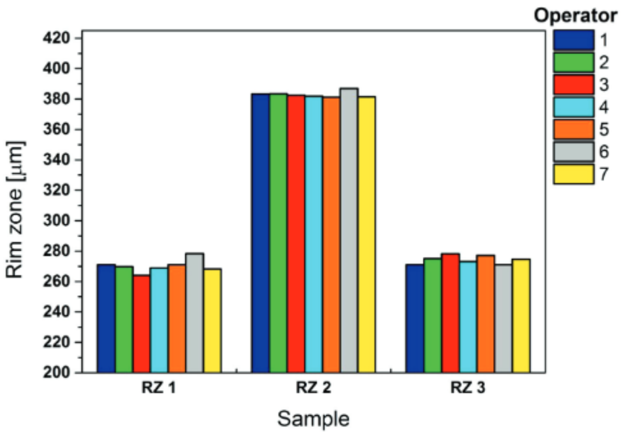

Figure 2: Highest values of different operator's rim-zone measurements

maximum difference between the two operator's measurements is 14.3 µm, but the *% RA* is only 1 %.

3.2 Dendrite arm spacing (DAS)

The importance of DAS measurements is in the connection to the mechanical properties. The same as the crystal grains, the dendrites also have an influence with their homogeneity and average size.[10] The higher the DAS value, the lower are the mechanical properties.[11] Besides the heat treatments, chemical composition and purity of the alloys, the solidification velocity is the most influential parameter for DAS. As presented and confirmed by the results in **Table 3**, DAS can be equivalently measured using polarised light (**Figure 3a**) or in the bright field (**Figure 3b**). With the bright-field method, where the dendrites are not coloured differently, the operators must be especially careful when determining the dendrites' length. The 95 *% CI* for the results of the three samples is 1.7 %, but the *% RA* is 4.7 %. Shown in **Figure 4,** the DAS values are between 29.2 and 38.5 and the operator's measurements range from 0 µm to 5.5 µm.

3.3 Intermetallic phases

Observing the selected three samples (the whole surface area of the sample) the operators found and measured the longest intermetallic phases on four areas for each sample. **Figure 5a** presents the example of different and composed intermetallic phases. The operators need to focus on the continuous, non-interrupted intermetallic

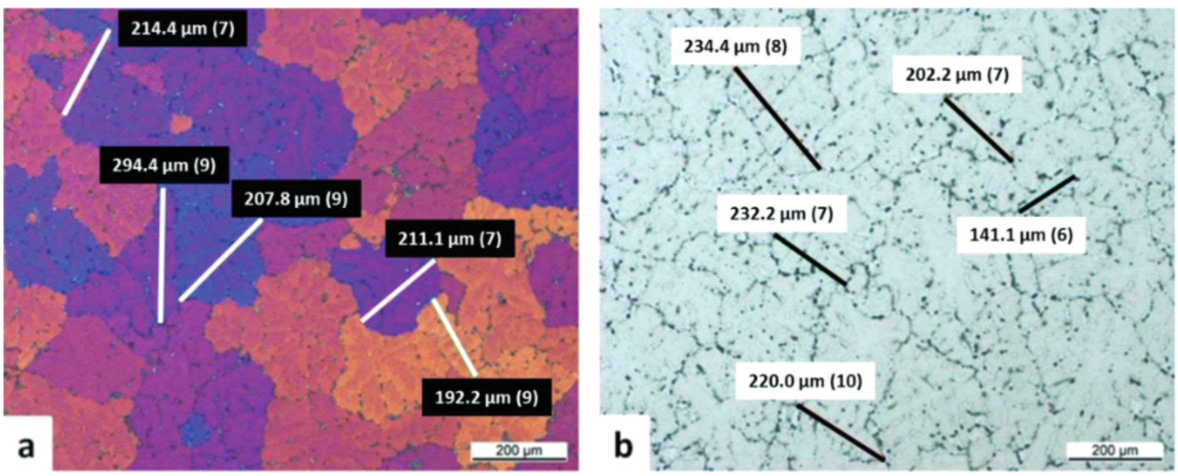

Figure 3: DAS measurements: a) in polarised light, b) in bright field

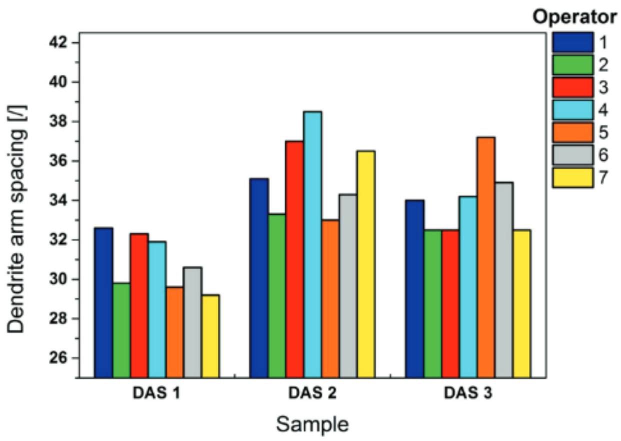

Figure 4: Average values of different operators' DAS measurements

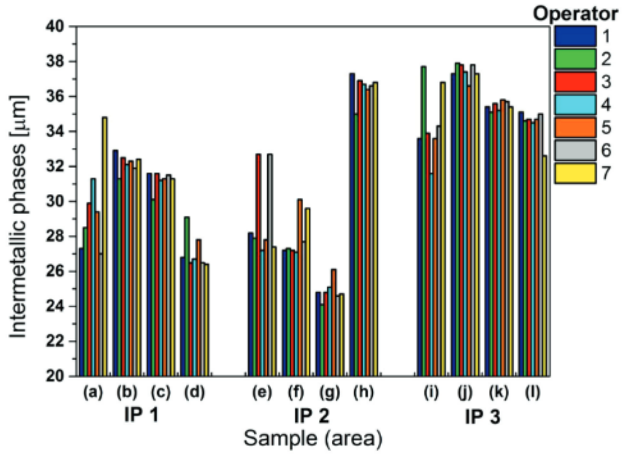

Figure 6: Found and measured longest intermetallic phases by different operators

phases, as presented in **Figure 5b**, where the measurement was performed. For the identification and determination of the intermetallic phases, the colour or grey-scale distinction is very useful in most cases.[24] The range from 26.5 µm (sample IP 2, area g) as the shortest found intermetallics to the 39.2 µm (sample IP 3, area j) as longest measured intermetallic phase, is presented in **Figure 6**. Besides the very low *s* and 95 % *CI* (1.1 µm and 1.0 %, respectively) the % *RA* is relatively high. However, the results of different operators show the suitability of this metallographic method. A more detailed, final, statistical evaluation of this technique is presented in **Table 3**.

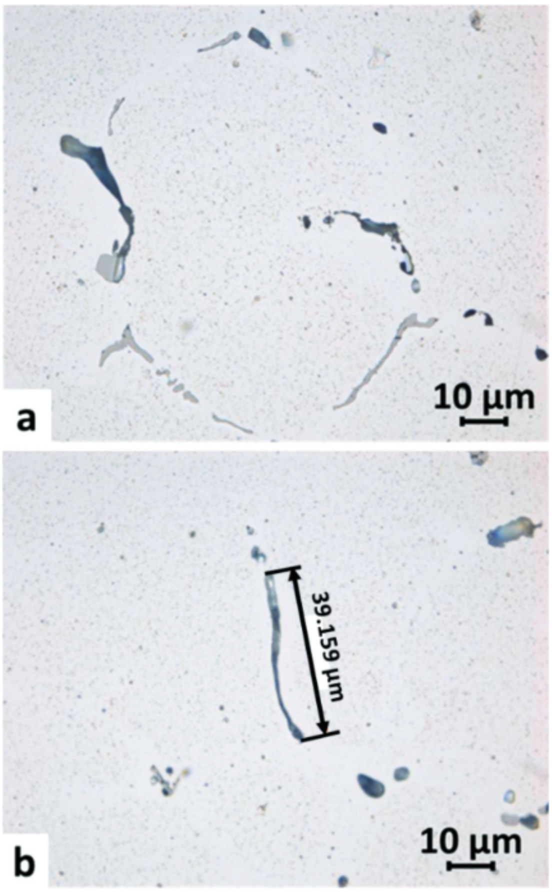

Figure 5: Intermetallic phases: a) example of different and composed intermetallic phases, b) example of one continuous, non-interrupted intermetallic phase with measurement

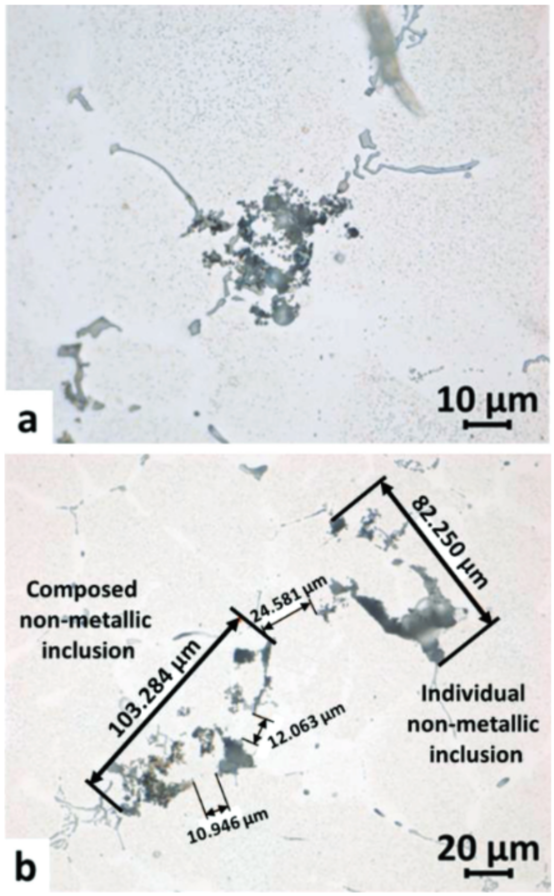

Figure 7: Non-metallic inclusions: a) nest of unreacted grain refiner as typical type of non-metallic inclusion, b) non-metallic inclusions size measurements (thicker lines) and verification of the distance between them (thinner lines) for a possible consideration of one inclusion

Table 3: Statistical evaluation of metallographic techniques

	MIN difference (µm)	MAX difference (µm)	Standard deviation (µm)	Confidence index (95 % *CI*) (%)	Relative accuracy (*RA*) (%)
Rim zone	0	14.3	3.0	2.8	1.0
DAS	0	5.5	1.7	1.6	4.7
Intermetallic phases	0	7.8	1.1	1.0	3.4
Non-metallic inclusions	0	9.0	1.1	1.0	2.3

3.4 Non-metallic inclusions

On the one representative sample NMI N the operators found between 61 and 65 different non-metallic inclusions larger than 40 µm. For this metallographic control method is the defined rule that one large non-metallic inclusion can lead to the rejection and reclamation of the material. Between seven different operators there is statistically only an 8 % possibility to overlook the non-metallic inclusion with critical size (in our case 40 µm). With the decrease of the defined critical size of non-metallic inclusions, the possibility of overlooking them will increase due to the easier overlooking of the smaller inclusions.

Figure 7a presents the nets of unreacted titanium boride grain refiner. It is also commonly found in the combination of non-metallic inclusions of unreacted grain refiner, oxides and oxide films, salts and intermetallic inclusions,[14,24] as shown in **Figure 7b**. In the same figure is the example of a composed non-metallic inclusion in the case, where two or more non-metallic inclusions are at a mutual distance less than 20 µm. There is also the example where the distance between non-metallic inclusions is larger than 20 µm, and where the measurements need to be performed separately for each inclusion. Seven areas on four samples were reviewed for the non-metallic inclusions and their dimensions were measured. In **Figure 8** are the graphically presented measurements for the individual non-metallic inclusions. When the operators considered all the previously defined rules, the results of the metallographic method for the determination of non-metallic inclusions do not differ significantly. Last but not least, it is also important to mention that two or more operators measured exactly the same size of one or more non-metallic inclusions.

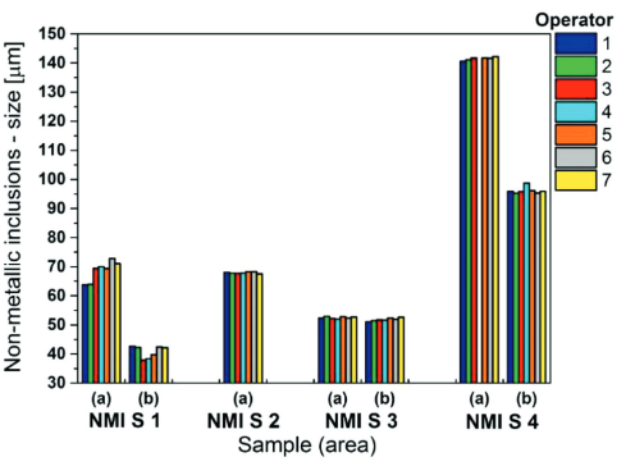

Figure 8: Found and measured largest non-metallic inclusions by different operators

A statistical evaluation of the presented metallographic methods (**Table 3**) indicates the high importance of precisely defined instructions and their strict application. The highest % *RA* was calculated for DAS measurements, because the rules do not define the exact measurement points. Only for the rim-zone measurements, the 95 % *CI* was higher than 2.0 %. For all four methods, the MIN difference was 0, which means that two or more operators have measured or determined the same value.

4 CONCLUSIONS

Metallographic methods and analyses techniques are an important part of scientific work as well as in industrial control systems. Non-standard methods and techniques are therefor often the result of the metallographic laboratory's creativity, where the potential validation presents a challenge. Four different non-standard metallographic methods were performed and evaluated based on a consideration of different impacts. With the combination of metallographic work and statistical calculations, we can conclude:

When we determine the longest length of the rim zone in the material, it is important to observe the whole rim/edge around the sample. Otherwise, the operators can observe and measure the longest value incorrectly. The longest value is only one measurement and not the average value of measurements.

The dendrite arm spacing (DAS) can be successfully determined using LM with polarised light or observed in bright field. In both cases it is necessary for the samples to be suitably etched (Barker reagent). When using the LM bright-field technique for measuring the dendrite length, it is necessary to be more careful, because dendrites in bright field are less evident compared to the ones in polarised light (colours).

The prior precise definition of non-metallic inclusions is the most important for a correct metallographic method's performance. However, only 8 % of the possibility to overlook the non-metallic inclusion with defined critical size was statistically calculated.

The relative accuracy (% *RA*) was less than 10 % for longest intermetallic phase measurement, which is in good agreement with standards for method's verification. Besides the longest intermetallic phase, the harmful impact of intermetallic phase's density, individual phases and intermetallic phases surrounded with non-metallic inclusions, can be the part of further investigations.

The suitability and the success of each presented and evaluated metallographic methodology is directly correlated with the precise definition and description for a determination of an individual microstructural component.

Acknowledgment

This research was part of an inter-laboratory proficiency project carried out between the company Impol and the accredited metallographic laboratory at the Institute of Metals and Technology, which was partly financed by the Slovenian Research Agency (research core funding No. P2-0132).

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