LAB PROJECT

No.: 191-2015

Příloha 1 - Měření tvrdosti nekovových vměstků:

To be completed by the client:	
Client:	Mr. Stika
Order number:	6543104
Customer / Type:	JLR AJ 200 Diesel
Parts no.	
Production date:	
To be finished till:	May, 21th 2015

☐ Macrograph ☐ Porosity ☐ S-DAS ☐ Hardness ☐ Mech. properties

☐ Micrograph ☒ Others: Analysis of black dots at the camshaft side

To be completed by the employee	
Complaints No.:	
Priority (A – F):	
Done by:	Mrs. Huber
Fixed date:	
Completed on:	May, 21th 2015

PROBLEM:

After machining black dots at the camshaft side are visible.

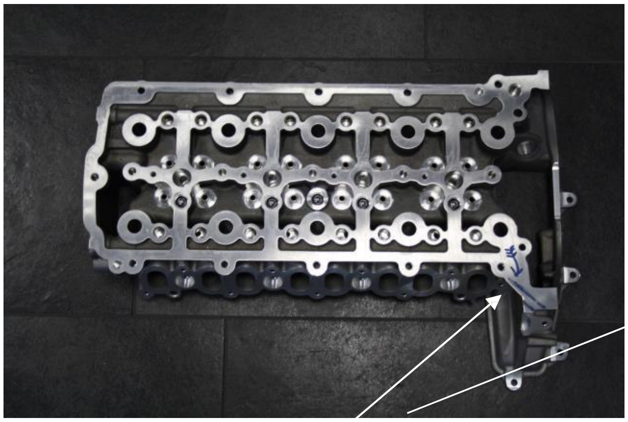
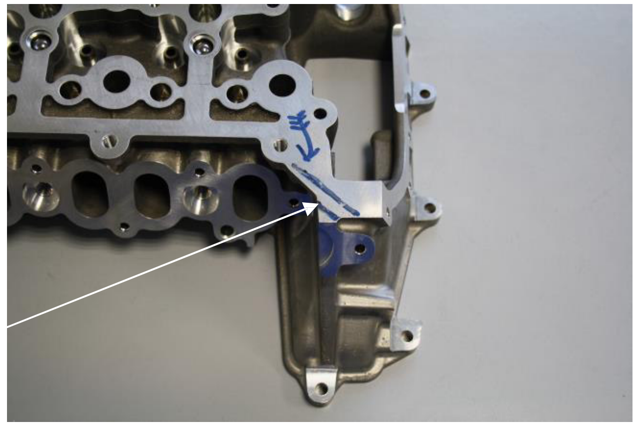
RESULT OF ANALYSIS:

The black dots are inclusions made of corundum (α- Al_2O_3) in a size of 0,3-0,6mm.

Corundum (α- Al_2O_3) is formed after long heating of the aluminum melt at temperatures above 700°C

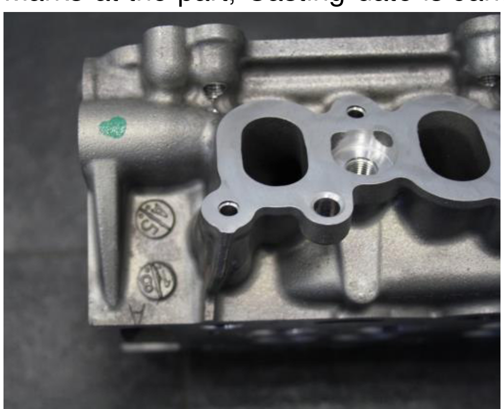
Evaluation of lab results by the client:

1 Overview

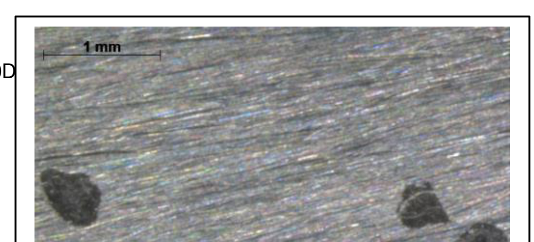



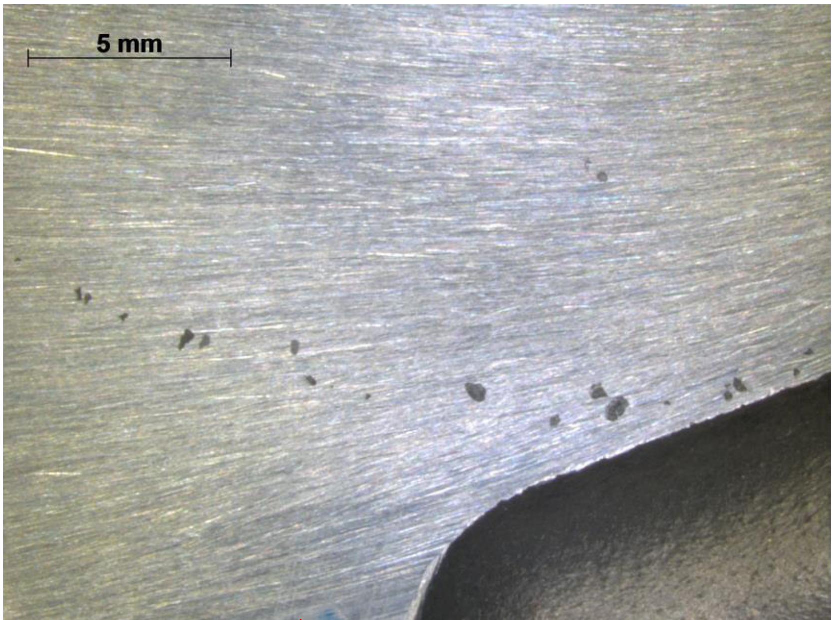
Positions of the black dots at the part.

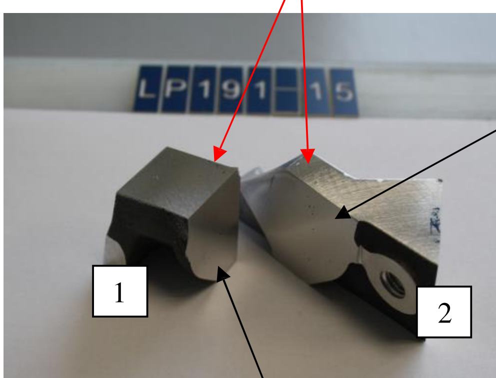
Marks at the part, Casting date is January, 18th 2015

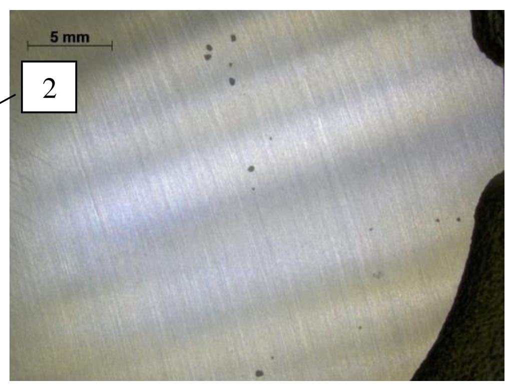


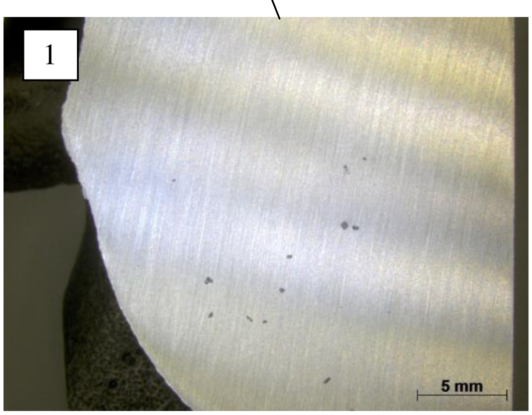
2 The black dots in detail









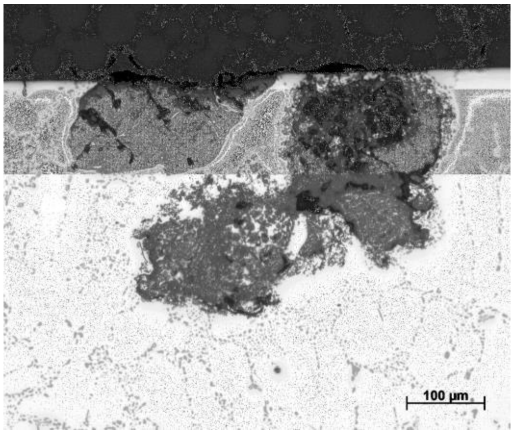


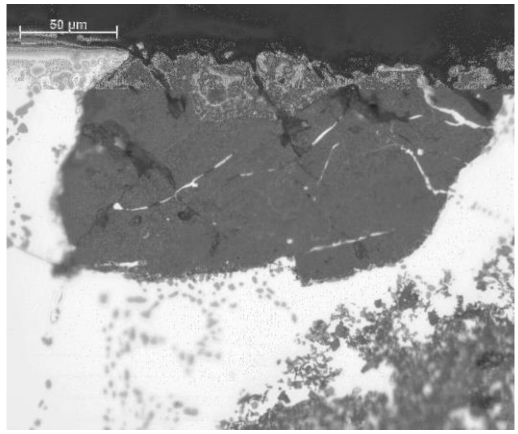
The black dots are inclusions in the material

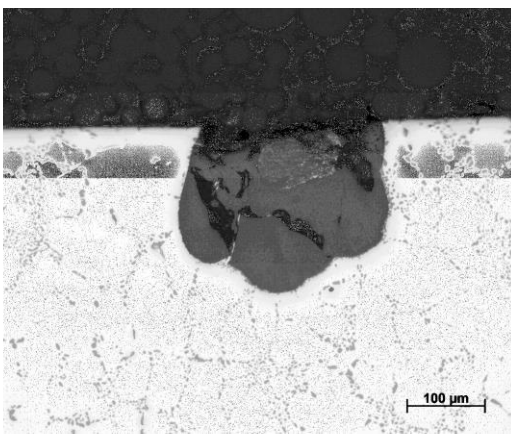
3 Mirco grindings from sample $\boxed{1}$

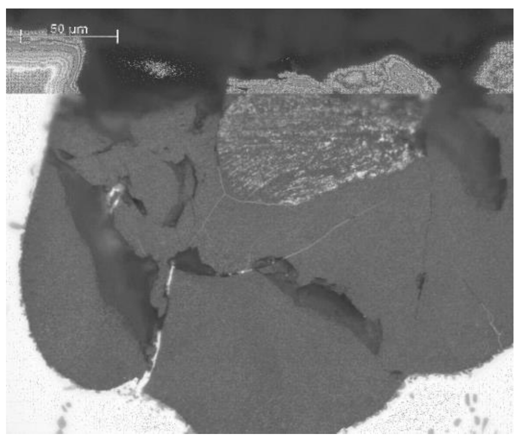
Micro grinding A (vertical)

Details of the inclusions





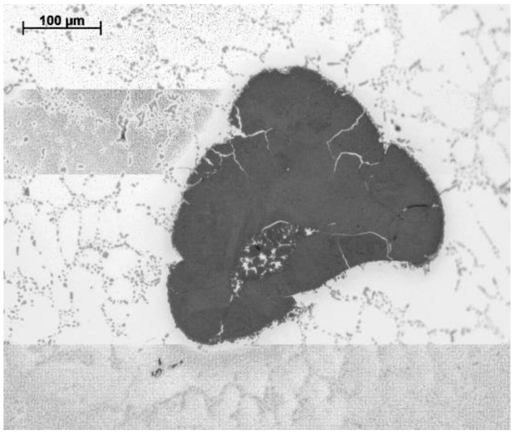


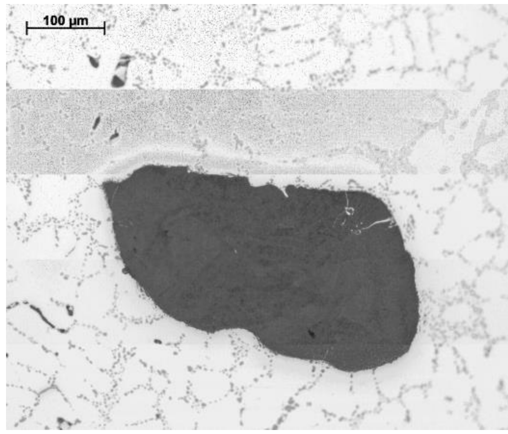


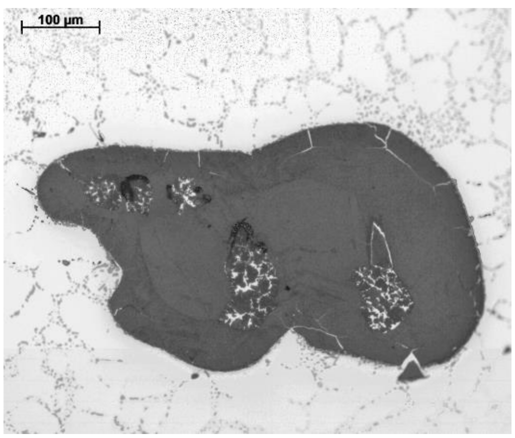
hardness 1380 HV 0,5

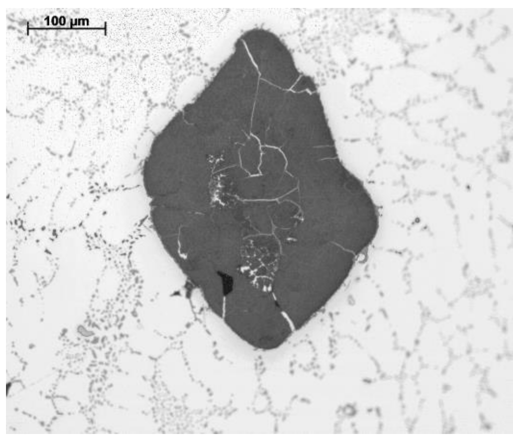
Micro grinding B (horizontal)

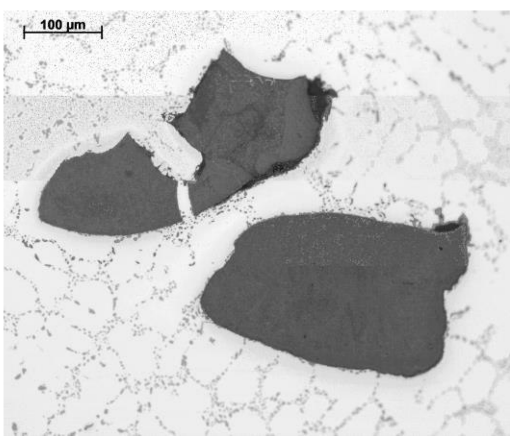
Details of the inclusions











hardness 1100 to 1440 HV 0,5

ZKUŠEBNÍ ZPRÁVA

Datum:	26. 06. 2015
Firma:	**NEMAK Czech Republic s.r.o.**
Vzorek:	Vměstky ve slitině hliníku
Jednáno s:	**M. Hawelka**
Měření provedeno:	VUT Brno, FSI
Měření provedl:	Doc. Ing. Bohumil Pacal CSc.
Rozdělovník:	akta zákazníka, S. Ivanov

Vměstky ve slitině hliníku MgO . Al_2O_3 ($MgAl_2O_4$)

	Mohsova stupnice tvrdosti	Absolutní tvrdost
Diamant	10	1600
HV0,05 MgO	5,5	60
Al_2O_3 – korund (α)	9	400

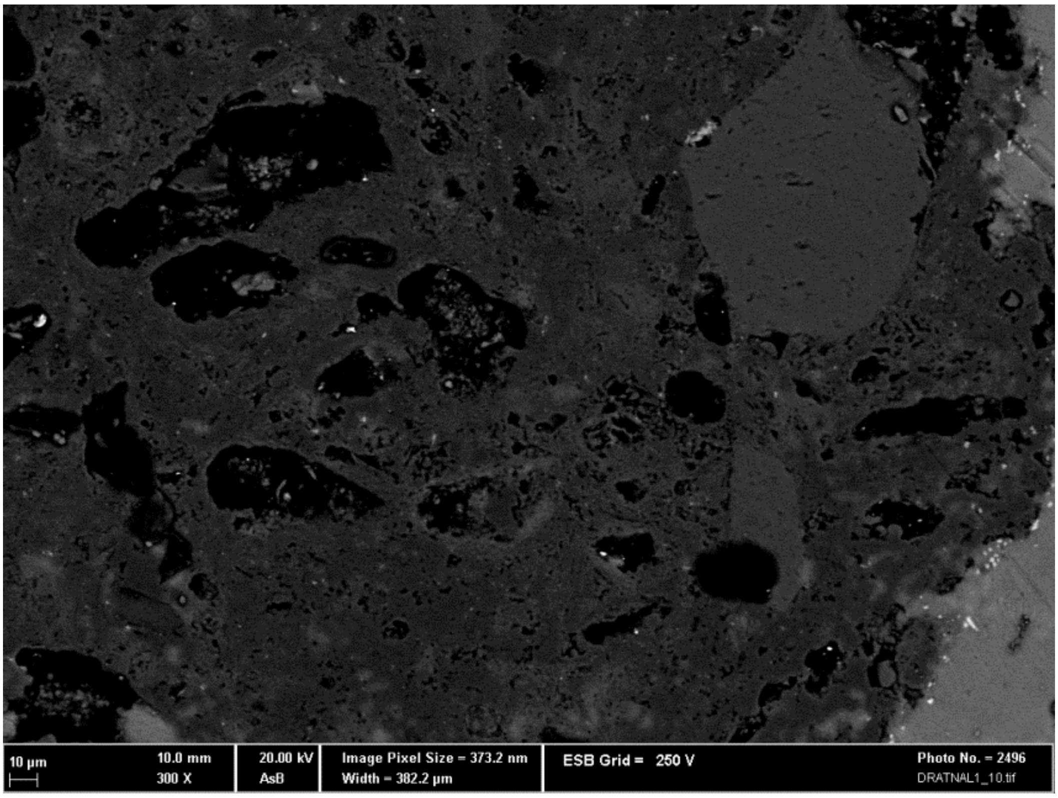
	Přepočtená tvrdost na Vickerse
Al_2O_3:	2070 HV
MgO:	633 HV

Naměřené hodnoty tvrdosti vměstků na vzorku:

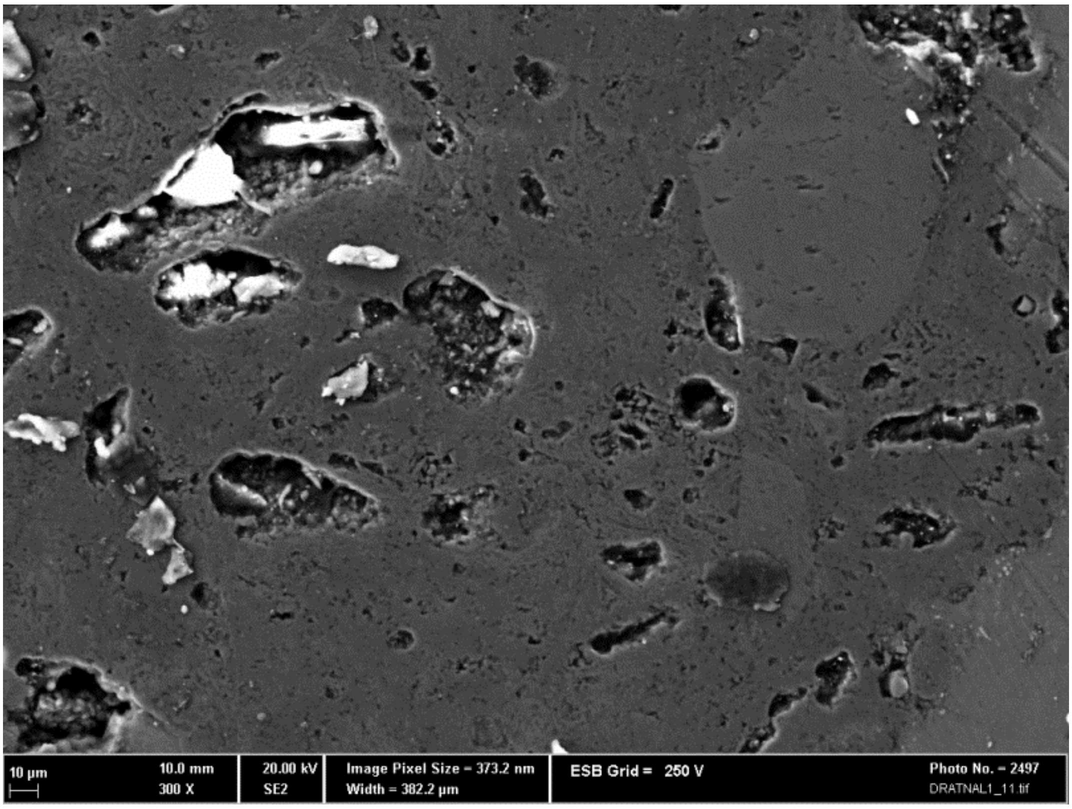
P.č. měření	Tvrdost HV 0,3
1	1369
2	1205
3	1224
4	1382
5	1322

Pozn.:

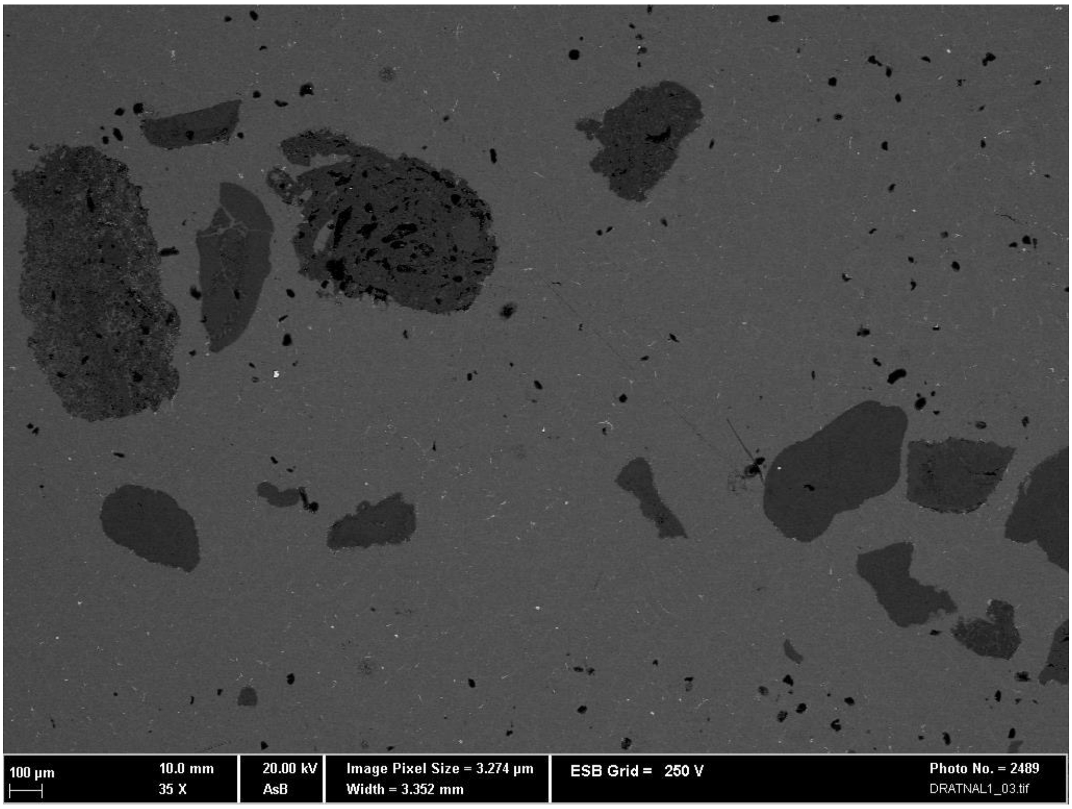
Příloha 2 – Snímky z elektronového a světelného mikroskopu:



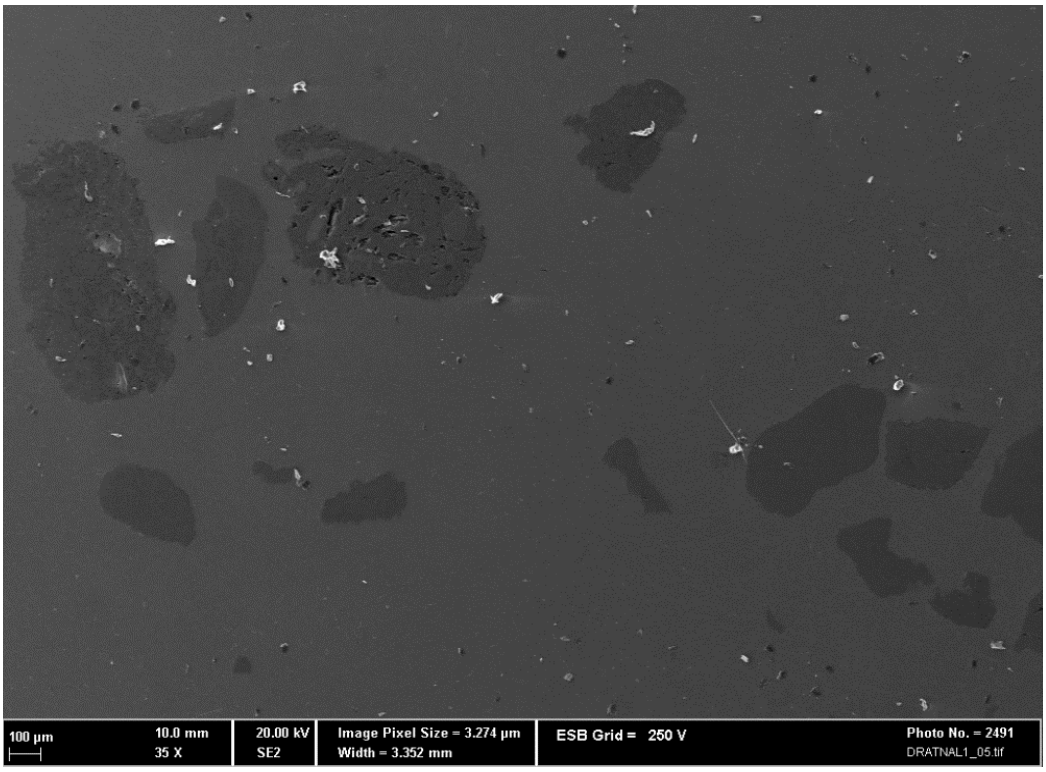
Obrázek 1: Morfologie vměstku – SEM [zdroj: vlastní]



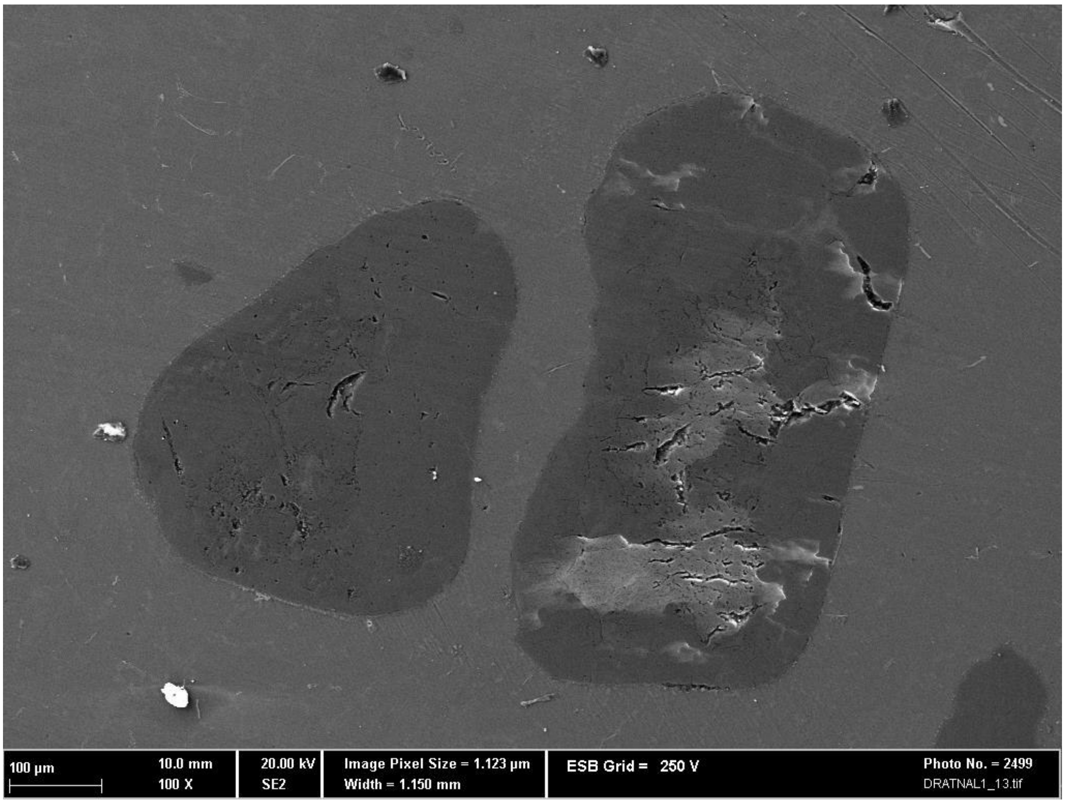
Obrázek 2: Morfologie vměstku – SEM [zdroj: vlastní]



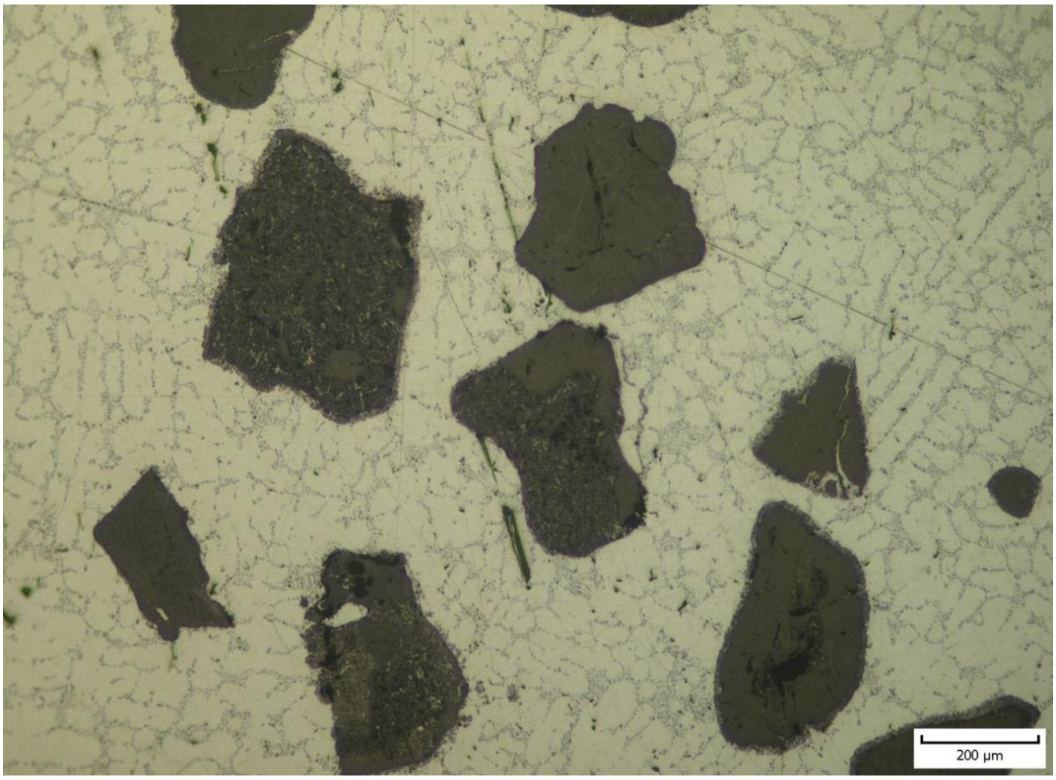
Obrázek 3: Uspořádání vměstků – SEM [zdroj: vlastní]



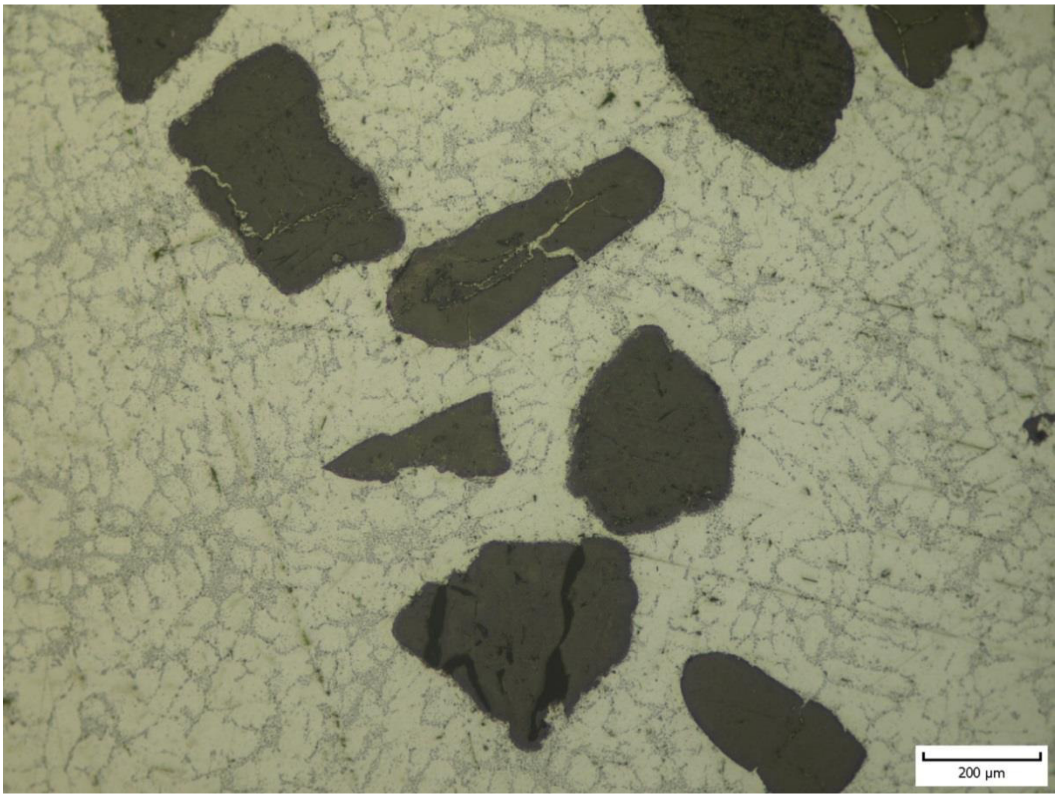
Obrázek 4: Uspořádání vměstků – SEM (prachové částice) [zdroj: vlastní]



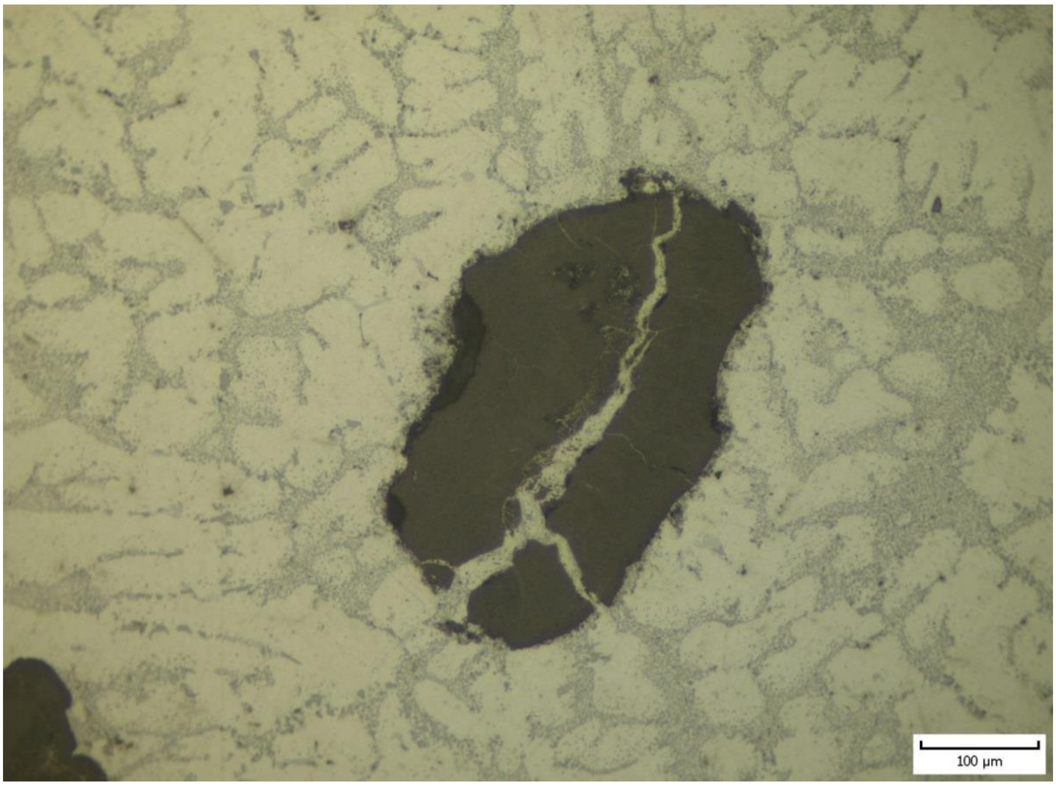
Obrázek 5: Morfologie vměstku – SEM [zdroj: vlastní]



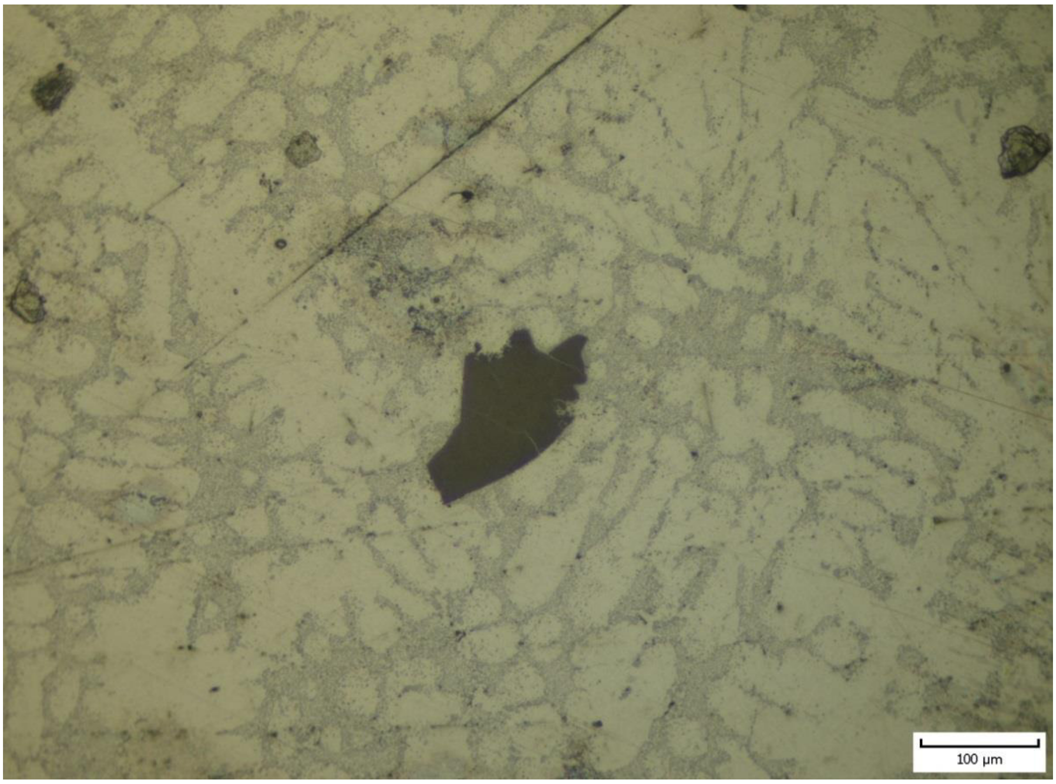
Obrázek 6: Uspořádání a velikosti vměstků - světelný mikroskop [zdroj: vlastní]



Obrázek 7: Uspořádání a velikosti vměstků - světelný mikroskop [zdroj: vlastní]



Obrázek 8: Uspořádání a velikosti vměstků - světelný mikroskop [zdroj: vlastní]



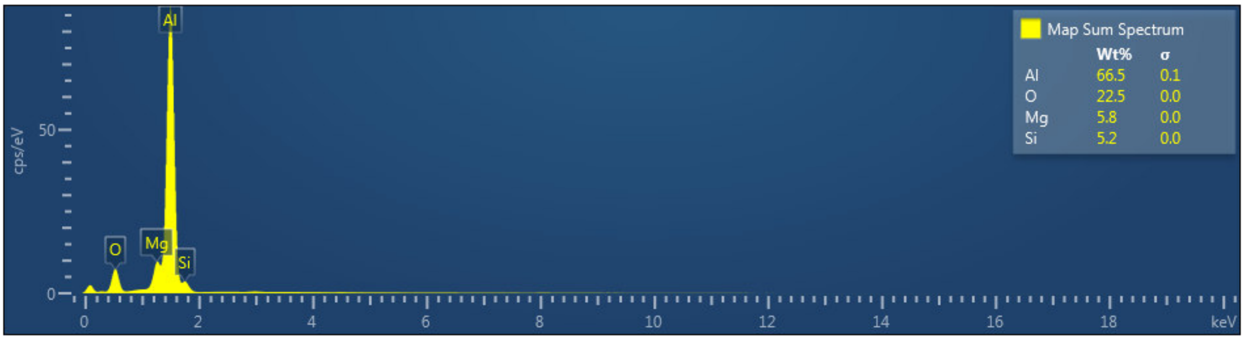
Obrázek 9: Uspořádání a velikosti vměstků - světelný mikroskop [zdroj: vlastní]

Příloha 3 - Analýza chemického složení vměstků - SEM:

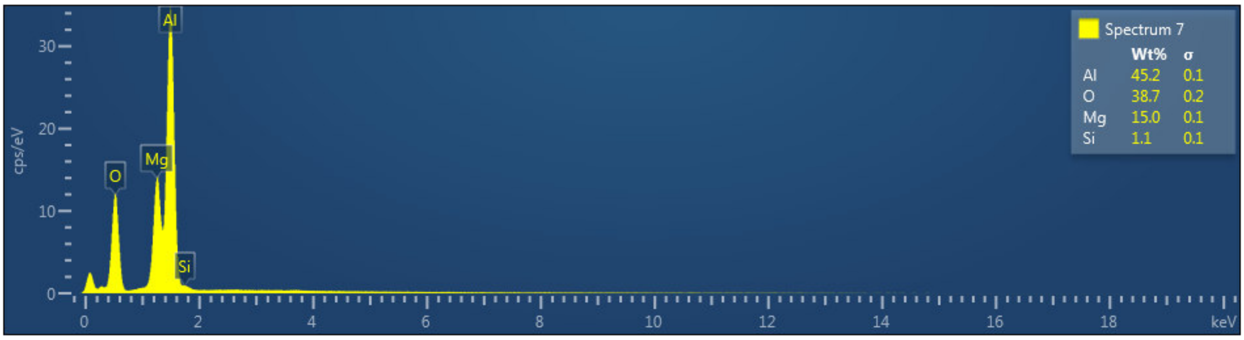
Spectrum 1								
Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	47,98	0,16147	37,9	0,12	50,05	SiO2	Yes
Mg	K series	16,96	0,11251	14,5	0,08	12,62	MgO	Yes
Al	K series	44,92	0,32265	46,8	0,12	36,65	Al2O3	Yes
Si	K series	0,55	0,00439	0,91	0,06	0,68	SiO2	Yes
Total:				100		100		
Spectrum 2								
Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	48,73	0,16398	38,2	0,13	50,44	SiO2	Yes
Mg	K series	17,11	0,1135	14,6	0,08	12,71	MgO	Yes
Al	K series	43,77	0,31436	45,6	0,12	35,68	Al2O3	Yes
Si	K series	0,96	0,0076	1,55	0,06	1,17	SiO2	Yes
Total:				100		100		
Spectrum 3								
Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	49,17	0,16545	38,6	0,15	50,89	SiO2	Yes
Mg	K series	16,58	0,10995	14,3	0,09	12,41	MgO	Yes
Al	K series	43,54	0,31273	45,5	0,13	35,55	Al2O3	Yes
Si	K series	0,94	0,00743	1,52	0,07	1,14	SiO2	Yes
Total:				100		100		
Spectrum 4								

Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	50,61	0,1703	38,9	0,13	51,13	SiO2	Yes
Mg	K series	17,09	0,11332	14,6	0,08	12,6	MgO	Yes
Al	K series	44,23	0,31765	45,8	0,12	35,71	Al2O3	Yes
Si	K series	0,47	0,00371	0,75	0,06	0,56	SiO2	Yes
Total:				100		100		
Spectrum 5								
Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	47,03	0,15825	37,8	0,16	50	SiO2	Yes
Mg	K series	16,57	0,1099	14,3	0,09	12,48	MgO	Yes
Al	K series	43,66	0,31358	45,8	0,15	35,97	Al2O3	Yes
Si	K series	1,25	0,00988	2,04	0,08	1,54	SiO2	Yes
Total:				100		100		
Spectrum 6								
Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	50,89	0,17123	39	0,13	51,21	SiO2	Yes
Mg	K series	17,6	0,11676	15	0,08	12,96	MgO	Yes
Al	K series	43,02	0,30902	44,8	0,12	34,87	Al2O3	Yes
Si	K series	0,81	0,00643	1,29	0,06	0,97	SiO2	Yes
Total:				100		100		
Spectrum 8								

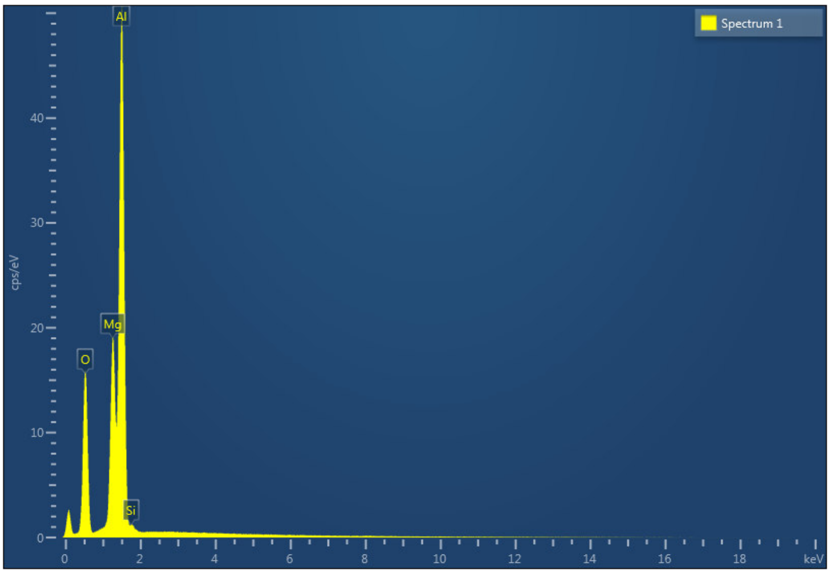
Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	39,48	0,13284	38,4	0,22	51,1	SiO2	Yes
Mg	K series	4,36	0,02892	4,26	0,08	3,73	MgO	Yes
Al	K series	56,01	0,40225	56,1	0,21	44,25	Al2O3	Yes
Si	K series	0,65	0,00516	1,22	0,07	0,92	SiO2	Yes
Total:				100		100		
Spectrum 9								
Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	43,18	0,14531	38,3	0,24	50,55	SiO2	Yes
Mg	K series	14,27	0,09466	13,2	0,13	11,49	MgO	Yes
Al	K series	43,29	0,31093	47,7	0,22	37,35	Al2O3	Yes
Si	K series	0,46	0,00361	0,8	0,07	0,6	SiO2	Yes
Total:				100		100		
Spectrum 10								
Element	Line Type	Apparent Concentration	k Ratio	Wt%	Wt% Sigma	Atomic %	Standard Label	Factory Standard
O	K series	42,64	0,1435	38,4	0,26	50,7	SiO2	Yes
Mg	K series	14,52	0,09627	13,7	0,14	11,92	MgO	Yes
Al	K series	41,18	0,29579	46,7	0,23	36,51	Al2O3	Yes
Si	K series	0,65	0,00513	1,16	0,08	0,87	SiO2	Yes
Total:				100		100		



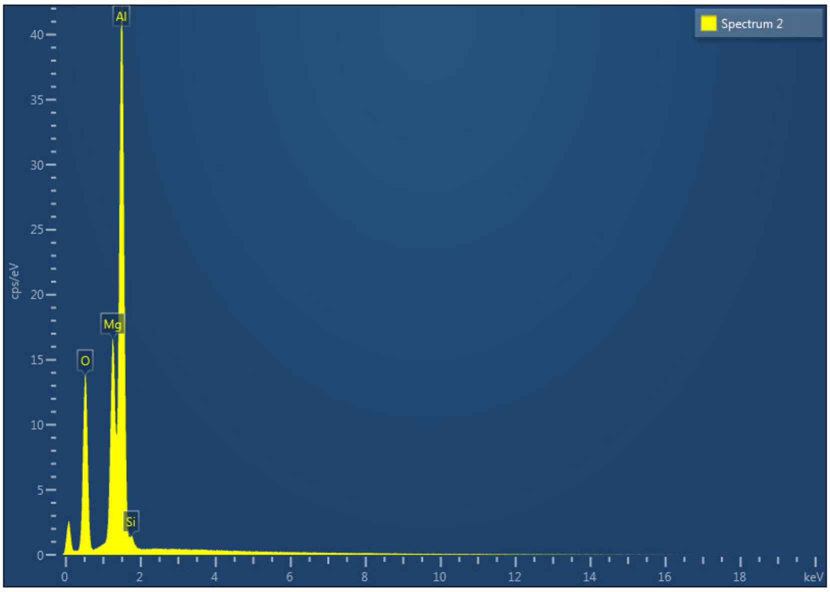
Obrázek 10: EDS mapování – celá oblast [zdroj: vlastní]



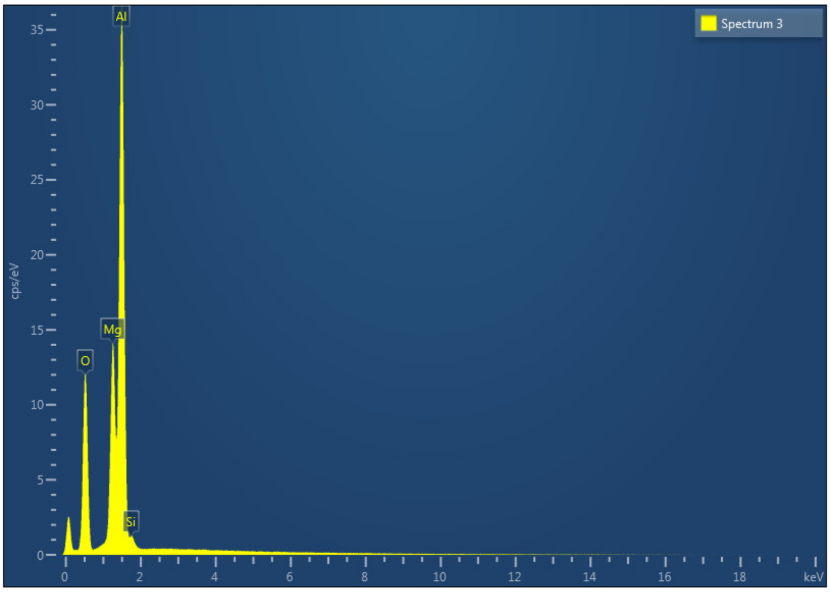
Obrázek 11: EDS mapování – složení plošné oblasti [zdroj: vlastní]



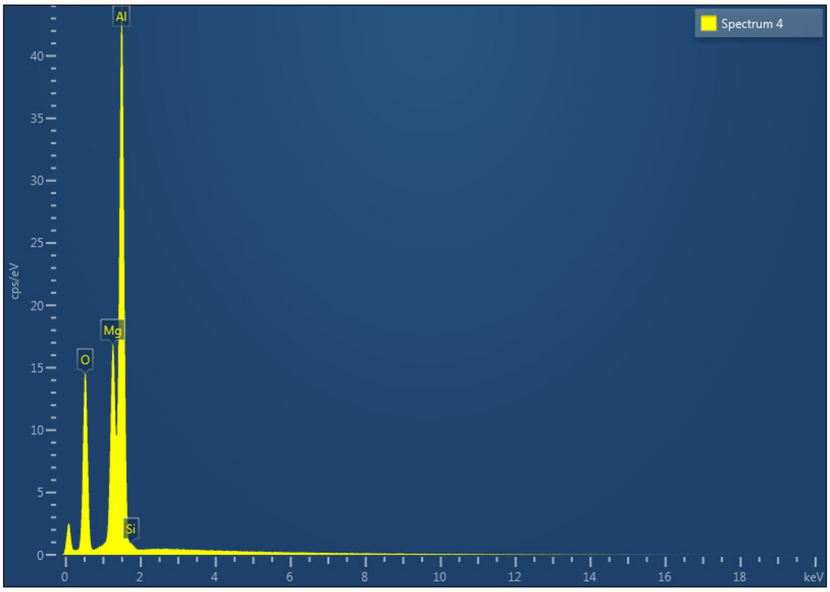
Obrázek 12: Chemické složení – spectrum 1 [zdroj: vlastní]



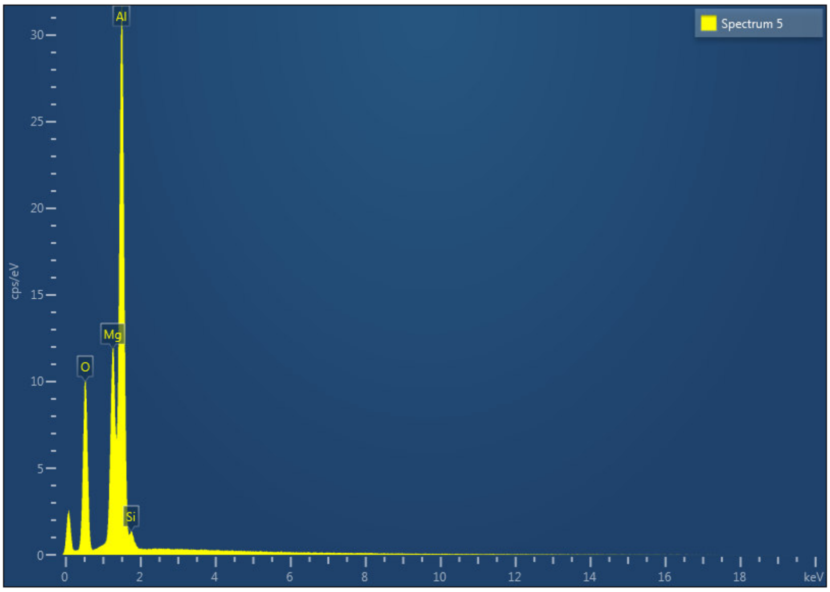
Obrázek 13: Chemické složení – spectrum 2 [zdroj: vlastní]



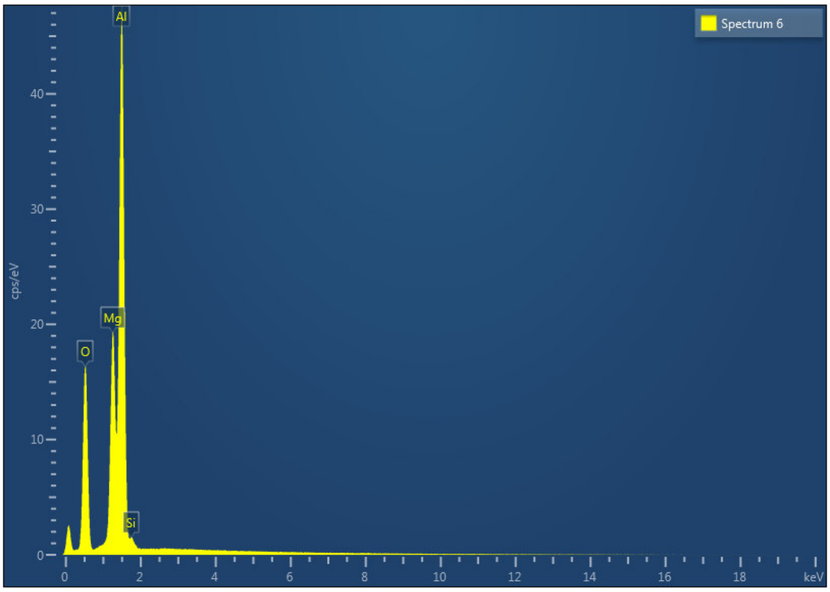
Obrázek 14: Chemické složení – spectrum 3 [zdroj: vlastní]



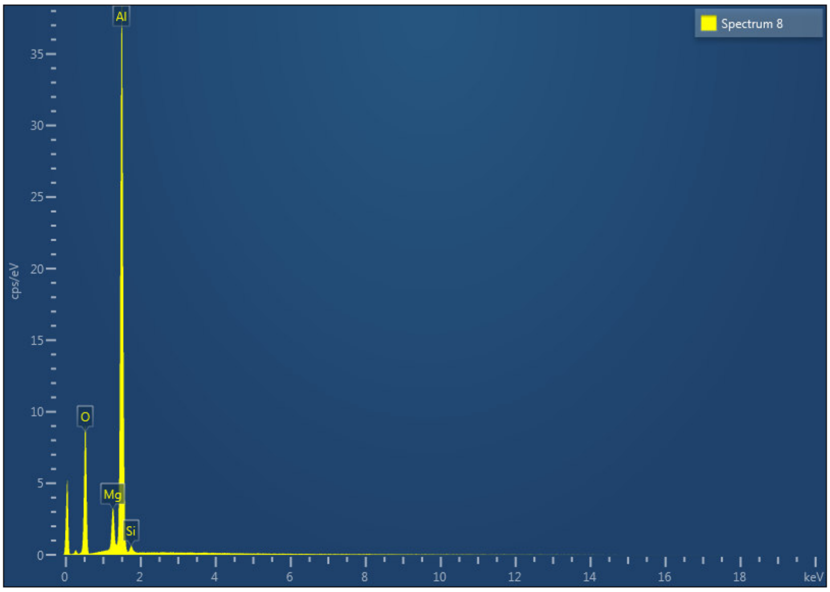
Obrázek 15: Chemické složení – spectrum 4 [zdroj: vlastní]



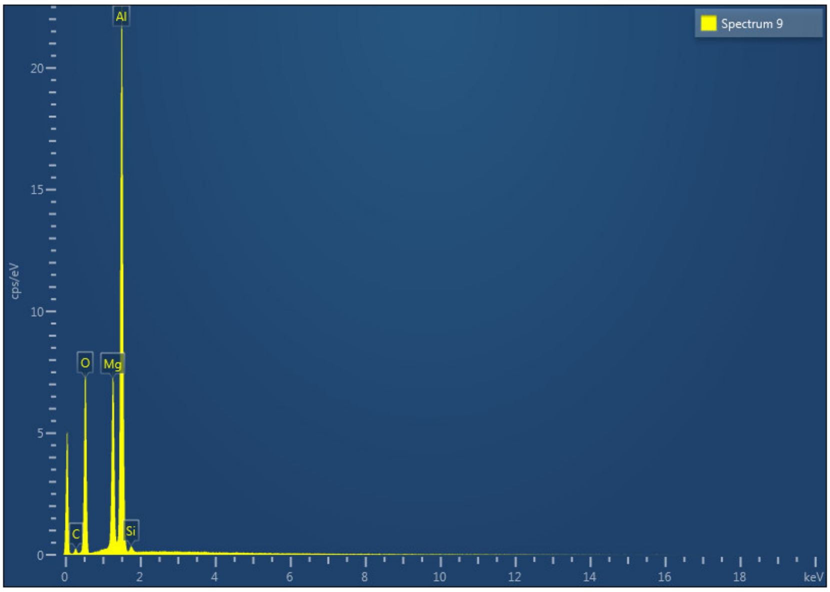
Obrázek 16: Chemické složení – spectrum 5 [zdroj: vlastní]



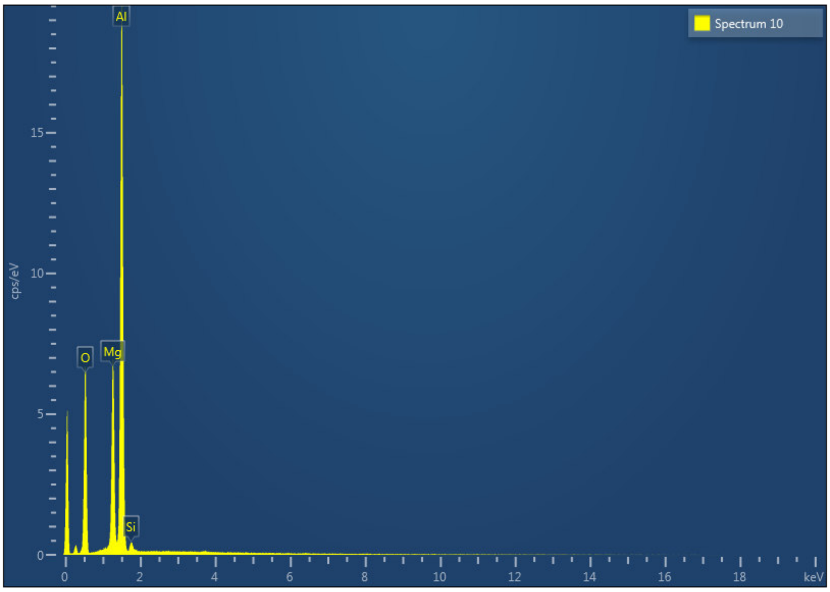
Obrázek 17: Chemické složení – spectrum 6 [zdroj: vlastní]



Obrázek 18: Chemické složení – spectrum 8 [zdroj: vlastní]



Obrázek 19: Chemické složení – spectrum 9 [zdroj: vlastní]



Obrázek 20: Chemické složení – spectrum 9 [zdroj: vlastní]

Příloha 4 – čistota taveniny v udržovacích pecích:

Tabulka 1: Spektrální analýza kovu v udržovacích pecích [zdroj: vlastní]

Prvek	ZB			DOL			ODPL		
	KP2	KP3	KP4	KP2	KP3	KP4	KP2	KP3	KP4
Si	6,97	6,9	6,93	6,98	6,92	6,85	7,1	6,82	6,82
Fe	0,086	0,091	0,092	0,086	0,086	0,089	0,088	0,085	0,087
Cu	0,513	0,532	0,526	0,516	0,519	0,525	0,528	0,514	0,515
Mn	0,064	0,068	0,063	0,064	0,067	0,063	0,066	0,066	0,063
Mg	0,366	0,377	0,373	0,368	0,378	0,372	0,373	0,371	0,368
Zn	0,005	0,005	0,011	0,005	0,005	0,008	0,006	0,005	0,008
Ni	0,003	0,003	0,003	0,003	0,002	0,003	0,003	0,002	0,003
Cr	0,001	0,001	0,002	0,001	0,001	0,001	0,001	0,001	0,001
Pb	0,001	0,001	0,001	0,001	0,001	0,001	0,001	0,001	0,001
Sn	0	0	0	0	0	0	0	0	0
Ti	0,126	0,137	0,131	0,125	0,146	0,128	0,125	0,149	0,128
B	<1	<1	<1	<1	<1	<1	<1	<1	<1
Ca	3,1	5,23	7,48	3,0	7,44	5,45	2,3	7,2	4,86
Na	2,5	2,53	3,85	2,41	3,57	3,33	2,32	3,48	2,74
P	7,63	8,74	10,17	8,44	8,99	8,43	9,91	8,79	10,57
Sb	<2	<2	<2	<2	<2	<2	<2	<2	<2
Sr	201	199	215	202	212	211	206	209	211
Al	91,84	91,86	91,85	91,86	91,85	91,94	91,69	91,96	91,98

Tabulka 2: Hodnoty naplynění taveniny měřené metodou dvojího vážení [zdroj: vlastní]

Hodnota naplynění	ZB			DOL			ODPL		
	KP2	KP3	KP4	KP2	KP3	KP4	KP2	KP3	KP4
%	1.49	0.74	1.12	7.09	9.33	8.58	1.12	1.12	0.37

Obrázek 21: Vzorky pro spektrální analýzu slitiny - pec KP3 [zdroj: vlastní]

Obrázek 22: Vzorky pro určení hodnoty naplynění (vlevo ztuhlé ve vakuu za 80 mbar, vpravo ztuhlé za atmosférického tlaku) – pec KP3

FOSECO FOUNDRY R&D
4604 Campbells Run Road
Pittsburgh PA 15205 USA

FOSECO

Příloha 5 – Protokol z měření metodou Vmet:

Foseco R&D – Technical memorandum

Date: 15-Jan-16
Subject: LWR-PI-3277-15 Nemak Vmet Analysis
Author: B. Groth, Sr. Research Engineer, Foseco Foundry R&D – Pittsburgh, PA

Summary:
Six aluminum samples from Nemak foundry were sent to the PI lab for SEM and Vmet automated inclusion detection analysis. The Vmet analysis showed that the quality of metal in the crucible after filling from the transfer ladle was poor, containing a greater number of oxide inclusions. Following treatment in the crucible, the metal quality improved with many of the oxides being removed from the melt. This was consistent over repeated testing and treatment.

Background
A Nemak foundry casting cylinder heads for Jaguar was having issues with spinel inclusions within their castings. A study on their metal was conducted, with Foseco products used to treat the melt. Given below in Table 1 is the information on each casting sample taken, with the 2nd set of samples being taken from a repeat of the treatment process.

Table 1. Sample identification for the K-molds.

Sample ID	Condition	Treatment
1A	Metal Residue (before filling)	None
1B	After Filling	
1C	After Treatment (in crucible)	FDU, 18min XSR220, 30l/min
2A	Metal Residue (before filling)	None
2B	After Filling	
2C	After Treatment	FDU, 18min XSR220, 30l/min

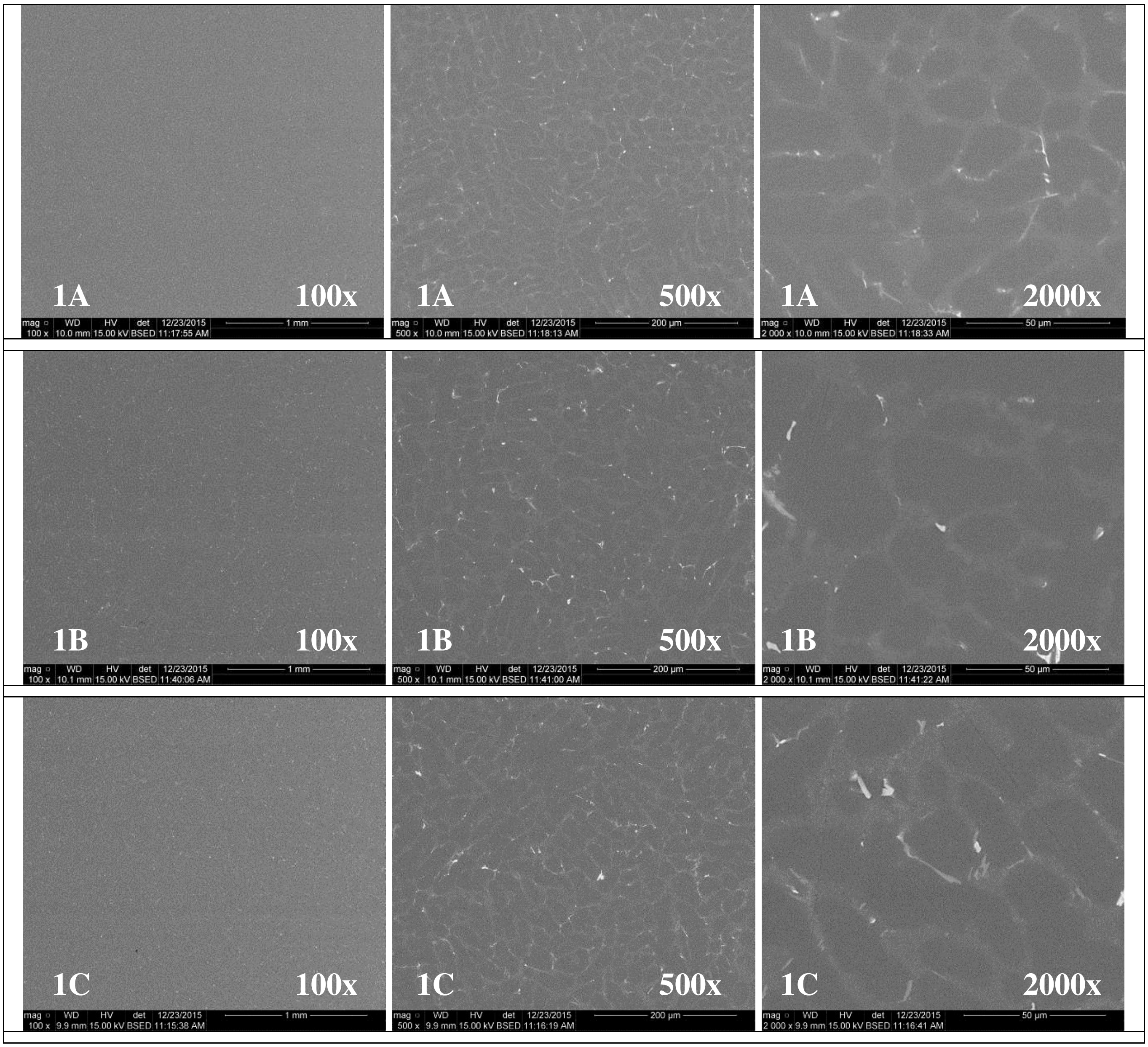
Experimental Procedure
The received samples were cut to fit in a 32 mm diameter sample cup. They were mounted in a heat set epoxy resin and polished using a Struers Hexamatic polisher. Polishing of aluminum Vmet samples follows a standard routine to ensure even polishing depth and sample uniformity. The routine used is as follows: grinding using a silicon carbide wheel for 20-30 seconds, followed by a 220 grit silicon carbide pad for 1 minute, then a nap pad and 9 micron diamond suspension is used for ~20 minutes followed by ~2 minutes of polishing with a 3 micron diamond suspension. The sample polishing is finished with a 400 nm colloidal silica suspension for 1 minute and buffing with an alcohol soaked nap pad. Samples are cleaned in an ultrasonic bath between each polishing step as well as at the end of polishing.

Micrograph images of polished samples are acquired using an SEM, with images taken at 100, 500, and 2000x magnification. Images are taken to provide a qualitative indication of the metal microstructure and porosity. Finally the samples are evaluated by Vmet analysis, an automated SEM analysis used to classify all found features by size and chemistry.

Results and Discussion

Given below in Figure 1 are the micrograph images taken from the SEM analysis. These are used a s a means of quickly qualifying the melt cleanliness of different samples. Dirtier melts will contain more pores and inclusions, and these typically show up at dark spots within the image. At lower magnifications these are more readily seen as the inclusions tend to appear in clusters and are often large. At higher magnifications the smaller inclusions are seen, along with any structural modification to the alloy. Micrographs from cleaner melts often do not show many inclusions and only at the higher magnifications.

For the images below, very few inclusions are seen at any magnification. Some small clusters and dark areas can be seen in the second set of samples at 100x, however these castings are overall quite clean and will require the quantitative Vmet analysis to fully differentiate between the level of cleanliness.



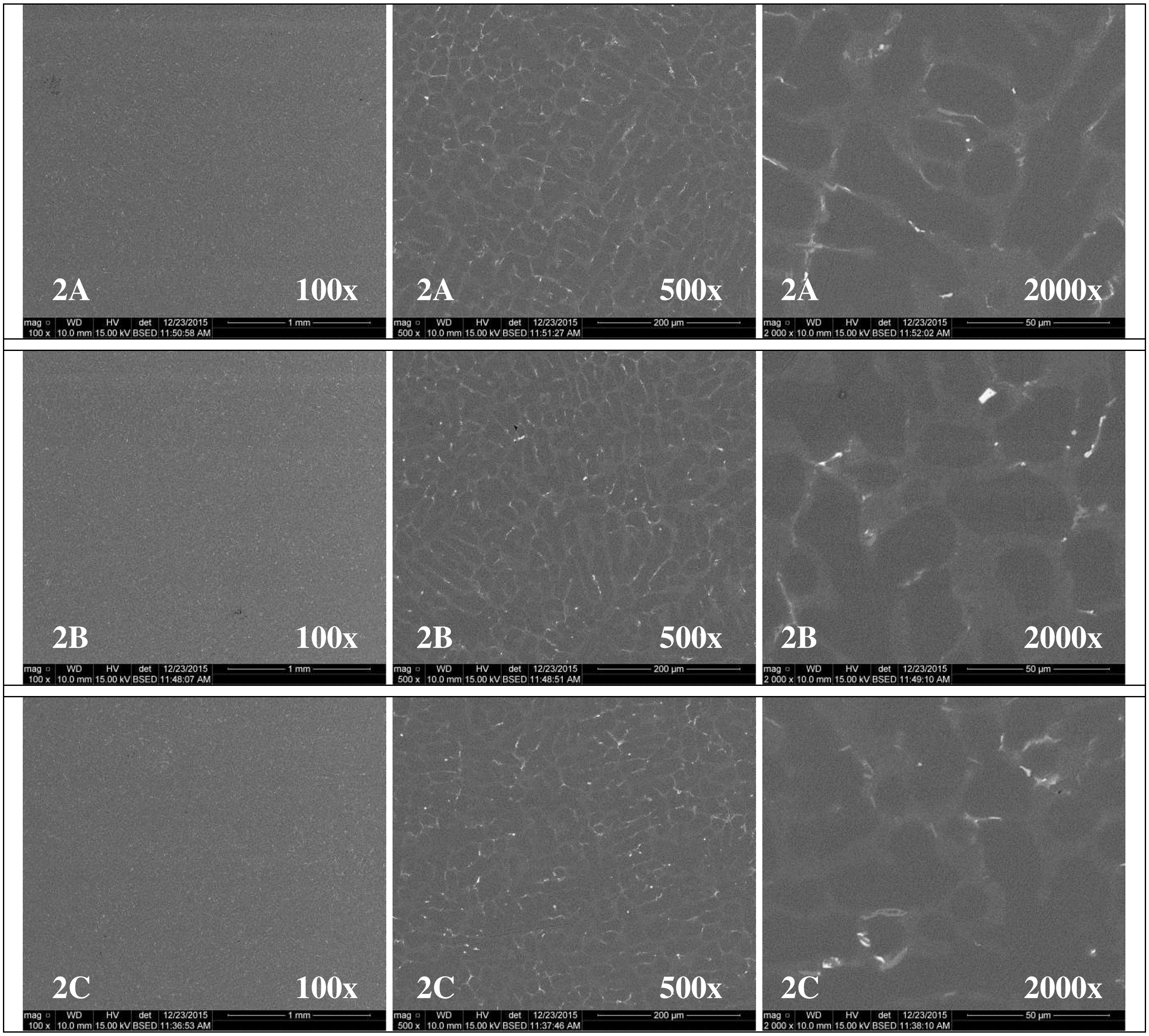


Figure 1. SEM images of the polished K-mold sections at increasing magnification.

Given below in Table 2 are the results of the Vmet analysis on the 6 k-mold castings in this trial. The Vmet analysis is an automated process that scans over a defined area on the sample and uses an analysis software to determine the inclusion content within the scanned area. This is done by comparing video level of the image, pixel by pixel against a threshold level. When an inclusion is found, chemical information is obtained on the area and it is measured to quantify the size and shape of the feature. All of this information is gathered and combined, providing a series of metrics to assess the cleanliness of the melt. Of these metrics, the “Inclusion Index” gives the best representation of the melt quality as this number is a ratio of inclusion area to total analyzed area; a lower inclusion index indicates fewer, or smaller, features and thereby a better quality casting.

Both sample sets “1” and “2” showed consistent results from the analysis. The residual metal was fairly clean in both cases, not surprising but also not typical depending on the melt level and condition of the vessel (crucible, transfer ladle, holding furnace etc.) After filling (“B” samples) the quality of the melt decreased, with a large increase in pores and specifically for “2B” a great increase in the number of oxides. It is unclear whether these are formed in the ladle or after filling, however following treatment ("C" samples) the level of both pores and oxides is reduced back below that of the residual melt level. Additionally, the size of inclusions is reduced slightly following treatment allowing for an even better inclusion index in the treated samples.

Table 2. Vmet data for all samples, 1A-2C.

Sample	**1A**	**1B**	**1C**	**2A**	**2B**	**2C**
Inclusion Index	2.3	12.8	1.8	3.7	24.9	3.2
Total Features	62	629	45	221	998	139
Area Analyzed (mm^2)	100.44	100.44	100.44	100.44	100.44	100.44
By Chemistry						
Pore	48	469	40	160	683	103
Dross	7	87	1	18	87	8
Alloy Dross	7	73	4	43	228	28
By Size						
0.50 – 2.50 µm	15	163	3	74	228	33
2.50 – 5.00 µm	23	332	22	115	456	68
5.00 – 15.0 µm	21	131	18	30	306	37
15.0 – 30.0 µm	3	1	2	2	8	1
30.0 – 75.0 µm	0	2	0	0	0	0
> 75.0 µm	0	0	0	0	0	0
EV Range (µm)	20.3-3.2	37.8-7.1	18.1-2.4	20.7-4.6	27.2-9.1	19.8-4.6