

OPTIMISATION OF SHRINKAGE IN THE DESIGN OF COMPACTION TOOLING FOR WC-C₀

Krzysztof Blaski

A dissertation submitted to the Faculty of Engineering and the Built environment,
University of the Witwatersrand, in fulfilment of the requirements for the Degree of
Master of Science in Engineering

Johannesburg, 2007

Declaration

I declare that this dissertation is my own work. It is being submitted for the Degree of Master of Science in Engineering in the University of the Witwaterstand, Johannesburg. It has not being submitted before for any degree or examination in any other University



K.Blaski

31st day of March (year) 2007

Abstract

Tungsten carbide-cobalt powder is pressed before sintering into a compacted form using punches and a die cavity. After the powder has been pressed to a specific shape, it is sintered and shrinks a certain amount to a final size. To accommodate this shrinkage, the pressing tools are designed to a certain “shrinkage percentage” and thus the pressed component or compact is larger than the sintered component by that percentage amount. During the pressing process, there is a large amount of friction between the powder being compacted and the die cavity wall. To counter pressing friction, a lubricant is pre-mixed with the tungsten carbide powder. In the past at Powder Industries, the powder was mixed with wax and all of the tools were designed to a 20% shrinkage. In recent times, the wax in the powder has been replaced by PEG (polyethylene glycol) by most manufacturers as this increases the quality of the final product and is easier to remove in the furnaces. As a result of the new PEG lubricant, the tool wear rate at Powder Industries increased and because a higher pressure had been necessary to achieve powder pressing to the same shape and form, often the pressed components exhibited cracks or were not pressed ideally.

On account of the problems introduced by PEG, correct tool design for the shrinkage was obtained by a ‘trial & error’ process. This project has been motivated by the need of establishing pressing and/or design ‘rules’ that would do away with trial and error when designing compaction tooling.

The project has consisted of investigating the physical properties of 23 grades of WC-Co powder (with or without TiC and TaC) and of performing a series of pressing tests for each grade. A relationship between the apparent density of a powder and the ideal green density of the green compact pressed from the same powder has been found. Using this relationship, an equation has been derived between ideal shrinkage, powder apparent density, component sintered density and powder volatile content. Since the last three parameters are known to the tool designer, this equation can be used to calculate the ideal shrinkage when designing new compaction tooling. This method of calculating shrinkage is now in general use at Powder Industries and many successful sets of compaction tooling have already been manufactured.

Dedication

I dedicate this work to my family, thank you for your support.

Acknowledgements

I would like to thank Powder Industries for initiating this project and for sponsoring it. Also I would like to thank Professor Luyckx for her invaluable support and guidance throughout this project.

Thank you to James Morrison and Nigel Morrison for all of the discussions and ideas.

A special thanks to Steven Volschenk whose observation skills contributed greatly to the success of this project.

Table of contents

Declaration.....	ii
Abstract.....	iii
Dedication.....	iv
Table of contents.....	vi
List of tables.....	x
List of figures.....	xiii
Nomenclature.....	xix
Nomenclature.....	xix
1. Introduction.....	1
1.1 Compaction of tungsten carbide cobalt powder	1
1.2 Powder Industries	1
1.3 PEG & shrinkage related problems	2
1.4 Specific aims of the project	2
2. Literature review and background knowledge.....	4
2.1 Review of previous work and available literature	4
2.1.1 Previous work on the problem at Powder Industries	4
2.1.2 Mechanical and hydraulic press documents	5
2.1.3 Published papers and articles.....	5
2.2 Background knowledge on the manufacturing processes.....	6
2.2.1 Tungsten carbide-cobalt products.....	6
2.2.2 Definition of shrinkage	6
2.2.3 Negative and positive inserts	7
2.2.4 Pressing powder and powder grades.....	8
2.2.5 Milling & spray drying	9
2.2.6 Pressing.....	10
2.2.7 Sintering.....	11
2.2.8 Wax and PEG	12
3. Methods, test facilities and materials.....	13
3.1 Experimental methods	13
3.1.1 Pressing experiment description and aims.....	13
3.1.2 Simulation of shrinkage.....	14
3.1.3 Pressing experiment procedure.....	16

3.1.4 Positive insert pressing tests	19
3.1.5 Standard pressing experiment test sheet	20
3.1.6 Apparent density measurement of test powder samples.....	26
3.1.7 Screen fractioning of test powder samples	27
3.1.8 Inspection of sintered test pieces	28
3.2 Test equipment.....	29
3.2.1 Pressing equipment.....	29
3.2.2 Sinter-HIP furnace	29
3.2.3 Powder analysis equipment	30
3.2.4 Mounting and polishing equipment	31
3.3 Materials	33
3.3.1 Powders tested	33
3.3.2 Grade data tables.....	33
4. Experimental results and discussion	43
4.1 Layout for presentation of experimental results	43
4.2 Experimental data results.....	44
4.2.1 Grade ‘230’ experimental data	45
4.2.2 Grade ‘AP20’ experimental data	47
4.2.3 Grade ‘CN10’ experimental data.....	49
4.2.4 Grade ‘CR10’ experimental data	51
4.2.5 Grade ‘H1P’ experimental data	53
4.2.6 Grade ‘H6’ experimental data	55
4.2.7 Grade ‘K15M’ experimental data.....	57
4.2.8 Grade ‘MT16’ experimental data	59
4.2.9 Grade ‘MZ15’ experimental data	61
4.2.10 Grade ‘N6 / 194’ experimental data	63
4.2.11 Grade ‘P7’ experimental data	65
4.2.12 Grade ‘P25M’ experimental data.....	67
4.2.13 Grade ‘P30M’ experimental data.....	69
4.2.14 Grade ‘P40M’ experimental data.....	71
4.2.15 Grade ‘P511’ experimental data	73
4.2.16 Grade ‘PBF’ experimental data	75
4.2.17 Grade ‘PBT’ experimental data	77
4.2.18 Grade ‘PC20’ experimental data	79

4.2.19 Grade 'PI15' experimental data.....	81
4.2.20 Grade 'PI25' experimental data.....	83
4.2.21 Grade 'PI-40' experimental data	85
4.2.22 Grade 'Q3' experimental data	87
4.2.23 Grade 'S9' experimental data	89
4.3 Positive insert test data	91
4.4 Summary and discussion of results.....	93
4.4.1 Pressing and inspection of green	93
4.4.2 Test powder physical properties	94
4.4.3 Porosity of sintered components.....	95
4.4.4 Apparent density and ideal green density	96
4.4.5 Derivation of shrinkage equation.....	100
4.4.6 Positive insert considerations	103
4.4.7 Limitations of the ideal density vs. green density model	103
4.4.8 Validating the equation – cutting insert and shim tool production.....	103
5. Conclusions.....	105
5.1 Recommendations for further work.....	106
6. References.....	107
Appendices.....	109
Appendix 1.....	110
A1.1 Experimental data sheets for grade '230'	110
A1.2 Experimental data sheets for grade 'AP20'	114
A1.3 Experimental data sheets for grade 'CN10'	118
A1.4 Experimental data sheets for grade 'CR10'	122
A1.5 Experimental data sheets for grade 'H1P'	126
A1.6 Experimental data sheets for grade 'H6'	130
A1.7 Experimental data sheets for grade 'K15M'	133
A1.8 Experimental data sheets for grade 'MT16'	137
A1.9 Experimental data sheets for grade 'MZ15'	141
A1.10 Experimental data sheets for grade 'N6/194'	145
A1.11 Experimental data sheets for grade 'P7'	149
A1.12 Experimental data sheets for grade 'P25M'	153
A1.13 Experimental data sheets for grade 'P30M'	157
A1.14 Experimental data sheets for grade 'P40M'	161

A1.15 Experimental data sheets for grade ‘P511’	165
A1.16 Experimental data sheets for grade ‘PBF’	169
A1.17 Experimental data sheets for grade ‘PBT’	173
A1.18 Experimental data sheets for grade ‘PC20’	177
A1.19 Experimental data sheets for grade ‘PI-15’	181
A1.20 Experimental data sheets for grade ‘PI-25’	185
A1.21 Experimental data sheets for grade ‘PI-40’	189
A1.22 Experimental data sheets for grade ‘Q3’	193
A1.23 Experimental data sheets for grade ‘S9’	196
Appendix 2.....	200
A2.1 – Negative and positive insert tests using ‘CR10’ grade powder	200
A2.2 - Negative and positive insert tests using ‘PI-25’ grade powder	202
A2.3 - Negative and positive insert tests using ‘P40M’ grade powder	204
A2.4 - Negative and positive insert tests using ‘K15M’ grade powder.....	206
Appendix 3.....	208
A3.1 Porosity reports for sintered test pieces	208
A3.2 Company micrographs to evaluate porosity	210
A3.2 Company micrographs to evaluate porosity	211

List of tables

Table 3.1 – List of powders tested (sorted in alphabetical order by grade name)	34
Table 3.2 – Grade ‘230’ summary	35
Table 3.3 – Grade ‘AP20’ summary	35
Table 3.4 – Grade ‘CN10’ summary	36
Table 3.5 – Grade ‘CR10’ summary	36
Table 3.6 – Grade ‘H1P’ summary	36
Table 3.7 – Grade ‘H6’ summary	37
Table 3.8 – Grade ‘K15M’ summary	37
Table 3.9 – Grade ‘MT16’ summary	37
Table 3.10 – Grade ‘MZ15’ summary	38
Table 3.11 – Grade ‘N6/194’ summary	38
Table 3.12 – Grade ‘P7’ summary	38
Table 3.13 – Grade ‘P25M’ summary	39
Table 3.14 - Grade ‘P30M’ summary	39
Table 3.15 – Grade ‘P40M’ summary	39
Table 3.16 – Grade ‘P511’ summary	40
Table 3.17 - Grade ‘PBF’ summary	40
Table 3.18 – Grade ‘PBT’ summary	40
Table 3.19 – Grade ‘PC20’ summary	41
Table 3.20 - Grade ‘PI-15’ summary	41
Table 3.21 – Grade ‘PI-25’ summary	41
Table 3.22 – Grade ‘PI-40’ summary	42
Table 3.23 - Grade ‘Q3’ summary	42
Table 3.24 – Grade ‘S9’ summary	42
Table 4.2 - Grade ‘230’ experimental data summary	45
Table 4.3 – Pressing test results for grade ‘230’	45
Table 4.4 – Screen fraction report for grade ‘230’ test powder (granule sizes).....	45
Table 4.5 - Grade AP20 summary	47
Table 4.6 – Pressing test results for grade ‘AP20’	47
Table 4.7 – Screen fraction report for grade ‘AP20’ test powder (granule sizes)	47
Table 4.8 - Grade ‘CN10’ summary	49
Table 4.9 – Pressing test results for grade ‘CN10’	49

Table 4.10 – Screen fraction report for grade ‘CN10’ test powder (granule sizes)	49
Table 4.11 - Grade ‘CR10’ summary	51
Table 4.12 – Pressing test results for grade ‘CR10’	51
Table 4.13 – Screen fraction report for grade ‘CR10’ test powder (granule sizes)	51
Table 4.14 - Grade ‘H1P’ summary	53
Table 4.15 – Pressing test results for grade ‘H1P’	53
Table 4.16 – Screen fraction report for grade ‘H1P’ test powder (granule sizes)	53
Table 4.17 - Grade ‘H6’ summary	55
Table 4.18 – Pressing test results for grade ‘H6’	55
Table 4.19 – Screen fraction report for grade ‘H6’ test powder (granule sizes).....	55
Table 4.20 - Grade ‘K15M’ summary.....	57
Table 4.21 – Pressing test results for grade ‘K15M’	57
Table 4.22 – Screen fraction report for grade ‘K15M’ test powder (granule sizes)	57
Table 4.23 - Grade ‘MT16’ summary	59
Table 4.24 – Pressing test results for grade ‘MT16’	59
Table 4.25 – Screen fraction report for grade ‘MT16’ test powder (granule sizes).....	59
Table 4.26 - Grade ‘MZ15’ summary	61
Table 4.27 – Pressing test results for grade ‘MZ15’	61
Table 4.28 – Screen fraction report for grade ‘MZ15’ test powder (granule sizes).....	61
Table 4.29 - Grade ‘N6 / 194’ summary	63
Table 4.30 – Pressing test results for grade ‘N6 / 194’	63
Table 4.31 – Screen fraction report for grade ‘N6 / 194’ test powder (granule sizes)...	63
Table 4.32 - Grade ‘P7’ summary	65
Table 4.33 – Pressing test results for grade ‘P7’	65
Table 4.34 – Screen fraction report for grade ‘N6 / 194’ test powder (granule sizes)...	65
Table 4.35 - Grade ‘P25M’ summary	67
Table 4.36 – Pressing test results for grade ‘P25M’	67
Table 4.37 – Screen fraction report for grade ‘P25M’ test powder (granule sizes).....	67
Table 4.38 - Grade ‘P30M’ summary	69
Table 4.39 – Pressing test results for grade ‘P30M’	69
Table 4.40 – Screen fraction report for grade ‘P30M’ test powder (granule sizes).....	69
Table 4.41 - Grade ‘P40M’ summary	71
Table 4.42 – Pressing test results for grade ‘P40M’	71
Table 4.43 – Screen fraction report for grade ‘P40M’ test powder (granule sizes).....	71

Table 4.44 - Grade ‘P511’ summary	73
Table 4.45 – Pressing test results for grade ‘P511’	73
Table 4.46 – Screen fraction report for grade ‘P511’ test powder (granule sizes)	73
Table 4.47 - Grade ‘PBF’ summary	75
Table 4.48 – Pressing test results for grade ‘PBF’	75
Table 4.49 – Screen fraction report for grade ‘PBF’ test powder (granule sizes)	75
Table 4.50 - Grade ‘PBT’ summary	77
Table 4.51 – Pressing test results for grade ‘PBT’	77
Table 4.52 – Screen fraction report for grade ‘PBT’ test powder (granule sizes)	77
Table 4.53 - Grade ‘PC20’ summary	79
Table 4.54 – Pressing test results for grade ‘PC20’	79
Table 4.55 – Screen fraction report for grade ‘PC20’ test powder (granule sizes).....	79
Table 4.56 - Grade ‘PI15’ summary	81
Table 4.57 – Pressing test results for grade ‘PI15’	81
Table 4.58 – Screen fraction report for grade ‘PI15’ test powder (granule sizes)	81
Table 4.59 - Grade ‘PI25’ summary	83
Table 4.60 – Pressing test results for grade ‘PI25’	83
Table 4.61 – Screen fraction report for grade ‘PI25’ test powder (granule sizes)	83
Table 4.62 - Grade ‘PI40’ summary	85
Table 4.63 – Pressing test results for grade ‘PI40’	85
Table 4.64 – Screen fraction report for grade ‘PI40’ test powder (granule sizes)	85
Table 4.67 - Grade ‘Q3’ summary	87
Table 4.68 – Pressing test results for grade ‘Q3’	87
Table 4.69 – Screen fraction report for grade ‘Q3’ test powder (granule sizes).....	87
Table 4.70 - Grade ‘S9’ summary	89
Table 4.71 – Pressing test results for grade ‘S9’	89
Table 4.72 – Screen fraction report for grade ‘S9’ test powder (granule sizes)	89
Table 4.73 – Best pressing results for negative inserts	92
Table 4.74 – Best pressing results for positive inserts	92

List of figures

Figure 2.1 – A component with shrinkage of 20% - the sintered dimensions are the references and the green dimensions are larger by 20%.....	7
Figure 2.2 – A ‘negative’ insert with parallel edges and ‘positive’ insert with edges that are not parallel	8
Figure 2.3 – Diagram showing powder compaction using punches and a die cavity	10
Figure 3.1 – Pressing experiment procedure.....	13
Figure 3.2 – Green negative insert shape and dimensions (in mm).....	15
Figure 3.3 – Relationship between shrinkage and green density of WC-Co as obtained from previous experiments at Powder Industries [1].....	15
Figure 3.4 – Compacts (bottom) and corresponding sintered pieces (top) of a grade arranged in order from hardest to softest (from left to right) ready for inspection.	17
Figure 3.5 – Inspection of green compacts under a stereo-microscope	17
Figure 3.6 – Green compacts and sintered components for all the powder grades tested	19
Figure 3.7 – Green positive insert shape and dimensions (in mm).....	20
Figure 3.8 – Standard pressing test sheet.....	25
Figure 3.9 – Measured 25 cm ³ and weighed test powder sample in an air-tight container.	27
Figure 3.10 – Histogram plot of typical powder granule size distribution	28
Figure 3.11 – Mounted and polished sintered test pieces	28
Figure 3.12 – Dorst TPA15HS Hydraulic CNC press	29
Figure 3.13 – Ultra temp sinter-HIP furnace	30
Figure 3.14 – Digital scale and Hall flow meter funnels	30
Figures 3.15 & 3.16 – The screen stack loaded into the shaker and the sieves used for screen fractioning the test powder samples.	31
Figure 3.17 – Leco mounting press.....	31
Figure 3.18 – Struers grinding and polishing equipment.....	32
Figure 3.19 – Nikon microscope used to check for porosity	32
Figure 4.1 – Measured granule size distribution of test powder sample of grade 230 (apparent density of 3.64 g/cm ³).....	46
Figure 4.2 – Historical granule size distribution of grade 230 (historical apparent density of 3.70 g/cm ³)	46

Figure 4.3 – 100x magnification image of polished section of the best shrinkage sample (least porosity): 21.4% and the worst shrinkage (most porosity) sample: 23.7% .. 46	
Figure 4.4 – Measured granule size distribution of test powder sample of grade AP20 (apparent density of 3.42 g/cm ³)..... 48	
Figure 4.5 – Historical granule size distribution of grade AP20 (historical apparent density of 3.44 g/cm ³)..... 48	
Figure 4.6 – 100x magnification image of polished section of the best shrinkage sample (least porosity) 22.1% and the worst shrinkage (most porosity) 24.4% for AP20 48	
Figure 4.7 – Measured granule size distribution of test powder sample of grade CN10 (apparent density of 3.18 g/cm ³)..... 50	
Figure 4.8 – Historical granule size distribution of grade CN10 (historical apparent density of 3.29 g/cm ³)..... 50	
Figure 4.9 – 100x magnification image of polished section of the best shrinkage (least porosity) 24.1 % and the worst shrinkage (most porosity) 26.5 % for grade CN1050	
Figure 4.10 – Measured granule size distribution of test powder sample of grade CR10 (apparent density of 3.26 g/cm ³)..... 52	
Figure 4.11 – Historical granule size distribution of grade CR10 (historical apparent density of 3.28 g/cm ³)..... 52	
Figure 4.12 – 100x magnification image of polished section of the best shrinkage (least porosity) 24.0 % and the worst shrinkage (most porosity) 25.5 % for grade CR1052	
Figure 4.13 – Measured granule size distribution of test powder sample of grade H1P (apparent density of 3.25 g/cm ³)..... 54	
Figure 4.14 – Historical granule size distribution of grade H1P (historical apparent density of 3.21 g/cm ³)..... 54	
Figure 4.15 – 100x magnification image of polished cross-section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 25 % for grade H1P54	
Figure 4.16 – Measured granule size distribution of test powder sample of grade H6 (apparent density of 2.87 g/cm ³)..... 56	
Figure 4.17 – Historical granule size distribution of grade H6 (historical apparent density of 3.41 g/cm ³) 56	
Figure 4.18 – 100x magnification image of polished cross-section of the best shrinkage (least porosity) 23.5 % and the worst shrinkage (most porosity) 25.5 % for grade H6 56	

Figure 4.19 – Measured granule size distribution of test powder sample of grade K15M (apparent density of 3.12 g/cm ³).....	58
Figure 4.20 – Historical granule size distribution of grade K15M (historical apparent density of 3.19 g/cm ³).....	58
Figure 4.21 – 100x magnification image of polished cross-section of the best shrinkage (least porosity) 23 % and the worst shrinkage (most porosity) 25 % for grade K15M	58
Figure 4.22 –Measured granule size distribution of test powder sample of grade MT16 (apparent density of 3.43 g/cm ³).....	60
Figure 4.23 – Historical granule size distribution of grade MT16 (historical apparent density of 3.37 g/cm ³).....	60
Figure 4.24 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % and the worst shrinkage (most porosity) 24 % for grade MT16	60
Figure 4.25 – Measured granule size distribution of test powder sample of grade MZ15 (apparent density of 3.37 g/cm ³).....	62
Figure 4.26 – Historical granule size distribution of grade MZ15 (historical apparent density of 3.31 g/cm ³).....	62
Figure 4.27 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % and the worst shrinkage (most porosity) 23 % for grade MZ15	62
Figure 4.28 – Measured granule size distribution of test powder sample of grade N6 / 194 (apparent density of 3.62 g/cm ³).....	64
Figure 4.29 – Historical granule size distribution of grade N6 / 194 (historical apparent density of (3.72 g/cm ³).....	64
Figure 4.30 – 100x magnification image of polished section of the best shrinkage (least porosity) 22.5 % and the worst shrinkage (most porosity) 24 % for grade N6 / 19464	
Figure 4.31 – Measured granule size distribution of test powder sample of grade P7 (apparent density of 3.38 g/cm ³).....	66
Figure 4.32 – Historical granule size distribution of grade P7 (historical apparent density of 3.72 g/cm ³)	66
Figure 4.33 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % and the worst shrinkage (most porosity) 23 % for grade P7	66
Figure 4.34 – Measured granule size distribution of test powder sample of grade P25M (apparent density of 3.01 g/cm ³).....	68

Figure 4.35 – Historical granule size distribution of grade P25M (historical apparent density of 3.14 g/cm ³).....	68
Figure 4.36 – 100x magnification image of polished section of the best shrinkage (least porosity) 20 % and the worst shrinkage (most porosity) 24 % for grade P25M	68
Figure 4.37 – Measured granule size distribution of test powder sample of grade P30M (apparent density of 2.91 g/cm ³).....	70
Figure 4.38 – Historical granule size distribution of grade P30M (historical apparent density of 3.09 g/cm ³).....	70
Figure 4.39 – 100x magnification image of polished section of the best shrinkage (least porosity) 20 % and the worst shrinkage (most porosity) 24 % for grade P30M	70
Figure 4.40 – Measured granule size distribution of test powder sample of grade P40M (apparent density of 3.09 g/cm ³).....	72
Figure 4.41 – Historical granule size distribution of grade P40M (historical apparent density of 3.26 g/cm ³).....	72
Figure 4.42 – 100x magnification image of polished section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 24 % for grade P40M	72
Figure 4.43 – Measured granule size distribution of test powder sample of grade P511 (apparent density of 3.41 g/cm ³)	74
Figure 4.44 – Historical granule size distribution of grade P511 (historical apparent density of 3.53 g/cm ³).....	74
Figure 4.45 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % (left) and the worst shrinkage (most porosity) 24 % for grade P51174	
Figure 4.46 – Measured granule size distribution of test powder sample of grade PBF (apparent density of 3.86 g/cm ³).....	76
Figure 4.47 – Historical granule size distribution of grade PBF (historical apparent density of 3.70 g/cm ³).....	76
Figure 4.48 – 100x magnification image of polished section of the best shrinkage (least porosity) 21.5 % and the worst shrinkage (most porosity) 24 % for grade PBF	76
Figure 4.49 – Measured granule size distribution of test powder sample of grade PBT (apparent density of 3.88 g/cm ³).....	78
Figure 4.50 – Historical granule size distribution of grade PBT (historical apparent density of 3.84 g/cm ³).....	78
Figure 4.52 – Measured granule size distribution of test powder sample of grade PC20 (apparent density of 3.55 g/cm ³).....	80

Figure 4.54 – 100x magnification image of polished section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 23 % for grade PC20	80
Figure 4.55 – Measured granule size distribution of test powder sample of grade PI15 (apparent density of 3.40 g/cm ³).....	82
Figure 4.56 – Historical granule size distribution of grade PI15 (historical apparent density of 3.52 g/cm ³).....	82
Figure 4.57 – 100x magnification image of polished section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 24 % for grade PI15.....	82
Figure 4.58 – Measured granule size distribution of test powder sample of grade PI25 (apparent density of 3.37 g/cm ³).....	84
Figure 4.59 – Historical granule size distribution of grade PI25 (historical apparent density of 3.53 g/cm ³).....	84
Figure 4.60 – 100x magnification image of polished section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 24 % for grade PI25.....	84
Figure 4.61 – Measured granule size distribution of test powder sample of grade PI40 (apparent density of 3.27 g/cm ³).....	86
Figure 4.62 – Historical granule size distribution of grade PI40 (historical apparent density 3.40 g/cm ³)	86
Figure 4.63 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % and the worst shrinkage (most porosity) 25 % for grade PI40.....	86
Figure 4.64 – Measured granule size distribution of test powder sample of grade Q3 (apparent density of 3.13 g/cm ³).....	88
Figure 4.65 – Historical granule size distribution of grade Q3 (historical apparent density of g/cm ³)	88
Figure 4.66 – 100x magnification image of polished section of the best shrinkage (least porosity) 22.5 % and the worst shrinkage (most porosity) 24 % for grade Q3	88
Figure 4.67 – Measured granule size distribution of test powder sample of grade S9 (apparent density of 3.20 g/cm ³).....	90
Figure 4.68 – Historical granule size distribution of grade S9 (historical apparent density of 3.25 g/cm ³)	90
Figure 4.69 – 100x magnification image of polished section of best shrinkage (least porosity) 19 % and the worst shrinkage (most porosity) 23 % for grade S9	90
Figure 4.70 – General trend of ideal green density vs. apparent density for all powders tested (data from the tables in section 4.2)	96

Figure 4.71 – Historical granule size distribution (standard) for all grades.	98
Figure 4.72 – Ideal green density vs. apparent density	99
Figure 4.73 – Examples of successful new components made due to the research project. The components with central holes are examples of cutting tool inserts, the remaining component is a mining insert.	104

Nomenclature

Apparent density – This is the poured density of a powder. The powder is non-tapped and is poured into a container from a fixed height using a Hall flow meter funnel

Granules – The individual spheres of WC-Co and lubricant that make up a powder. Granule sizes typically vary from 63 μm to 300 μm

Green compact - Component that has been pressed to shape and is still in the “soft” stage prior to sintering.

Green density - The density of a component before sintering but after being pressed to shape.

Negative insert - A component where all edges are either parallel or perpendicular to each other.

Positive insert - This refers to a component where the edges in the vertical plane of the component are not parallel i.e. there is an angle greater than zero degrees.

Powder grade – This indicates a WC-Co powder with a specific makeup of constituents of Co content, mixed crystal content and WC grain size.

S.D. – This is the sintered density of a component.

Shrinkage – This is the percentage that a dimension varies in a linear direction from the sintered size to compact size. This is usually measured in 3 dimensions and an average taken as the overall shrinkage.

1. Introduction

1.1 Compaction of tungsten carbide cobalt powder

WC-Co cutting inserts are manufactured from WC and Co powder that is compacted to a specific shape and then sintered. During sintering, the compact or 'green' component shrinks by a certain percentage to a final size. To accommodate this shrinkage, compaction tooling is made larger than the desired sintered dimensions by a percentage equal to the shrinkage.

Therefore, a main pressing problem is to know the correct shrinkage when designing the compaction tooling. If the correct shrinkage is obtained, it would mean that the applied pressure was in the ideal range. This would have the added benefit of significantly reducing tool wear and tool breakage.

1.2 Powder Industries

The project was motivated by a problem of a South African WC-Co manufacturer, Powder Industries. Powder Industries is a South African company that produces hardmetal (WC-Co) products such as cutting inserts, wear parts and mining components. The core business is mainly wear and mining WC-Co bits with an ever increasing focus on cutting inserts.

At Powder Industries, raw materials such as WC, Cobalt and PEG (Polyethylene glycol – used as a lubricant) are mixed, milled and spray-dried and from this a ready-to-press WC-Co powder is produced. This powder is compacted and subsequently sintered to final sized components. All of the stages from powder preparation to compaction and to sintering occur within the company. Compaction tooling used by Powder Industries to produce green compacts (ready for sintering) is manufactured in-house and is also made from WC-Co. This tooling (die cavity and punches) is also designed in-house.

In recent times there has been an increasing need at Powder Industries to develop the cutting insert product range as well as to develop new designs therefore there has been an increasing need to design and manufacture new compaction tooling.

1.3 PEG & shrinkage related problems

In order to aid pressing and reduce friction during compaction, a pressing lubricant is pre-mixed in the WC-Co powder. Previously at Powder Industries, wax was used as the pressing lubricant but this has been changed to PEG in recent times.

When the change to PEG from wax was made, it was found that the tooling for pressing the compacts no longer worked as well as before. There were suddenly many incidences of tool breakages (punches chipping or breaking) as well as compact defects. Defects included cracks, de-laminations, and hard burrs on the compact edges as well as compacts that were under-pressed or over-pressed. Some compacts could not be pressed i.e. the pressures necessary for the desired green dimensions could not be attained.

In the days when wax was the lubricant used, the tooling was designed to accommodate a 20% shrinkage and this did work fairly well and with a variety of different types of tooling. After the introduction of PEG it was apparent that the shrinkage had changed but by how much and what the correct shrinkage should be when designing new tools was unknown.

If there is a mismatch between the actual shrinkage of a compact and the shrinkage accommodation designed into the tooling, the pressure during compaction will turn out to be too high or too low.

1.4 Specific aims of the project

The scope of this research project is to determine through experimentation with various grades of powder (i.e. powders of different compositions), if a method or equation can be derived to find the correct shrinkage for WC-Co compacts, ending the “trial and error” approach applied at Powder Industries since the introduction of PEG.

The tooling designer has much information available to him such as the composition of the powder grade, its required sintered density, its volatile content, its physical properties and the mass of the sintered component. What the designer doesn't know is

what the ideal green density of the compact should be in order to obtain a pore-free sintered product, i.e. a product which shrunk optimally during sintering, and thus does not know what shrinkage value should be used in the design of tooling. This project aimed at providing this information.

An attempt has been made to write the dissertation in a style that can be understood by the practitioner in the hardmetal industry, so that the results can be used to the benefit of the industry.

2. Literature review and background knowledge

2.1 Review of previous work and available literature

2.1.1 Previous work on the problem at Powder Industries

Prior to the start of this research project, some work was carried out by a colleague that was formerly employed by Powder Industries (the sponsor of this project) to try and understand the impact of shrinkage on tooling design [1]. It was known that the components being pressed were often pressed incorrectly. It was observed that sometimes too much pressure was being recorded during pressing and the compact was over-pressed. At other times, the pressure being recorded was too low and the compact was under-pressed. This happened when the same tooling was being used with different grades of powder.

If the compaction tooling has been designed with the correct shrinkage, a green compact will shrink by the correct amount so that the sintered component dimensions are to the required specification. If a compact shrinks too much, it will be undersized in the sintered state. To compensate for a component shrinking too much, slightly more powder is added during the compaction step and pressing is done to the same volume as before – effectively increasing the green density of the compact. Because the green density has been increased, the component will shrink less than before [15] and the final sintered dimensions will be to specification. This however results in higher pressing forces being applied. The higher pressures result in a higher chance of tool breakage as well as an increase in the rate of tool wear.

New tooling was manufactured but this was based on a trial and error process. If a new component was to be pressed, the amount of shrinkage that the compact would undergo during sintering was essentially a guess and the tooling was designed with this guessed shrinkage in mind. If the guess was incorrect, then the tooling would be manufactured again based on a new guess. This was a very long and costly process in the design of new components and tooling. It was established from this previous work [1] that the linear shrinkage range for a component was from 20% to 25% (depending on the powder grade) and at least the limits of guessing a shrinkage value were set.

At the onset of this project, it was still not known if every grade had a unique ideal shrinkage that needed to be taken into account during tooling design.

2.1.2 Mechanical and hydraulic press documents

In the pressing department of the company, there are two main types of presses that are used to compact WC-Co powder. These are both mechanical and hydraulic presses and there are extensive user manuals and compaction notes for this machinery. The user manuals and notes were reviewed but as with the company literature, the information available tells one how to use the machine but there is no specific information on the green density that needs to be achieved in order to press an ideal compact.

2.1.3 Published papers and articles

A survey of the available literature showed that most of the material available to aid in the design of tooling with regards to shrinkage is not directly relevant to the aims of this project. [e.g. 2 to 14]. The papers reviewed deal mostly with modeling and simulation of compaction and were not of assistance to this project because its aim was to optimize compaction experimentally, i.e. through systematic compression tests.

An overview of papers and publications revealed that very little information is available on predicting shrinkage for compaction tooling by using known powder properties for cemented carbides. The compaction simulation models described in the papers [4,7,8,9,10,11,12,13] help predict what happens during compaction theoretically but no method to obtain specific applicable solutions could be found. This kind of information is proprietary, i.e. already be known within companies but is not published, at least in cemented carbides.

In books and texts on powder metallurgy, shrinkage of cemented carbides is not dealt with specifically [15,18] . Other powdered materials such as steel, which are described in books shrink much less than WC-Co and their compaction is not a problem of the same magnitude as it is for WC-Co.

2.2 Background knowledge on the manufacturing processes

2.2.1 Tungsten carbide-cobalt products

Tungsten carbide-cobalt or cemented carbide (also known as hard metal) is a material that has been used in the manufacture of cutting tools since the 1920's and today is still a very important material in the cutting tool industry and is one of the only materials available with the mechanical properties required to handle a broad range of cutting conditions. The mechanical properties of tungsten carbide-cobalt, a combination very high hardness and toughness as well as resistance to heat and wear make it ideal for cutting and wear applications. [15]

It is economical to produce large volumes of carbide products as high tolerances and near net shape can be achieved with a minimum amount of machining and finishing processes. In the manufacture of tungsten carbide-cobalt or hardmetal products by powder metallurgy, there is a series of steps that are followed: preparation of powder, spray drying, pressing to shape and sintering. After sintering, there are additional steps necessary such as grinding and cleaning as many of the cutting inserts produced require coating.

The basic constituents of hardmetal powder are WC, a binder such as Cobalt and in some cases, mixed crystals such as TiC, TaC or NbC to enhance hardness and heat resistance. Mixed crystals are usually added to grades of powder known as 'tool grades' and these are used in the manufacture of high quality cutting inserts [16]. High quality cutting inserts are inserts that perform consistently with a good tool life and low instances of breakage. In the case of cemented carbides, this means inserts with very low or no porosity.

At Powder Industries, the types of WC-Co products produced include components for the mining industry, various types of wear parts and cutting inserts that are used in milling and turning processes.

2.2.2 Definition of shrinkage

Shrinkage is the percentage difference in size between a sintered component and the same component in its green or pre-sintered state (Please refer to pg xviii –

Nomenclature) which is caused by the closing of porosity during sintering [15]. The shrinkage percentage is referenced from the sintered size, thus 20% shrinkage indicates that the green component is 20% larger dimensionally than the sintered component (this is a common convention in the cemented carbide industry). The definition of shrinkage varies in other companies where for example, the reference point is the green component and not the sintered component. This standard at Powder Industries can be misleading as a '20% linear shrinkage' would lead one to conclude that a compact shrinks by 20% from the green to sintered state. This is not the case and it is important to remember that when describing shrinkage or referring to a shrinkage percentage, it is the percentage amount linearly, that the green compact is larger by, when compared to the same component in the sintered state.

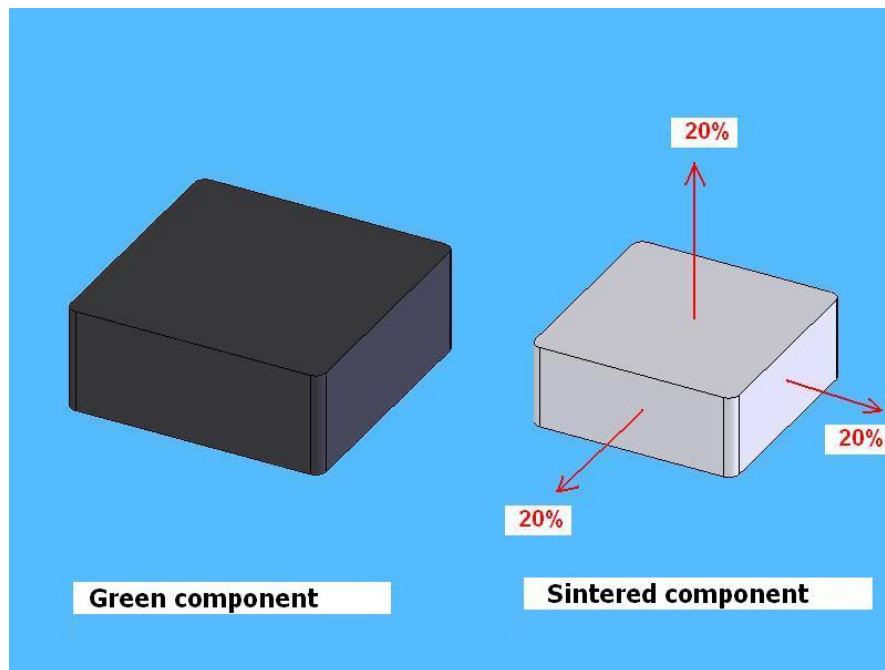


Figure 2.1 – A component with shrinkage of 20% - the sintered dimensions are the references and the green dimensions are larger by 20%

2.2.3 Negative and positive inserts

Among the products manufactured by Powder Industries, there are two main categories of cutting inserts, namely 'negative' or 'positive' inserts. When looking at an insert from a side view, a 'negative' insert means that the edges of the insert are parallel to each other. A 'positive' insert has sides that are not parallel – the sides from an angle

greater than 0° but less than around 25° from the vertical plane. Both 'negative' and 'positive' inserts have been included in this investigation because they present different compaction problems, since acute angles are more difficult to fill thoroughly with powder than right angles.

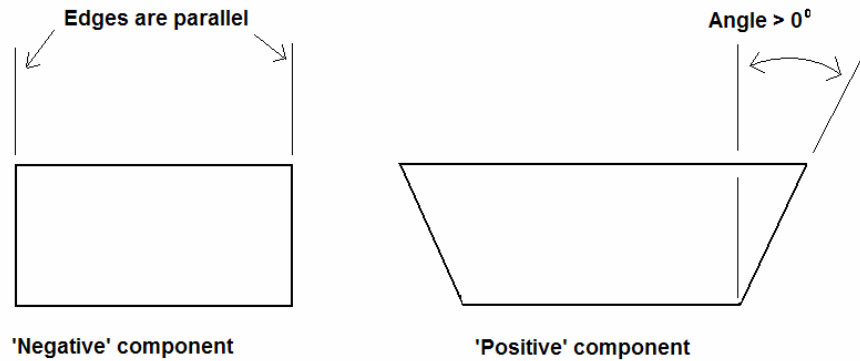


Figure 2.2 – A 'negative' insert with parallel edges and 'positive' insert with edges that are not parallel

2.2.4 Pressing powder and powder grades

Tungsten carbide-cobalt products are produced by compaction of powder – which is then sintered to a final size. This powder consists of WC particles (of a specified size range), cobalt which acts as the binder and a pressing lubricant. In some cases, further additions of mixed crystals are made to give specific properties to a product. These mixed crystals are usually titanium carbide (TiC), tantalum carbide (TaC) or niobium carbide (NbC) [16].

There are 23 main grades of tungsten carbide powder used at Powder Industries to manufacture various types of products. These grades are differentiated from each other by amount of cobalt, WC grain size variations and additions of mixed crystals. All of the grades however, have an almost constant amount of pressing lubricant - approximately 2 - 2.5 wt % - which is the smallest amount of lubricant that allows complete coverage of the powder particles [18]. Pressing lubricant is used to reduce friction between the powder granules and the die cavity that shapes the compact as well as to allow the granules to hold after pressing. If no pressing lubricant was used, the component would generally break apart after pressing.

There is a large variety of tungsten carbide-cobalt products manufactured at Powder Industries and the uses for these products are quite diverse. In the manufacture of these various types of components, different grades of tungsten carbide-cobalt powder are used. For example, wear parts and mining components require high toughness and wear resistance and cutting inserts need to be very hard and rigid.

These different characteristics are achieved by varying the amount of cobalt and/or the grain size of the tungsten carbide powder. For example, a component requiring a very high toughness and impact resistance would have a high amount of cobalt - up to 20% - where a cutting insert would have typically around 6%. Varying the WC grain size will change the hardness and wear resistance properties of a component [18].

2.2.5 Milling & spray drying

A 'grade of powder' is prepared by mixing WC with cobalt and pressing lubricant. The particles of the grade powder are WC grains that are coated by fine particles of cobalt and by the lubricant. Homogenization of all powders is done by wet milling. This is achieved at Powder Industries by means of ball mills (the milling 'balls' in this case are cylindrical in shape and are made from tungsten carbide-cobalt). Milling does help control the average grain size to a certain extent and to even out the average particle size. The main result of milling is the thorough coating of the WC particles by the cobalt. The medium used in the milling process is alcohol – the purpose of the medium is to ensure optimum dispersion of the ingredients as well as to prevent oxidation from taking place through heating. The end result of the milling process is a homogenous slurry that is then transferred to the spray drier in order to dry and granulate the grade of powder. Granulation is important in automatic uniaxial compaction as the powder needs to fill a die cavity to the same volume and weight after each pressing cycle [18].

Spray drying is the process of drying and granulating the powder suspension obtained from the wet milling process. The process involves spraying the powder suspension under high pressure into a flow of nitrogen gas which causes rapid drying and the formation of spherical granules. The granule diameters and size distributions can be controlled by process parameters of the spray drying equipment such as the atomizing pressure and diameter of the spray nozzle. By optimising the spray drying process, a

granulated powder with good flow properties, uniform fill density and required particle size distribution can be obtained. After this process, the powder is ready to be pressed to shape by mechanical or hydraulic presses. [17]

2.2.6 Pressing

In order to produce cutting tool inserts, granulated grade powders are compacted or pressed to a net shape using a top and bottom punch and a die cavity. The powder is filled into the die cavity and uniaxial pressing i.e. pressing along a single direction, is carried out to achieve a specified shape and volume. In a hydraulic press, pressing occurs equally from the top punch and bottom punch (known as double-action pressing) but due to wall friction (between the powder granules and the cavity wall), a density gradient exists [15]. The density gradient is symmetrical in the component when double action pressing is used but due to this gradient, a 'neutral zone' or relatively low-density region exists in the middle of the component and the density increases in both directions heading away from this zone in the vertical direction. A pressing lubricant (proprietary waxes or PEG) is used to improve pressing and to help minimise the effects of wall friction and thus the density gradient. A consequence of the density gradient is distortion of the component during sintering, thus it is important to reduce friction during compaction as much as possible [15].

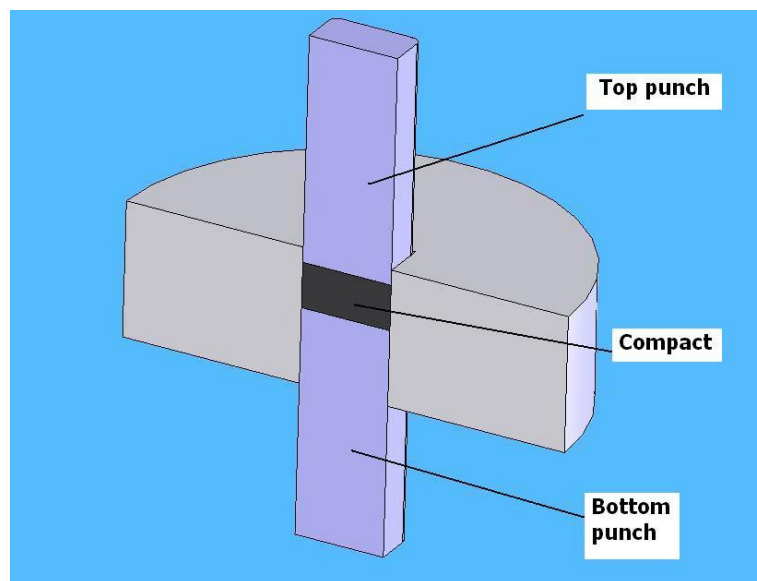


Figure 2.3 – Diagram showing powder compaction using punches and a die cavity

The compacted component is known as a 'green' component and it has adequate strength to be handled. The green components are however delicate and have a density of between 50% to 60% of final sintered density [18]. After pressing, the green components are visually inspected for physical defects such as cracking, chipping or burring and are then sent to the sintering furnaces.

The tooling i.e. the top and bottom punch as well as the die cavity is manufactured from tungsten carbide-cobalt as very high stiffness and dimensional stability is required for compacting hardmetal powder. Also, tungsten carbide-cobalt has exceptional wear resistance and this is important for a long tool life.

2.2.7 Sintering

Sintering is the final stage of the manufacturing process and is the step where the powder particles are bonded together and final densification occurs under high temperature where the temperature depends on the material being sintered but is always higher than the eutectic temperature of the cobalt binder (~ 1330°C). Sintering can be described as the heat treatment of a porous compact in order to change the properties towards the properties of a solid, pore-free body [10]. At Powder Industries, sintering is done in a sinter/HIP furnace – this is a furnace with an added high-pressure cycle – known as HIP or hot isostatic pressing. Hipping is done to improve the quality of the components and to remove any residual porosity by exposing the material to a highly pressurised inert gas (Argon at a pressure of 50-100 MPa). The sinter / HIP technology allows de-waxing or de-pegging (see 2.2.8), final sintering and secondary compaction to be done in a single cycle.

There are a number of important processes that occur during sintering [18]:

- The removal of pressing lubricant and other volatiles such as trapped air
- Desorption of moisture
- Reduction of Co, W and mixed carbide oxides
- Shrinkage of the components as densification takes place
- Grain growth through solution and re-precipitation of WC in and out of Co.

After sintering, the components are removed and sent for quality-control inspection and metallurgical property analysis. Sintered density or S.D. refers to WC-Co density after

the sintering cycle and is one of the properties measured in the laboratory after sintering [16].

2.2.8 Wax and PEG

Pressing lubricant, as discussed in section 2.2.4 and 2.2.6, is necessary to reduce friction during pressing and to aid in producing a high quality green compact with sufficient strength to facilitate easy handling. Previously at the Powder Industries (approximately 5-6 years ago), paraffin wax was the preferred lubricant and was used in all of the powder grades. It was decided to change the lubricant to PEG (polyethylene glycol) for reasons including better component quality and easier processing through the furnaces. Different lubricants lead to green compacts of different quality because their different composition and molecular structure lead to different lubrication ability. Wax also presented problems during sintering by ‘cracking’ (breaking down of carbon chains) and causing excess carbon to be present in the system [18]. This is undesirable as the delicate carbon balance in the tungsten carbide-cobalt may be adversely affected.

Another factor was that there was wax residue buildup in the furnace after many sintering runs and this was very difficult to clean – excess wax in the system would also break down during sintering and affect the product as described above.

PEG is easier to control through the furnaces during sintering and does not leave a residue behind. Generally, it is the experience of most hardmetal producers that the PEG lubricated compact quality (i.e. the ability of the compact to lead to pore-free sintered parts) was better than that of compacts which were made with wax as a lubricant. The trend of large international companies switching to PEG as a lubricant was also a motivator in keeping up with the latest advances in powder compaction.

It is generally known that the powder properties that most affect the compactibility (as well as the apparent density) of a powder are mean particle size, particle size distribution, particle surface roughness and particle shape [15].

3. Methods, test facilities and materials

3.1 Experimental methods

3.1.1 Pressing experiment description and aims

All of the testing and experimentation was carried out on equipment available at Powder Industries and done in conjunction with standard daily production runs. The focus of the research was on pressing and subsequent sintering of the test pieces so the majority of the work was carried out in the company pressing department and sintering department.

The aim of the pressing experiments was to press green compact (and then sinter) samples of each powder grade available, collect data (such as mass of compact and mass of sintered component, pressed compact dimensions, pressing forces, powder volatile content and powder apparent density) then analyse the collected information to try and determine a relationship between the properties of powder, green compact and sintered component. If some a general relationship would be found, then it would be possible to derive a general equation or method to predict shrinkage when designing tooling. ¹

Below, is a flow chart describing the experimental procedure for pressing green compact samples of a grade of powder (grades of powder vary from each other by Co wt% , average WC particle size and mixed carbide content) :

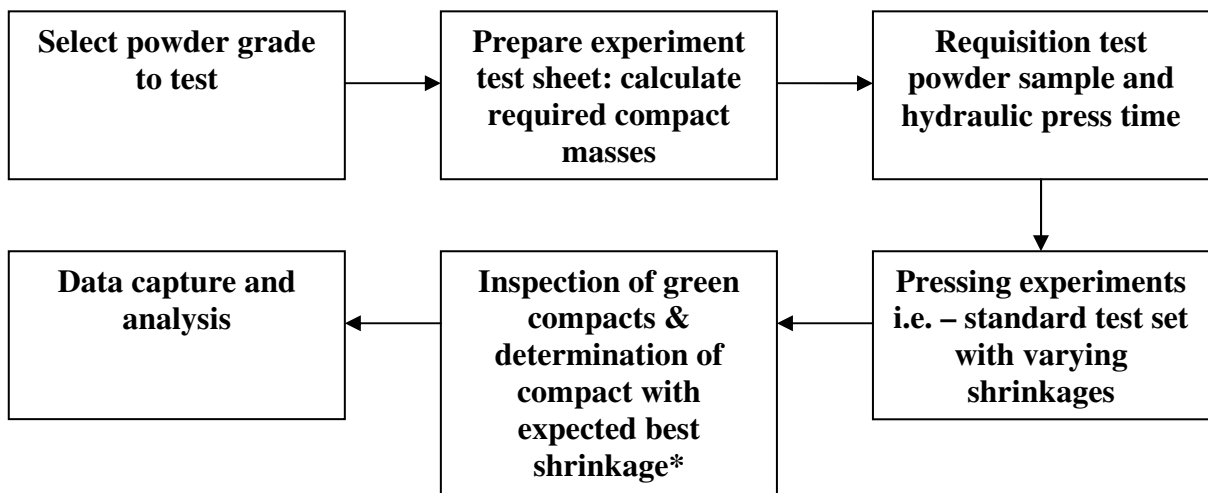


Figure 3.1 – Pressing experiment procedure

* the meaning of 'expected best' shrinkage is given in section 3.1.2.

For a pressing experiment for each specific grade, compacts were pressed in duplicate in order to keep a set of green compacts and to send another set for sintering. This was to compare a green component with a corresponding sintered component. A detailed explanation of the pressing procedure is in the following sections.

3.1.2 Simulation of shrinkage

In order to find a shrinkage that a certain grade undergoes, it is necessary to press a test piece, sinter it and then measure the actual shrinkage that the compact underwent. The linear shrinkage in each of the 3 dimensions is measured and the average of these three values is taken as the mean shrinkage.

It was known that the ideal shrinkage for a grade lay between 20% and 25% (from previous work) but the exact ideal shrinkage was unknown. To test the shrinkage of a grade of powder, it would be necessary to make tooling for every shrinkage that one would like to test for, for example in steps of 0.5% from 20% to 25%. The compacts pressed in the different tools would all shrink during sintering by various amounts (as per tool %) to the same final dimensions i.e. all of the green compact masses would be the same but due to differing expected shrinkages, the green volumes would vary. For the green volumes to vary, the recorded pressing pressure would have to vary accordingly. The green compacts would be inspected and the one that was pressed ‘the best’ would be selected. ‘The best’ can be selected by inspecting a green compact under a stereo-microscope. If a compact is under-pressed, imperfectly pressed granules can be seen. If a compact is over-pressed, cracks form and the compact appears very shiny. The best green density would correspond to a particular shrinkage which would correspond to the correct shrinkage for the grade of powder.

It is however expensive and impractical to manufacture many toolsets out of which only one would be correct. It was decided to use one standard toolset (with the green compact shape being a basic square) and to simulate varying shrinkages. This was done for every powder by pressing components of different masses to a set thickness (i.e. to a fixed volume) and thereby simulating different shrinkages by changing the green mass of each pressed component. Changing green mass at a fixed volume effectively changes the green density of the green compact.

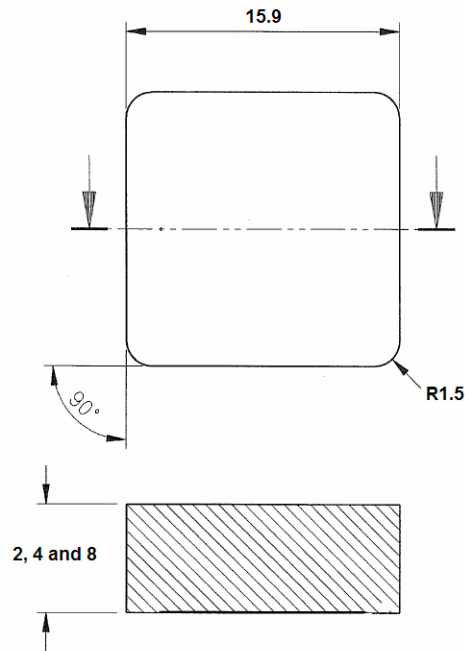


Figure 3.2 – Green negative insert shape and dimensions (in mm)

Since all of the green compacts in a set of pressing experiments were all pressed to a fixed volume but each compact weight differed by a fixed amount, the compacts each shrank by a different amount to a final size. It is important to note that the final sintered dimensions were different from component to component because the mass was different. In this way, it was possible to press a range of compacts from a grade of powder by using one toolset (two punches and a die cavity) and have different shrinkages. Figure 3.3 shows how the shrinkage is affected by varying the green mass of a component and thus changing its green density.

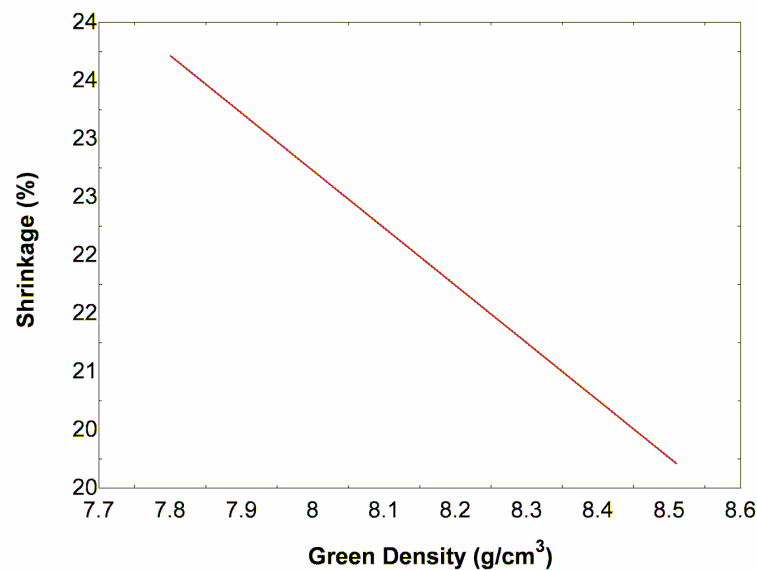


Figure 3.3 – Relationship between shrinkage and green density of WC-Co as obtained from previous experiments at Powder Industries [1]

3.1.3 Pressing experiment procedure

A grade of powder was selected. Each grade of powder had all of the information relevant to the powders stored on the company server, such as ideal S.D. of components sintered from each powder grade, apparent density and volatile content. A test sheet was then prepared and the required green weights needed to achieve a certain shrinkage when pressing at constant volume were calculated. (See section 3.1.5 – sample test sheet).

In order to change the amount of shrinkage that a component underwent the green weight was varied by calculated amounts keeping the pressing volume constant (Refer to section 3.1.5 for an example). This effectively changed the green density of a compact. The required sintered density of a component was constant but the final sintered volume varied due to the change in green weight. Each compact was pressed in duplicate in order to sinter one piece and then to compare the sintered dimensions to the green dimensions and to measure the actual shrinkage that a component underwent. The shrinkage was measured in three directions i.e. along the thickness as well as across the width in two directions. The average of the three measurements was then calculated to obtain the mean actual shrinkage of a component. The expected shrinkage for each successive component was changed in 0.5% steps (starting at 20%) and an average of 9 test pieces were pressed (over the shrinkage range up to 25%) for a grade of powder. The compact thickness was set to 4mm and the cross-sectional area to 250 mm².

The pressing experiment was repeated but with the thickness of the component set at half of the original 4mm and keeping the area constant since the ratio between thickness and width of a component affects the density distribution in the component [15], experiments were done to determine the effect of thickness variations in the present case.

After this, the experiment was repeated again but with component thickness set at double the original thickness (i.e. 8mm). Thus, for each grade of powder, there were three sets of pressings with a range of simulated shrinkages. Once an experimental run had been completed, the compacts were arranged in order from “the hardest” to “the softest pressing” i.e. lowest to highest shrinkage and prepared for inspection.



Figure 3.4 – Compacts (bottom) and corresponding sintered pieces (top) of a grade arranged in order from hardest to softest (from left to right) ready for inspection

The aim was to find the compact that was pressed best and therefore establish the best green density for a particular grade. To determine how well a compact had been pressed, visual inspection under a stereo-microscope was done (Refer to section 3.1.2). Inspecting green compacts under a microscope for defects is a standard quality control practice at the Powder Industries and is done by people with many years of experience in the carbide industry.



Figure 3.5 – Inspection of green compacts under a stereo-microscope

The compacts were inspected under the microscope with the help of an experienced inspector and graded according to how well they were pressed. The results were recorded. Each component was marked according to target shrinkage: for example, a

component pressed with CR10 grade powder and desired shrinkage of 24% would be marked “CR10-24”.

Initially, the aim of varying the component thickness was to determine if this would have an impact on ‘best’ shrinkage (same grade, same simulated shrinkage, but different sizes) but after numerous comparisons it was found that the best shrinkage for a particular grade was the same for all three different thicknesses. However, this was used to find an average best shrinkage for a grade. So, instead of having one set of components to inspect and decide which shrinkage was best, there were three sets and the average of the three then was taken as the ideal shrinkage for a grade. The three different sets were however not all inspected at the same time – they were inspected on different days among other pressing experiments in order to confirm that the ideal shrinkage for a grade was being quoted consistently. If all three sets for a grade were to be inspected straight after each other, it may have been easy to be complacent and say that the best shrinkage was the same in all cases. It must be noted that the inspector was extremely consistent and never contradicted his conclusions.

The experiments were carried out using ‘negative’ shapes i.e. parallel vertical surfaces (See section 2.2.3). After inspection, one set of green compacts was sent to be sintered and when the set came back, the sintered components were measured with a Mitutoyo digital vernier (resolution of 0.01 mm) and the actual shrinkage that each compact underwent was recorded. The green compact dimensions were measured with the same vernier. It must be noted that after pressing, a green compact expands very slightly – this is known as ‘spring-back’ and was assumed to be negligible.

The green dimensions were measured after pressing and a green compact volume was then calculated. The green compact volume was then calculated as the product of the measured compact thickness and the pressed cross-sectional area (this was a known constant that was obtained from the tooling technical drawings – the spring-back through the cross-sectional area was considered to be negligible).

The green compacts and sintered pieces for each grade were then placed in a storage area for future reference.



Figure 3.6 – Green compacts and sintered components for all the powder grades tested

3.1.4 Positive insert pressing tests

A set of experiments were performed to investigate the effect of ‘positive’ angle on the ideal shrinkage for a grade. Many cutting inserts manufactured at Powder Industries are of the ‘positive insert’ type (See 2.2.3). The ‘positive’ angle in these kinds of inserts may up to 25° from the vertical. Cutting inserts are manufactured from a ‘tool’ grade powder (less than 10% Co, grain size less than $3\mu\text{m}$).

Four different tool grades were used with a ‘positive insert’ toolset (positive angle being 20°). This ‘positive insert’ toolset was chosen as the angle is quite large and most cutting inserts manufactured at Powder Industries have a smaller angle (usually 11°) and very few have an equal or larger angle. This cutting insert is also intricate and is considered difficult to press. The green dimensions are also close to the ‘negative’ insert toolset used in the previous experiments (with the exception of the positive angle).

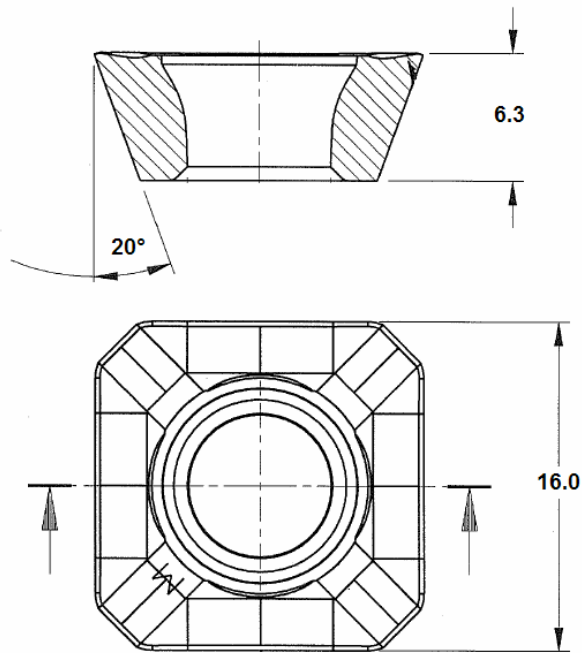


Figure 3.7 – Green positive insert shape and dimensions (in mm)

With each grade, a ‘negative’ (using same toolset as in previous experiments) and a ‘positive’ component were pressed and the two pieces were inspected and their green densities measured (the theoretical green volume for the positive insert was calculated using a 3D modeling program that was used in the original designing of the insert). The idea to press ‘negative’ and ‘positive’ inserts was to check if the two green densities were the same and thus the best shrinkage for a grade was the same for both component shapes. At the beginning of the project, some preliminary tests were performed and the component shapes were changed. At that time the shape of a component seemed to have an influence on shrinkage but these more accurate tests showed that it was not so.

3.1.5 Standard pressing experiment test sheet

A standard test sheet (see pg. 25) was setup for the pressing experiments for which each grade was being evaluated. On this sheet, all the relevant pressing data were captured and a theoretical mass for each green compact was calculated in order to achieve a target shrinkage. This theoretical green mass was printed on the test sheet and the actual weight achieved was recorded. The actual mass of powder that was pressed was usually to within 0.01g of the target mass required. When filling the die cavity, the mass of powder varies slightly from pressing to pressing, this is due to slight variations in

physical powder properties within a sample of powder such as granule size distributions and granule shapes, ‘fine granule’ content, etc.

To give an example of how a shrinkage was simulated, please note the following example:

Toolset shrinkage design: 20% (This is the reference or designed shrinkage)

Mass required for this toolset=(SD)×(required sintered volume)×(volatile content) (1)

The SD, required sintered volume and volatile content (expressed as a percentage of mass) are known, thus a powder mass is calculated. This is the amount of powder in the tool cavity when pressing a compact.

In the present work, in order to decrease or increase the shrinkage by a desired percentage, the mass needed to be decreased or increased by an inverse proportion. (keeping in mind that the green volume is constant).

Normally, to change shrinkage, the green volume is changed and the green mass is kept constant so to increase shrinkage by a certain percent, the new volume of the green compact would be calculated :

$$(\% \text{ volume change}) = 100 \times (\text{new shrinkage}^3 - \text{tool shrinkage}^3) / (\text{tool shrinkage}^3) \quad (2)$$

where the shrinkage is in the form ‘1.shrinkage’. e.g. 1.20 for 20% (See example below)

The shrinkages are cubed as shrinkage (by definition) is change of linear dimension in a linear direction and one needs to take 3 dimensions into account.

In the case where we have a constant green volume, the mass is changed by a certain amount to achieve a certain volumetric change during sintering. So by subtracting a fixed amount of powder, one decreases the green density proportionally and the shrinkage that the compact undergoes will be higher. (Note that a 1% decrease in mass means that the density decreases by 1% when pressing to a fixed volume. Due to the change in green density, volumetric shrinkage during sintering then increases by 1%. So, linear shrinkage change is actually the cube root of the volumetric change.)

So, in order to increase the shrinkage by 2 percent, the mass must be changed by the following amount:

$$(\% \text{ mass change}) = 100 \times (1.22^3 - 1.20^3) / (1.20^3) \times (-1) \quad (3)$$

Where:

- '1.20' is the toolset design shrinkage or reference i.e. 20%
- '1.22' is the desired shrinkage i.e. 22%
- (-1) is multiplied into the equation in order to have a negative change in mass

Thus to achieve a 2% increase in shrinkage when using a '20% tool', the mass must be decreased by approximately 5.1% from the original mass value when pressing to the same fixed volume.

The example shown above is for a toolset designed with 20% shrinkage. The toolset used in all of the pressing experiments was designed for a 25% shrinkage and the required shrinkages were in the range of 20 to 25%. Thus powder was added to the reference weight for 25% in order to achieve lower shrinkages.

When examining the sample test sheet, please note the following terms (some of the terms used are internal to Powder Industries and are not self-explanatory to someone from outside the organization):

- 'Equivalent tool %' refers to the target shrinkage (term used for the purpose of this project as is not standard at Powder Industries)
- 'Marking' indicates the physical inscription made on the test piece. E.g. 'CR10-24' indicates a compact pressed from grade CR10 to a shrinkage of 24%.
- 'Th. Mass' is the calculated or theoretical green compact mass needed to achieve the target shrinkage (i.e. the equivalent tool %).
- 'Height' is the actual test piece thickness after pressing (2,4 or 8mm as described in section 3.1.3)
- 'IC' is the width of the actual square test piece.
- 'Force' is the recorded pressing force in kN.
- 'Weight' in the 'Pressing' and 'Sintered' sections is the measured weight of the component in grams (green compact or sintered component)
- 'Fill height' is the distance of the lower punch in relation to the top of the die cavity. This is the effective height that the powder reaches when it fills the die before pressing.

- ‘Press O.S.’ is what is known as ‘press offset’ and is a parameter adjustment made on the hydraulic press during pressing in order to achieve a certain component pressing.
- ‘Rating’ is the inspection result after checking a green compact after pressing. The standard used to indicate quality of a pressed compact :
 - ‘SS’ means slightly soft (i.e. slightly under-pressed)
 - ‘S’ means soft (i.e. under-pressed)
 - ‘G’ or ‘OK’ indicates an adequate component that would normally pass quality control.
 - ‘B’ indicates the best pressed component from a set of pressings.
 - ‘H’ indicates a slightly over-pressed compact
 - ‘VH’ indicates a very over-pressed compact with possible defects such as cracking.
- ‘SNUN phase 1/2/3’ indicates the compact thickness where 1 = 2mm, 2 = 4mm and 3 = 8mm.
- ‘S height’ indicates the reference sintered thickness of a component for a 25% shrinkage (in mm)
- ‘SG’ is the powder grade specific gravity value after sintering (in g/cm^3).
- ‘Press height’ is the target thickness during pressing. E.g. 4mm.
- ‘Weight’ is the reference mass of powder needed for a 25% toolset (in grams).
- ‘Volume’ is the reference volume of the sintered component after shrinkage of 25% (in cm^3).
- ‘Volatile’ indicates the compact weight loss that occurs during sintering – this is mainly removed pressing lubricant as well as a very small amount of air that is trapped during pressing.
- ‘Item no’, ‘Batch no’, ‘PRZ reclaim’ are internal company references for a grade of powder.

- ‘App. Density’ refers to a powder apparent density (in g/cm³). This value was obtained from Powder Industries powder production database and indicates the apparent density for a batch of grade powder.

It should be noted that the test sheet evolved over a period of time and the sheet is not perfect – not all of the units are indicated and some of the data fields were obsolete or redundant (such as ‘PEG content’). Also the data grouping may seem misarranged (e.g. apparent density is in the ‘pressing information’ area). The test sheet function was mainly for capturing data that would then be input into relevant spreadsheets for later analysis.

Powder	SHUH120412 - Size 1/2/3	
Tool set	SHUH120412	Size 1/2/3
Area - pressed	250.1	
S Height		
SG		Volume cm ³
App. Density		Volatile
Press height		
Weight		
Tool	25	%

Powder details		Links
Date :		Powder sheet
Item no :		RTP
Batch no :		Spray drying
Co % :	%	
Grain size :	μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
	20.0												
	20.5												
	21.0												
	21.5												
	22.0												
	22.5												
	23.0												
	23.5												
	24.0												

Figure 3.8 – Standard pressing test sheet

3.1.6 Apparent density measurement of test powder samples

The apparent density of each test powder sample was measured according to company standards. A sample volume of the test powder was loaded into a Hall flow meter funnel (see 3.2.3) and allowed to pour into a 25 cm³ container. Any leftover powder at the top of the container was levelled off, thus a sample of 25 cm³ was left. This sample of powder was weighed and an average apparent density could be calculated. This was repeated with every grade of powder tested and an apparent density for each test sample was recorded. The ‘apparent density’ of a sample of powder is the measured density of powder that was poured into a container using the Hall flow meter funnel from a prescribed height. This is a ‘non-tapped’ sample of powder i.e. the density is measured ‘as poured’ with no further steps or procedures. The company standard for measuring apparent density of a metal powder is based [19].

Each test powder sample had been obtained from a batch of the same grade and during production of a grade of that powder; average apparent density is recorded and stored on the company database. Apparent density for each grade of powder was also recorded and compared to the test powder sample apparent density.

During a year of production, a number of batches of a particular grade are produced and the average apparent density of a batch is recorded. These values were collected and thus an average historical value of apparent density for a grade was obtained.

So, each grade of powder had three different apparent densities associated with it : the test sample value (used in the pressing experiments), a value for the batch from which the sample was obtained and a historical value.

Each 25 cm³ sample of powder that was weighed was stored in an air-tight labeled container for any future reference or re-measuring purposes.



Figure 3.9 – Measured 25 cm³ and weighed test powder sample in an air-tight container.

3.1.7 Screen fractioning of test powder samples

Ready-to-press powder (all grades) is made up of granules of varying sizes and size distribution.

In order to establish granule size distributions, the granulated test powder samples were sieved through 300µm, 212µm, 125µm, 90µm and 63µm mesh screens. The powder from each screen was collected and weighed. Thus, granule size distributions for each test powder sample could be established by mass percent. As with apparent densities, the screen fraction results are recorded during powder production and historical granule size distributions for each grade of powder could be established.

The test powder granule size distributions of a grade were compared to the historical granule size distribution for the same grade. In Chapter 4, the graphs for the test powder sample and historical values are shown for comparison as well as the measured screen fraction mass values for the test powder in table form. Historical screen fraction results can be found in Appendix 1 (in the relevant powder grade section).

Figure 3.10 shows a good granule size distribution (mass percent vs. granule size in µm).

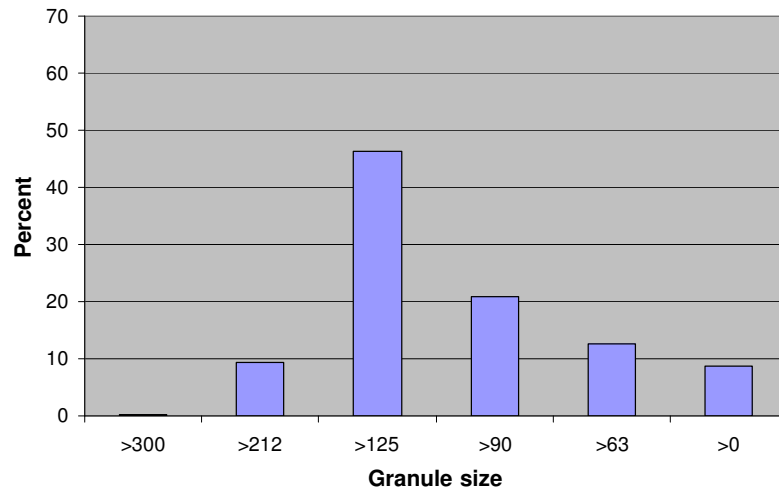


Figure 3.10 – Histogram plot of typical powder granule size distribution

3.1.8 Inspection of sintered test pieces

The best and worst sintered test pieces (original pressings of 4mm) from each grade were cleaned and mounted in a resin. These samples were then ground and polished to achieve a mirror finish (See section 3.2.4 for procedure description). Each of these pieces (46 sintered test pieces altogether) were examined for porosity at 100x magnification on the lab stereo-microscope. In Chapter 4, the best and worst test pieces of each grade are compared for porosity and then.

The SG of each of the sintered test pieces was measured in the lab at Powder Industries by using the Archimedes method. The measurement of the sintered density of a test piece was done according to company procedure [20].

The sintered test pieces were examined and a comparison of the observed surface was made with the company micrographs for porosity. Please refer to Appendix A2.2 for samples of the company micrograph examples.

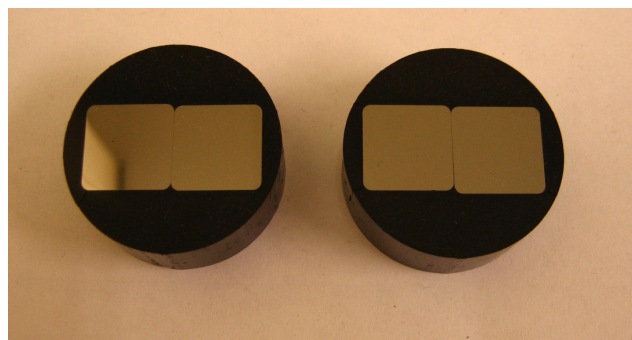


Figure 3.11 – Mounted and polished sintered test pieces

3.2 Test equipment

3.2.1 Pressing equipment

The pressing equipment used in all of the experiments is a fully programmable CNC controlled hydraulic press manufactured by the German company Dorst. The model is the TPA15HS capable of exerting a maximum pressing force of 150 kN.

The pressing accuracy (i.e. accuracy of compact height) is to within $\pm 2\mu\text{m}$ and the weight adjustment accuracy ± 0.01 grams.



Figure 3.12 – Dorst TPA15HS Hydraulic CNC press

3.2.2 Sinter-HIP furnace

All of the test pieces were sintered in the Powder Industries standard sinter-HIP furnace [18]. The furnace was manufactured by the American company Ultra temp. The test pieces were sintered together with standard production runs and were exposed to the Argon HIP cycle after sintering. It should be noted that in the assessment of the sintered components (section 3.1.8) for porosity, the sintering should have ideally been done without pressure (i.e. without HIPing) as HIPing removes pores and corrects pressing defects. The test pieces were pressed and sintered according to standard company procedure as the aim of the project was to solve the compaction problems specific to

Powder Industries. Figure 3.13 shows the sinter-HIP furnace used in the sintering of the test pieces.



Figure 3.13 – Ultra temp sinter-HIP furnace

3.2.3 Powder analysis equipment

To measure the apparent density of a sample of powder, a Hall flow meter funnel was used in conjunction with a 25 cm³ container. The 25 cm³ of powder was weighed on a digital scale with a resolution of 0.01g.



Figure 3.14 – Digital scale and Hall flow meter funnels

To sieve the granulated powder samples into constituent granule size groups, standard stackable lab sieves of 300μm, 212μm, 125μm, 90μm and 63μm mesh sizes were used. In order to facilitate good sieving, the screens were stacked with the smallest screen at

the bottom and the largest at the top. The stack was loaded into a Rototap sieve shaker which vibrated the stack for a period of two minutes. The initial powder mass which was loaded into the top screen was then broken down into the granule size groups as it fell through the stack during sieving.



Figures 3.15 & 3.16 – The screen stack loaded into the shaker and the sieves used for screen fractioning the test powder samples.

3.2.4 Mounting and polishing equipment

For mounting and preparing the sintered test pieces for surface grinding and polishing, a Leco PR-32 mounting press was used. The PR-32 is an automatic, dual mount press that automatically applies heat and pressure for a selected period of time. The mounting powder used was Struers multifast phenolic hot mounting resin.



Figure 3.17 – Leco mounting press

Grinding and polishing of the mounted test pieces was done using the Struers LaboPol 5 machine in the company laboratory. Grinding discs and water were used for the initial grinding of the test pieces and polishing was done using diamond paste and polishing discs. The grinding and polishing was done as specified by company procedures.



Figure 3.18 – Struers grinding and polishing equipment

The polished test pieces were all inspected for porosity using a Nikon metallurgical microscope at a magnification of 100x. The test pieces were compared to standard porosity micrographs (refer to Appendix 3). Images of the surfaces observed were photographed for reference and comparison purposes.



Figure 3.19 – Nikon microscope used to check for porosity

3.3 Materials

3.3.1 Powders tested

The most popular powders used during normal production at Powder Industries were selected for testing – there was no particular order of powders followed during pressing experiments. Once all of the powders were tested, the results and grades were sorted by grade name in alphabetical order. The major differences between powder grades are cobalt content, grain size, sintered density and mixed crystal content (TiC, TaC and NbC). In total, 23 different grades were tested. A table listing the various powder grades as well as their basic characteristics (as quoted by company lab specification) follows on page 34.

Table 3.1 – List of powders tested (sorted in alphabetical order by grade name)

Powder grade name	Average S.G. (g/cm³)	Co %	Average grain size (µm)	Mixed crystals?
230	14.55	9.7	2-6	N
AP20	14.40	10.5	2-3	N
CN10	14.35	6.7	< 1	Y
CR10	14.30	9.5	< 1	N
H1P	12.93	6.0	1	Y
H6	14.95	5.7	1-1.5	Y-very little
K15M	12.75	7.3	1-2	Y
MT16	13.90	16.0	2-3	N
MZ15	14.00	15.2	2-4	N
N6/194	14.90	6.0	1-3	N
P25M	12.55	9.5	1-2	Y
P30M	12.40	10.0	2-3	Y
P40M	13.20	10.0	2-3	Y
P511	14.40	11.7	3-6	Y
P7	14.80	7.5	3-4	N
PBF	14.95	5.9	2-3	N
PBT	14.93	6.0	2-4	N
PC-20	14.60	6.0	1-3	Y
PI-15	14.08	6.0	2-3	Y
PI-25	14.08	6.6	2-3	Y
PI-40	13.80	8.5	2-4	Y
Q3	13.25	23.0	3-6	N
S9	12.30	10.0	2-3	Y

The table above includes most of the powders used in the manufacture of various products at Powder Industries. The three main types of powders are tool grades, mining grades and wear grades – these have not been categorized in the above table but in the data results section (see 4.2), it is indicated what category that particular powder falls under.

3.3.2 Grade data tables

The physical and metallurgical properties of each grade (as listed in the previous section) that are relevant to this project are summarized in table form. Information listed for each grade includes: the grade name, cobalt content, average WC grain size, sintered density (S.G.) as per lab specification, historical S.G. , historical apparent density (over 1 year where possible), historical volatile content and mixed crystal content (TiC, TaC and NbC). The values have been obtained from the company laboratory reports, specification sheets and from information that is recorded on the company database during the manufacturing process. The historical data such as historical S.G. and historical apparent density was calculated from company records.

Table 3.2– Grade ‘230’ summary

Grade Name	230
Cobalt content	9.7 %
Average WC grain size	2-6 μm
SG (company specification)	14.55 g/cm^3
SG (Historical)	14.52 g/cm^3
Apparent density (Historical)	3.70 g/cm^3
Volatile content (Historical)	2.18 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.3– Grade ‘AP20’ summary

Grade Name	AP20
Cobalt content	10.5 %
Average WC grain size	2-3 μm
SG (company specification)	14.40 g/cm^3
SG (Historical)	14.39 g/cm^3
Apparent density (Historical)	3.44 g/cm^3
Volatile content (Historical)	2.24 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.4– Grade ‘CN10’ summary

Grade Name	CN10
Cobalt content	6.7 %
Average WC grain size	< 1 μm
SG (company specification)	14.35 g/cm^3
SG (Historical)	14.41 g/cm^3
Apparent density (Historical)	3.29 g/cm^3
Volatile content (Historical)	2.66 %
TiC / TaC / NbC / Ni / Cr_3C_2 content (%)	0.0 / 0.0 / 0.0 / 2.7 / 1.3

Table 3.5– Grade ‘CR10’ summary

Grade Name	CR10
Cobalt content	9.5 %
Average WC grain size	< 1 μm
SG (company specification)	14.30 g/cm^3
SG (Historical)	14.46 g/cm^3
Apparent density (Historical)	3.28 g/cm^3
Volatile content (Historical)	2.83 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.6– Grade ‘H1P’ summary

Grade Name	H1P
Cobalt content	6.0 %
Average WC grain size	1 μm
SG (company specification)	12.95 g/cm^3
SG (Historical)	12.92 g/cm^3
Apparent density (Historical)	3.21 g/cm^3
Volatile content (Historical)	2.54 %
TiC / TaC / NbC content (%)	7.50 / 1.00 / 0.00

Table 3.7– Grade ‘H6’ summary

Grade Name	H6
Cobalt content	5.7 %
Average WC grain size	1-1.5 μm
SG (company specification)	14.95 g/cm^3
SG (Historical)	14.92 g/cm^3
Apparent density (Historical)	3.41 g/cm^3
Volatile content (Historical)	2.51 %
TiC / TaC / NbC content (%)	0.00 / 0.09 / 0.01

Table 3.8– Grade ‘K15M’ summary

Grade Name	K15M
Cobalt content	7.3 %
Average WC grain size	1-2 μm
SG (company specification)	12.75 g/cm^3
SG (Historical)	12.81 g/cm^3
Apparent density (Historical)	3.19 g/cm^3
Volatile content (Historical)	2.55 %
TiC / TaC / NbC content (%)	7.50 / 0.00 / 0.00

Table 3.9– Grade ‘MT16’ summary

Grade Name	MT16
Cobalt content	16.0 %
Average WC grain size	2-3 μm
SG (company specification)	13.90 g/cm^3
SG (Historical)	13.88 g/cm^3
Apparent density (Historical)	3.37 g/cm^3
Volatile content (Historical)	2.48 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.10– Grade ‘MZ15’ summary

Grade Name	MZ15
Cobalt content	15.2 %
Average WC grain size	2-4 μm
SG (company specification)	14.00 g/cm^3
SG (Historical)	13.99 g/cm^3
Apparent density (Historical)	3.31 g/cm^3
Volatile content (Historical)	2.62 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.11– Grade ‘N6/194’ summary

Grade Name	N6 / 194
Cobalt content	6.0 %
Average WC grain size	1-3 μm
SG (company specification)	14.93 g/cm^3
SG (Historical)	14.88 g/cm^3
Apparent density (Historical)	3.72 g/cm^3
Volatile content (Historical)	2.04 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.12– Grade ‘P7’ summary

Grade Name	P7
Cobalt content	7.5 %
Average WC grain size	2-6 μm
SG (company specification)	14.80 g/cm^3
SG (Historical)	14.76 g/cm^3
Apparent density (Historical)	3.72 g/cm^3
Volatile content (Historical)	2.30 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.13– Grade ‘P25M’ summary

Grade Name	P25M
Cobalt content	9.5 %
Average WC grain size	1-2 μm
SG (company specification)	12.55 g/cm^3
SG (Historical)	12.58 g/cm^3
Apparent density (Historical)	3.14 g/cm^3
Volatile content (Historical)	2.37 %
TiC / TaC / NbC content (%)	6.50 / 13.05 / 1.45

Table 3.14 - Grade ‘P30M’ summary

Grade Name	P30M
Cobalt content	10.0 %
Average WC grain size	2-3 μm
SG (company specification)	12.40 g/cm^3
SG (Historical)	12.39 g/cm^3
Apparent density (Historical)	3.09 g/cm^3
Volatile content (Historical)	2.42 %
TiC / TaC / NbC content (%)	7.00 / 8.00 / 2.00

Table 3.15– Grade ‘P40M’ summary

Grade Name	P40M
Cobalt content	10.0 %
Average WC grain size	2-3 μm
SG (company specification)	13.20 g/cm^3
SG (Historical)	13.24 g/cm^3
Apparent density (Historical)	3.26 g/cm^3
Volatile content (Historical)	2.30 %
TiC / TaC / NbC content (%)	4.00 / 4.80 / 1.20

Table 3.16– Grade ‘P511’ summary

Grade Name	P511
Cobalt content	11.7 %
Average WC grain size	3-6 μm
SG (company specification)	14.40 g/cm^3
SG (Historical)	14.33 g/cm^3
Apparent density (Historical)	3.53 g/cm^3
Volatile content (Historical)	2.37 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.17 - Grade ‘PBF’ summary

Grade Name	PBF
Cobalt content	5.9 %
Average WC grain size	2-3 μm
SG (company specification)	14.95 g/cm^3
SG (Historical)	14.95 g/cm^3
Apparent density (Historical)	3.70 g/cm^3
Volatile content (Historical)	2.30 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.18 – Grade ‘PBT’ summary

Grade Name	PBT
Cobalt content	6.0 %
Average WC grain size	2-4 μm
SG (company specification)	14.95 g/cm^3
SG (Historical)	14.94 g/cm^3
Apparent density (Historical)	3.84 g/cm^3
Volatile content (Historical)	2.20 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.19 – Grade ‘PC20’ summary

Grade Name	PC20
Cobalt content	6.0 %
Average WC grain size	1-3 μm
SG (company specification)	14.60 g/cm^3
SG (Historical)	14.60 g/cm^3
Apparent density (Historical)	3.71 g/cm^3
Volatile content (Historical)	2.50 %
TiC / TaC / NbC content (%)	0.86 / 2.23 / 0.26

Table 3.20 - Grade ‘PI-15’ summary

Grade Name	PI-15
Cobalt content	6.0 %
Average WC grain size	2-3 μm
SG (company specification)	14.05 g/cm^3
SG (Historical)	14.05 g/cm^3
Apparent density (Historical)	3.52 g/cm^3
Volatile content (Historical)	2.32 %
TiC / TaC / NbC content (%)	2.83 / 3.47 / 0.39

Table 3.21– Grade ‘PI-25’ summary

Grade Name	PI-25
Cobalt content	6.6 %
Average WC grain size	2-3 μm
SG (company specification)	14.05 g/cm^3
SG (Historical)	14.07 g/cm^3
Apparent density (Historical)	3.53 g/cm^3
Volatile content (Historical)	2.35 %
TiC / TaC / NbC content (%)	2.30 / 2.76 / 0.69

Table 3.22– Grade ‘PI-40’ summary

Grade Name	PI-40
Cobalt content	8.5 %
Average WC grain size	2-4 μm
SG (company specification)	13.85 g/cm^3
SG (Historical)	13.70 g/cm^3
Apparent density (Historical)	3.40 g/cm^3
Volatile content (Historical)	2.36 %
TiC / TaC / NbC content (%)	2.70 / 2.88 / 0.72

Table 3.23 - Grade ‘Q3’ summary

Grade Name	Q3
Cobalt content	23.0 %
Average WC grain size	3-6 μm
SG (company specification)	13.25 g/cm^3
SG (Historical)	13.24 g/cm^3
Apparent density (Historical)	3.10 g/cm^3
Volatile content (Historical)	2.30 %
TiC / TaC / NbC content (%)	0.00 / 0.00 / 0.00

Table 3.24 – Grade ‘S9’ summary

Grade Name	S9
Cobalt content	10.0 %
Average WC grain size	1-3 μm
SG (company specification)	12.30 g/cm^3
SG (Historical)	12.38 g/cm^3
Apparent density (Historical)	3.25 g/cm^3
Volatile content (Historical)	2.29 %
TiC / TaC / NbC content (%)	8.50 / 2.40 / 0.60

4. Experimental results and discussion

4.1 Layout for presentation of experimental results

The experimental results presented are laid out as per grade. There were a number of different tests performed for each grade of powder (as described in Chapter 3) and all the results are presented together for clarity. This is to give an overall idea or ‘feel’ for a particular grade of powder and how it behaves. In total, there are 23 different grades that have been evaluated and are ordered alphabetically by grade name (section 4.2). All of the original experimental test sheets as well as supporting data for the shown results can be found in Appendix A1 (also arranged by grade name in alphabetical order).

The first table in each section shows experimental data as well as important information for each grade of powder such as SD (sintered density), apparent density, volatile content, green density and porosity found in sintered components. Note that ‘batch’ values indicate company recorded data from a large batch of powder and ‘test sample’ values indicate data from a sample taken from the same batch of powder.

The second table in each section shows the best pressing results from a variety of pressing experiments performed. The table is a summary showing best shrinkage obtained, the green density achieved for the best pressed components, the green weight of the pressed component as well as the recorded force and pressure during compaction.

The heading “best compact” in the second table indicates the green component that was evaluated to be the best (or best compacted component) from a set of components in an experiment and this is indicated in terms of theoretical or expected shrinkage for that particular mass of powder. The “actual shrinkage” is the amount of shrinkage that the same test piece actually underwent after sintering and this was found by physically measuring the test piece.

After the tables are graphs showing granule size distributions for the test powder used as well as the historical data for the grade being evaluated (granule size in μm). Finally,

the micrographs for the best and worst obtained shrinkages are shown to indicate the level of porosity found in a sintered test piece, although it must be noted that the difference is often very small because the pieces were hot isostatically pressed (See 3.2.2).

The following information or results will be shown for each grade:

- Pressing results for ‘negative’ style tests for the three various sizes (common cross-sectional area – see section 3.1.2/3) – The outcome of this set of tests was to find the best green density and thus the best shrinkage (as measured after sintering).
- Measured green density for the chosen ‘best’ green components for all pressed sizes.
- Sintered component density or S.D. (measured by the Archimedes method)
- Measured apparent density of the granulated test powder from a grade as well as the historical apparent density of the same grade for a period of 1 year.
- Granule size distribution plots for a test sample of powder (historical data are shown as well), the plots show mass percent vs. granule size (μm).
- Porosity check for sintered components from a grade (the best and worst components from each grade were mounted and polished then examined under a microscope). Micrographs are shown in both cases for each grade.

Following the summaries for each grade when pressing ‘negative’ components is a section showing the pressing and experimentation results when varying the shape of a component (positive inserts) in order to evaluate if a complex shape or significant angle in the component geometry affects the ideal shrinkage for a particular grade.

4.2 Experimental data results

All of the data collected from pressing, sintering, powder analysis and sintered component analysis is shown in this section in tabular as well as graphical form. As there was a large amount of data collected, only the most important information is shown in this section. Other data such as historical data and supporting information relevant to the grade being presented can be found in the Appendix 1 & 2.

The order of data shown is alphabetical by grade name.

4.2.1 Grade ‘230’ experimental data

‘230’ grade powder is a mining grade (straight WC-Co) that has a relatively high cobalt content (9.7 wt %) and a wide grain size distribution (2-6µm). It is a commonly used grade in products such as shims which are used to support cutting inserts. It is an important grade as it is widely used in a variety of products. Pressing test sheets, historical apparent screen fraction reports as well as other test results and reports can be found in Appendix 1-A1.

Table 4.2 - Grade ‘230’ experimental data summary

SD (Batch)	14.52 g/cm ³
SD (Test samples)	14.46 g/cm ³
Apparent density (Grade batch)	3.60 g/cm ³
Apparent density (Test powder sample)	3.64 g/cm ³
Volatile content (Test sample)	2.07%
Best average green density (Measured)	8.29 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.3 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.3 – Pressing test results for grade ‘230’

Green thickness (mm)	Best compact ¹	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	21.0%	21.2%	4.15	8.30	21.9 / 116
4.00	21.0%	21.4%	8.29	8.29	25.0 / 100
8.00	21.0%	21.3%	16.58	8.29	23.9 / 95.6

Table 4.4 – Screen fraction report for grade ‘230’ test powder (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
107.5 g	0.5 g	12.1 g	61.7 g	19.4 g	9.1 g	4.7 g
Percent	0.5 %	11.3 %	57.4 %	18.0 %	8.5 %	4.4 %

¹ The values shown indicate the target or theoretical shrinkage for that compact

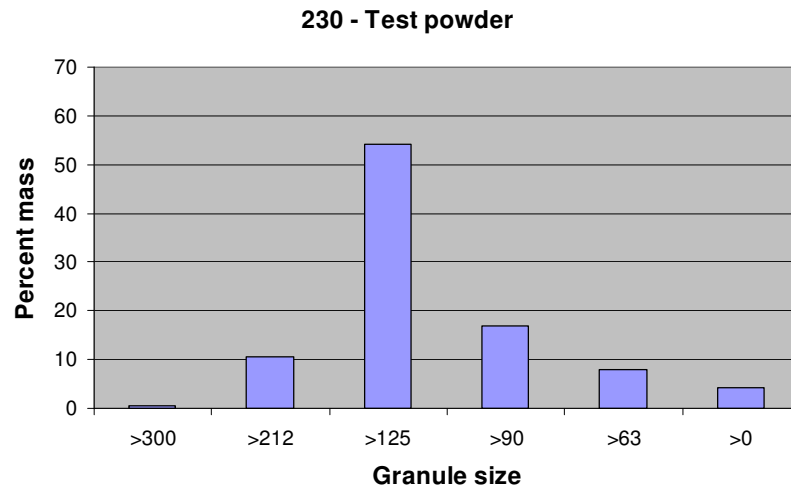


Figure 4.1 – Measured granule size distribution of test powder sample of grade 230
(apparent density of 3.64 g/cm³)

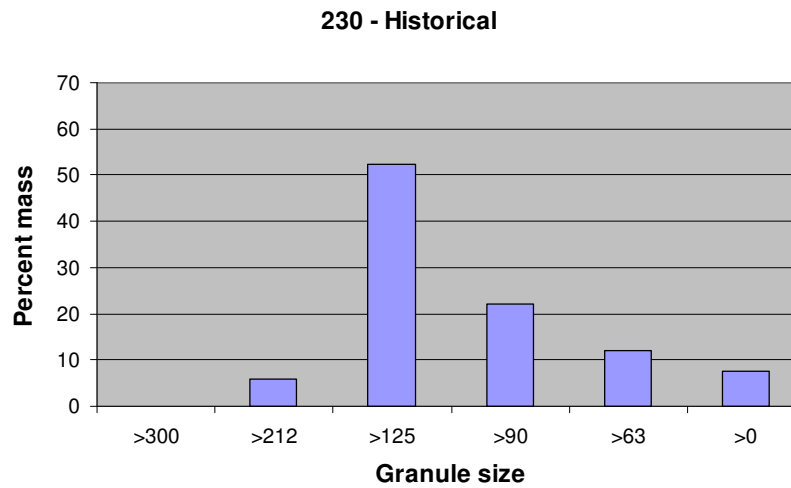


Figure 4.2 – Historical granule size distribution of grade 230 (historical apparent
density of 3.70 g/cm³)

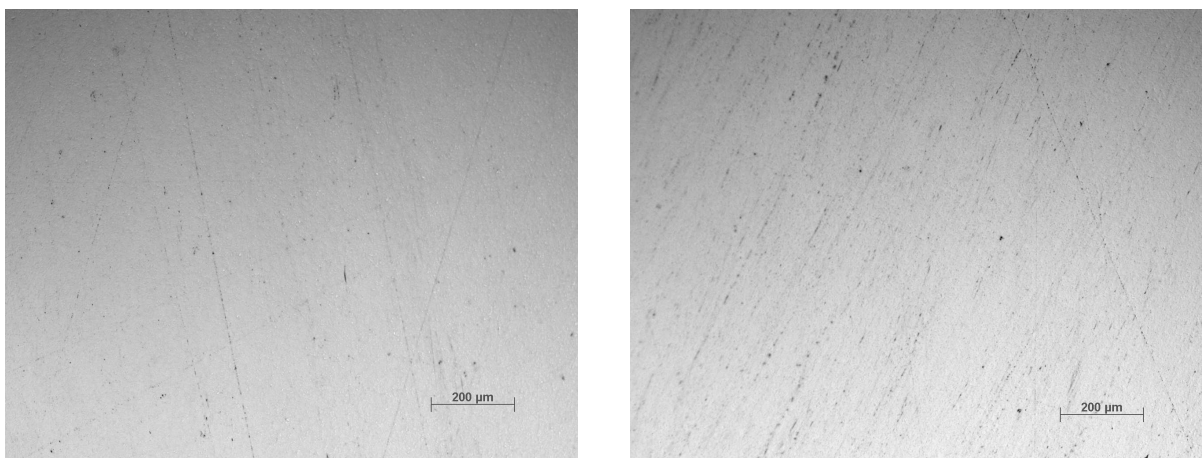


Figure 4.3 – 100x magnification image of polished section of the best shrinkage sample
(least porosity): 21.4% and the worst shrinkage (most porosity) sample: 23.7%

4.2.2 Grade ‘AP20’ experimental data

‘AP20’ grade powder is a wear grade (straight WC-Co) that has relatively high cobalt content (10.5 wt%) and a narrow coarse grain size distribution (2-3 μm). It is mainly used in the specials department where components are formed on machines after pre-sintering. It is an important grade as it is quite widely used in a variety of products. Pressing and all other test sheets can be found in Appendix 1-A2.

Table 4.5 - Grade AP20 summary

SD (Grade batch)	14.35 g/cm ³
SD (Test samples)	14.35 g/cm ³
Apparent density (Grade batch)	3.19 g/cm ³
Apparent density (Test powder sample)	3.42 g/cm ³
Volatile content (Test samples)	2.30 %
Best average green density	8.06 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	22.4 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.6 – Pressing test results for grade ‘AP20’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
1.99	21.5 %	21.7 %	4.04	8.12	30.9 / 123.6
4.00	21.5 %	22.1 %	8.07	8.07	27.1 / 108.3
8.00	22.0 %	22.6 %	15.96	7.98	23.4 / 94

Table 4.7 – Screen fraction report for grade ‘AP20’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
117.6 g	0.2 g	1.9 g	45.0 g	25.3 g	16.0 g	29.2 g
Percent	0.2 %	1.6 %	38.3 %	21.5 %	13.6 %	24.8 %

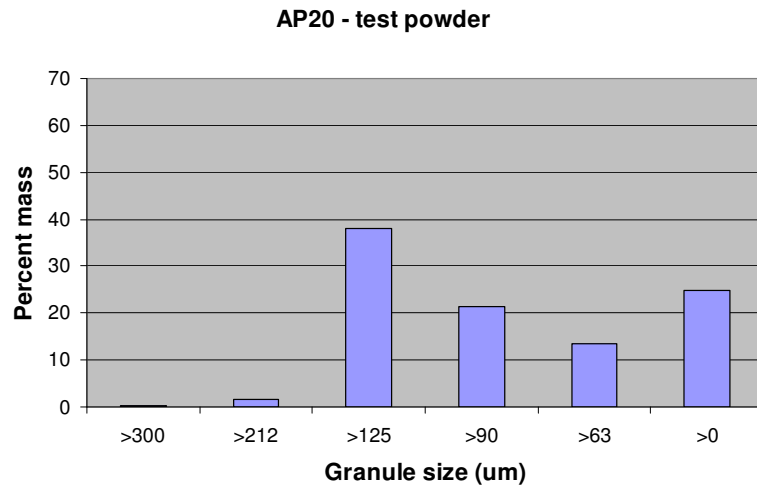


Figure 4.4 – Measured granule size distribution of test powder sample of grade AP20 (apparent density of 3.42 g/cm³)

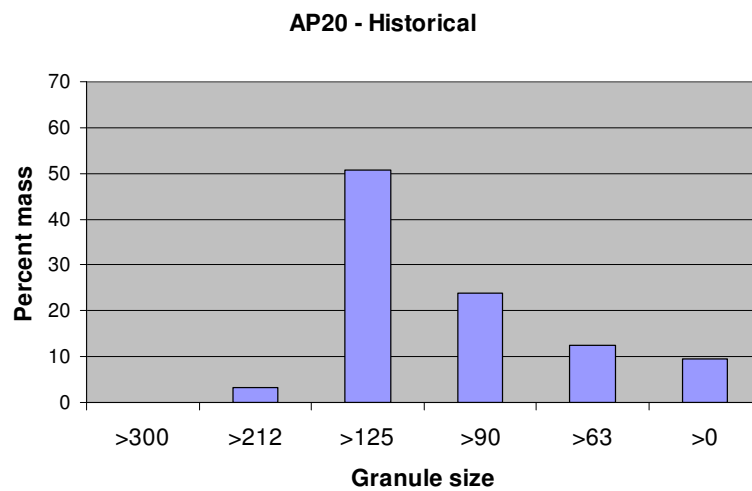


Figure 4.5 – Historical granule size distribution of grade AP20 (historical apparent density of 3.44 g/cm³)

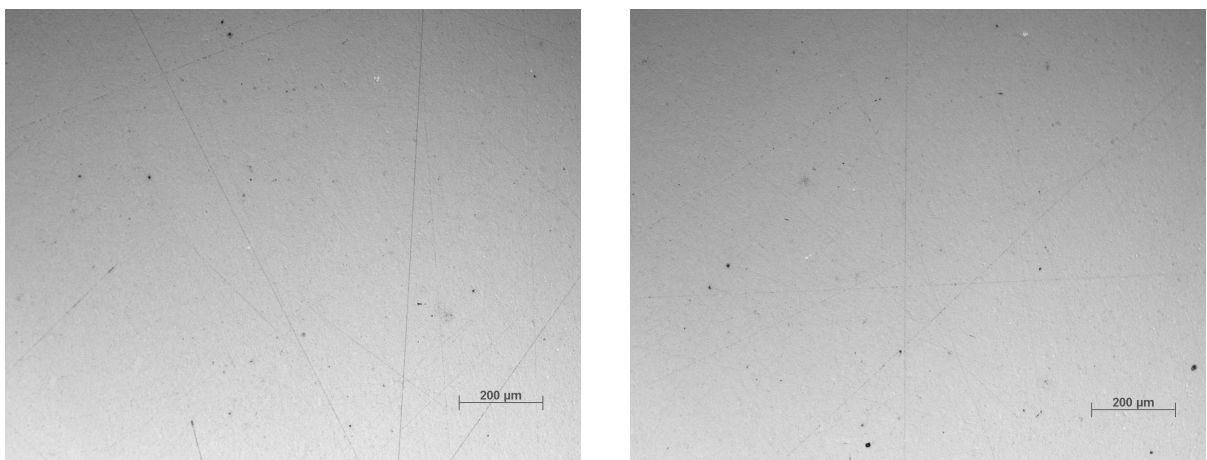


Figure 4.6 – 100x magnification image of polished section of the best shrinkage sample (least porosity) 22.1% and the worst shrinkage (most porosity) 24.4% for AP20

4.2.3 Grade ‘CN10’ experimental data

‘CN10’ grade powder is a tool grade that has relatively low cobalt content (6.7 wt%) and is a sub-micron grain size powder. There are mixed crystals of Ni and Cr₃C₂. It is a commonly used grade in products such as cutting inserts (milling type) where high hardness is required. This grade was known for being un-pressible like another grade known as ‘CR10’. Pressing test sheets as well as other test results and reports can be found in Appendix 1-A3

Table 4.8 - Grade ‘CN10’ summary

SD (grade batch)	14.41 g/cm ³
SD (Test sample)	14.36 g/cm ³
Apparent density (Grade batch)	3.32 g/cm ³
Apparent density (Test powder sample)	3.18 g/cm ³
Volatile content (Test sample)	2.76 %
Best average green density (Measured)	7.64 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	24.6 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.9 – Pressing test results for grade ‘CN10’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	24.0 %	24.2 %	3.85	7.70	41.3 / 165
4.00	24.0 %	24.1 %	7.72	7.72	40.2 / 161
8.02	25.0 %	25.4 %	15.08	7.52	28.8 / 116

Table 4.10 – Screen fraction report for grade ‘CN10’ test powder (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
185.3 g	0.7 g	1.7 g	110.8 g	38.4 g	22.9 g	10.8 g
Percent	0.4 %	0.9 %	59.8 %	20.7 %	12.4 %	5.8 %

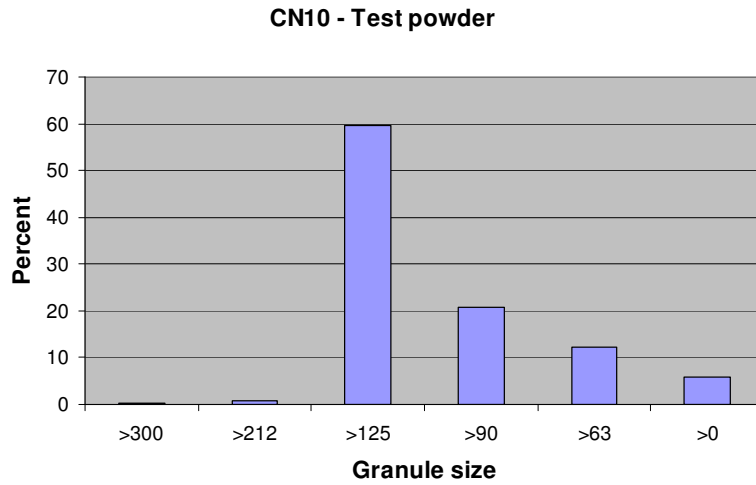


Figure 4.7 – Measured granule size distribution of test powder sample of grade CN10 (apparent density of 3.18 g/cm³)

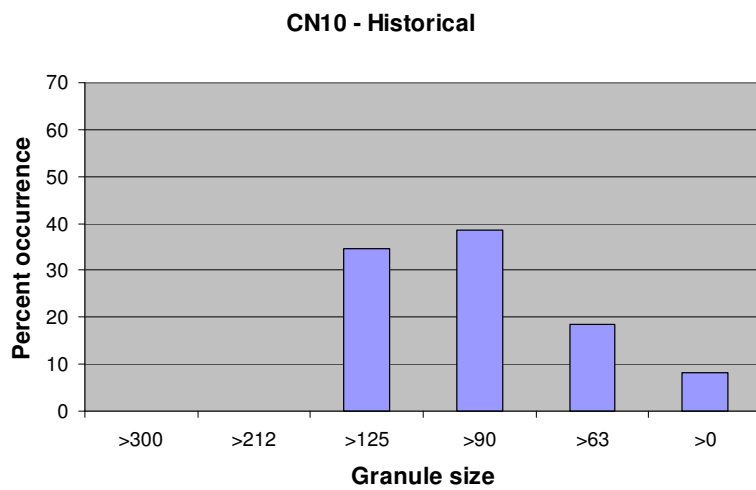


Figure 4.8 – Historical granule size distribution of grade CN10 (historical apparent density of 3.29 g/cm³)

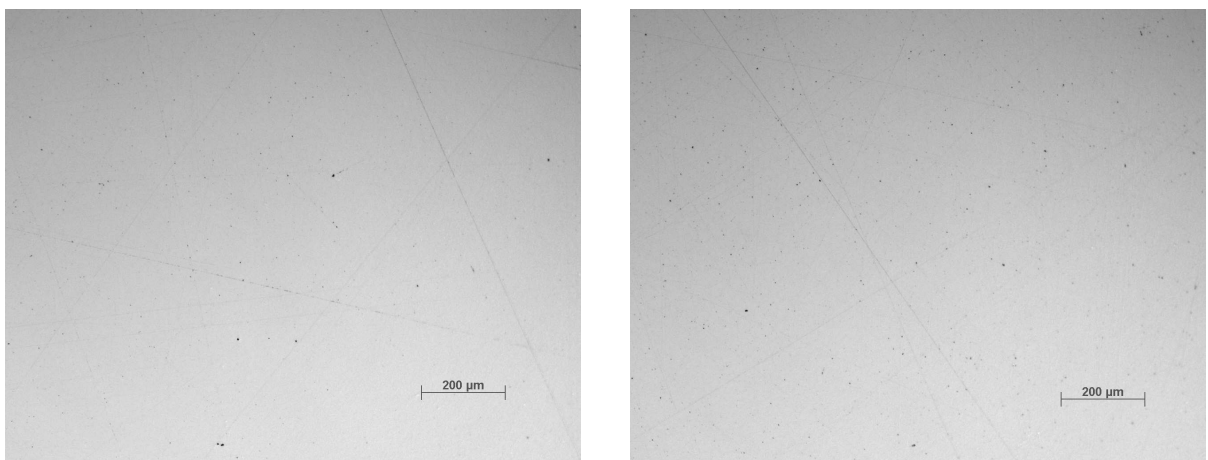


Figure 4.9 – 100x magnification image of polished section of the best shrinkage (least porosity) 24.1 % and the worst shrinkage (most porosity) 26.5 % for grade CN10

4.2.4 Grade ‘CR10’ experimental data

‘CR10’ grade powder is a tool grade that has sub-micron grain size and Co content of 9.5 wt%. It is a commonly used grade in products such as cutting inserts (milling) where toughness and high hardness are required. This is a straight grade with no mixed crystals. It is an important grade and in the past was very difficult to press due to a massively incorrect tool shrinkage consideration. Pressing test sheets as well as other test results and reports can be found in Appendix 1-A4.

Table 4.11 - Grade ‘CR10’ summary

SD (Grade batch)	14.45 g/cm ³
SD (Test sample)	14.56 g/cm ³
Apparent density (Grade batch)	3.39 g/cm ³
Apparent density (Test powder sample)	3.26 g/cm ³
Volatile content (Test sample)	3.23 %
Best average green density (Measured)	7.75 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	24.6 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.12 – Pressing test results for grade ‘CR10’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	24.0 %	24.5 %	3.87	7.74	35.3 / 141.2
4.00	24.0 %	24.8 %	7.75	7.75	33.8 / 135.0
7.98	24.0 %	24.4 %	15.52	7.78	36.1 / 144.4

Table 4.13 – Screen fraction report for grade ‘CR10’ test powder (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
146.3 g	0.1 g	0.3 g	44.5 g	53.3 g	27.8 g	20.3 g
Percent	0.1 %	0.2 %	30.4 %	36.4 %	19.0 %	13.9 %

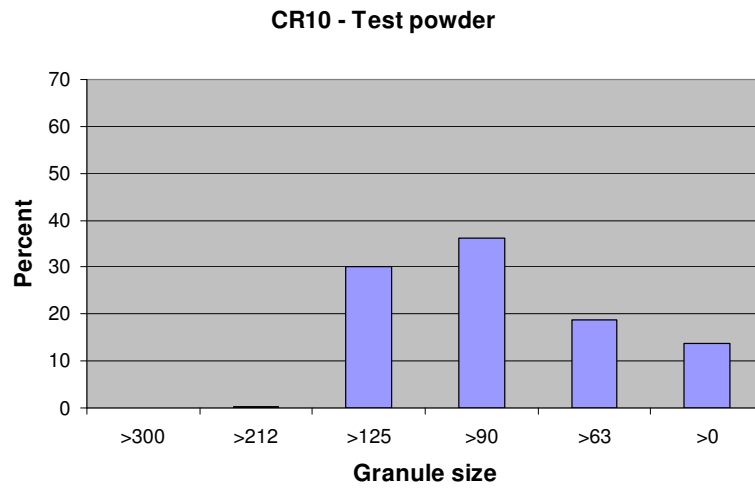


Figure 4.10 – Measured granule size distribution of test powder sample of grade CR10 (apparent density of 3.26 g/cm³)

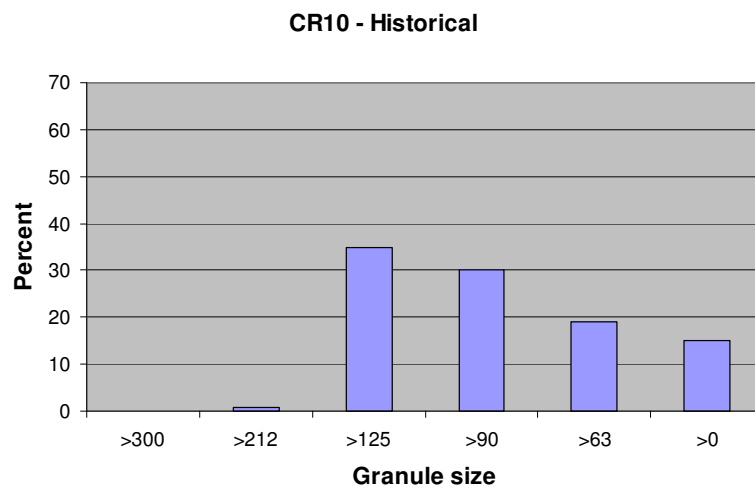


Figure 4.11 – Historical granule size distribution of grade CR10 (historical apparent density of 3.28 g/cm³)

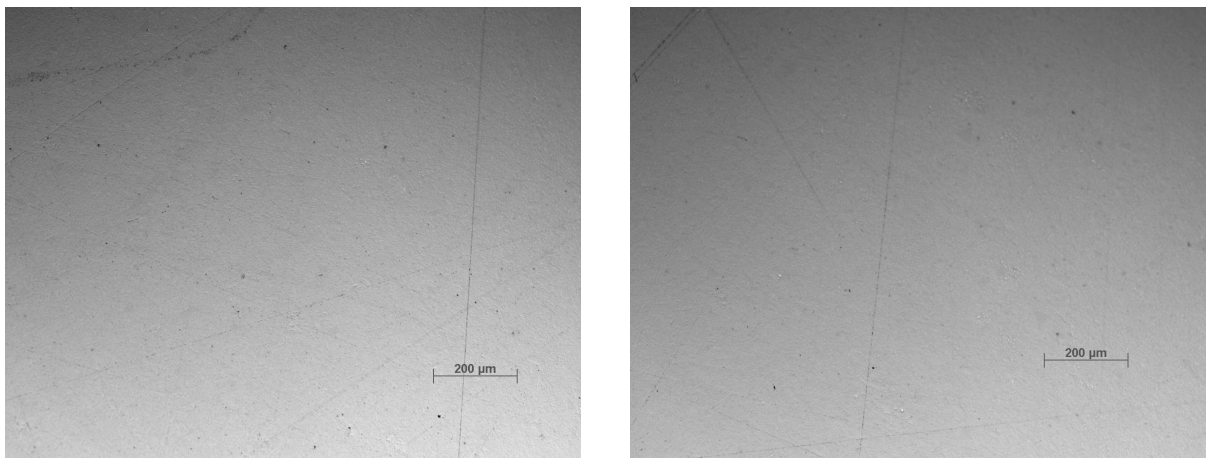


Figure 4.12 – 100x magnification image of polished section of the best shrinkage (least porosity) 24.0 % and the worst shrinkage (most porosity) 25.5 % for grade CR10

4.2.5 Grade ‘H1P’ experimental data

‘H1P’ grade powder is a tool grade that has 1 micron grain size and relatively low cobalt content (6.0 wt%). It is used where high hardness is required. Mixed crystals of TiC and TaC are present. Pressing test sheets as well as other test results and reports can be found in Appendix 1-A5.

Table 4.14 - Grade ‘H1P’ summary

SD (Grade batch)	12.89 g/cm ³
SD (Test samples)	12.89 g/cm ³
Apparent density (Grade batch)	3.24 g/cm ³
Apparent density (Test powder sample)	3.25 g/cm ³
Volatile content (Test samples)	2.46 %
Best average green density (Measured)	7.35 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	20.7 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.15 – Pressing test results for grade ‘H1P’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	21 %	20.6 %	3.68	7.36	48.5 / 193
4.02	21 %	20.8 %	7.37	7.33	48.7 / 195
-	-	-	-	-	-

Table 4.16 – Screen fraction report for grade ‘H1P’ test powder (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
158.9 g	0.1 g	1.3 g	75.0 g	47.2 g	25.1 g	10.2 g
Percent	0.1 %	0.8 %	47.2 %	29.7 %	15.8 %	6.4 %

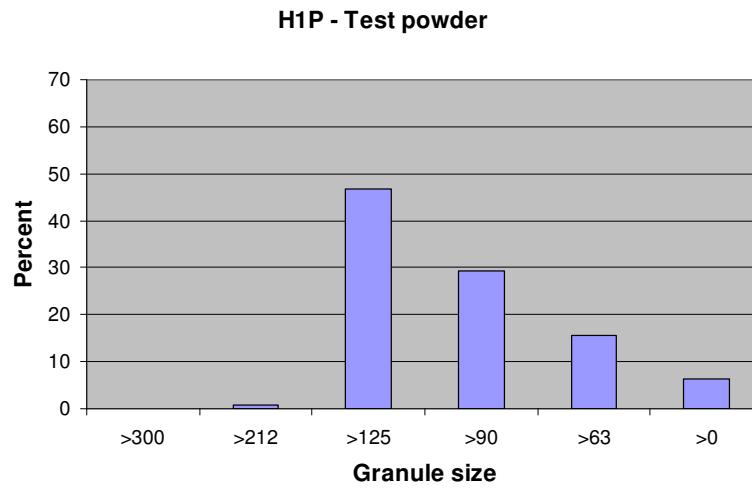


Figure 4.13 – Measured granule size distribution of test powder sample of grade H1P (apparent density of 3.25 g/cm³)

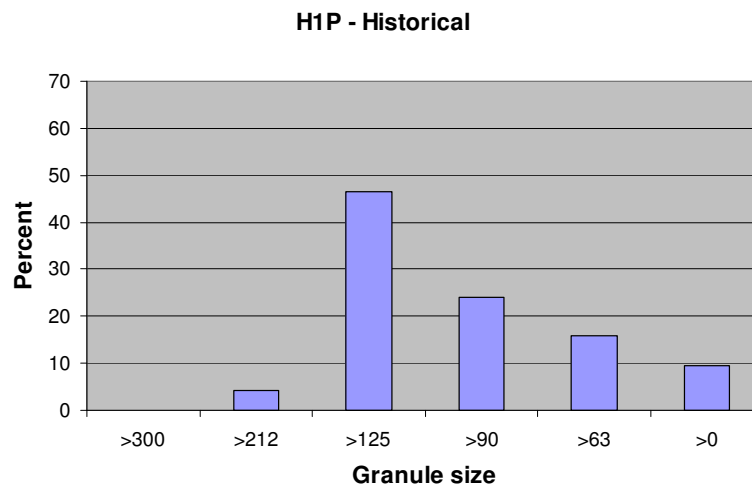


Figure 4.14 – Historical granule size distribution of grade H1P (historical apparent density of 3.21 g/cm³)

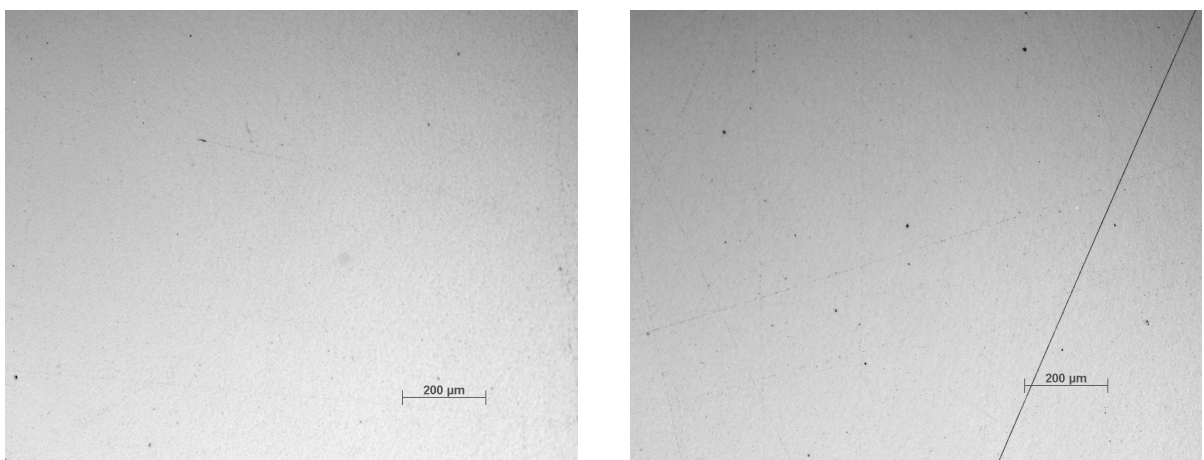


Figure 4.15 – 100x magnification image of polished cross-section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 25 % for grade H1P

4.2.6 Grade ‘H6’ experimental data

‘H6’ grade powder is a wear grade that has 1 to 1.5 micron grain size and relatively low cobalt content (5.6 wt%). It is used where high hardness is required. There are mixed crystals (TaC and NbC). Pressing test sheets, historical screen fractions as well as other test results and reports can be found in Appendix 1-A6.

Table 4.17 - Grade ‘H6’ summary

SD (Grade batch)	14.95 g/cm ³
SD (Test sample)	14.90 g/cm ³
Apparent density (Grade batch)	3.04 g/cm ³
Apparent density (Test powder sample)	2.87 g/cm ³
Volatile content (Test sample)	2.47 %
Best average green density (Measured)	8.08 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	23.4 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.18 – Pressing test results for grade ‘H6’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
3.15	23.5 %	23.1 %	6.40	8.12	82.3 / 329
6.34	23.5 %	23.7 %	12.75	8.04	74.4 / 298
-	-	-	-	-	-

Table 4.19 – Screen fraction report for grade ‘H6’ test powder (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
199.3 g	0.1 g	24.4 g	123.3 g	40.2 g	9.3 g	2.0 g
Percent	0.1 %	12.2 %	61.9 %	20.2 %	4.7 %	1.0 %

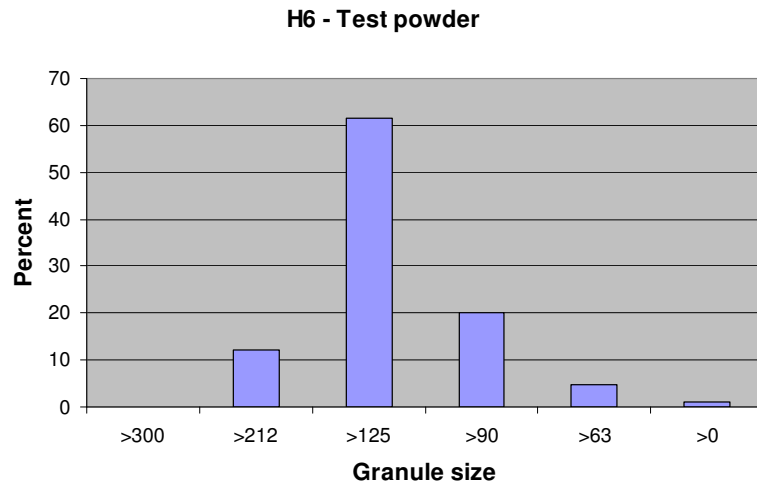


Figure 4.16 – Measured granule size distribution of test powder sample of grade H6 (apparent density of 2.87 g/cm³)

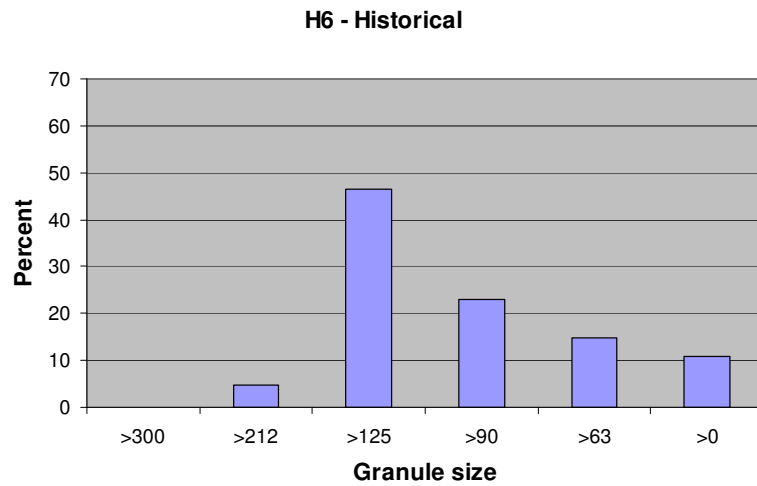


Figure 4.17 – Historical granule size distribution of grade H6 (historical apparent density of 3.41 g/cm³)

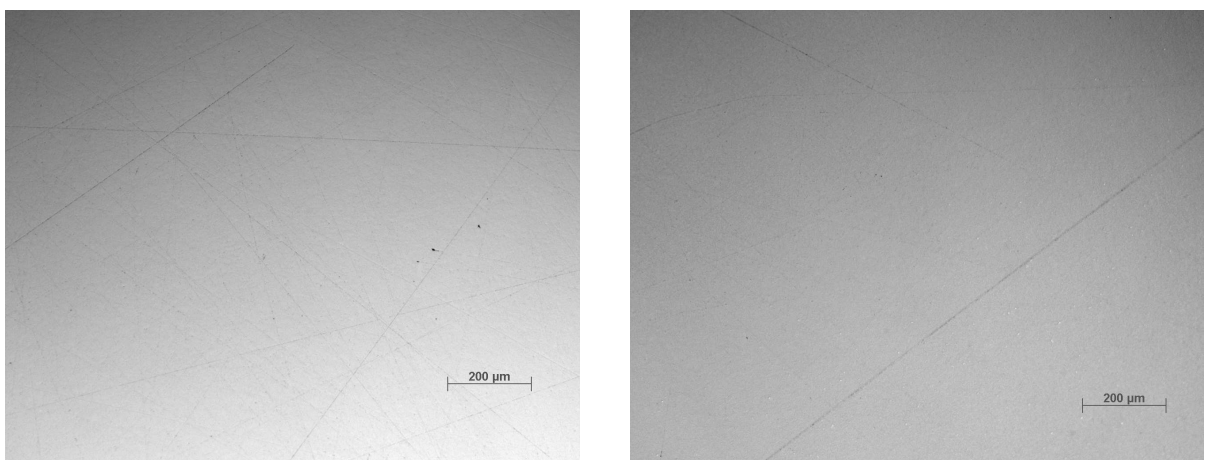


Figure 4.18 – 100x magnification image of polished cross-section of the best shrinkage (least porosity) 23.5 % and the worst shrinkage (most porosity) 25.5 % for grade H6

4.2.7 Grade ‘K15M’ experimental data

‘K15M’ grade powder is a tool grade that has 1-2 μm grain size band and relatively low cobalt content (7.3 wt%). It is used where high hardness is required. It contains mixed crystals (TiC). Pressing test sheets, historical screen fractions as well as other test results and reports can be found in Appendix 1-A7.

Table 4.20 - Grade ‘K15M’ summary

SD (Grade batch)	12.77 g/cm ³
SD (Test sample)	12.82 g/cm ³
Apparent density (Grade batch)	3.23 g/cm ³
Apparent density (Test powder sample)	3.12 g/cm ³
Volatile content (Test sample)	2.44 %
Best average green density (Measured)	6.98 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	23.4 %
Porosity result for ‘best shrinkage’ sintered components	A02 B02

Table 4.21 – Pressing test results for grade ‘K15M’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	23.0 %	23.3 %	3.49	6.98	28.1 / 112
4.00	23.0 %	23.3 %	6.98	6.98	26.2 / 105
8.00	23.0 %	23.6 %	13.95	6.97	24.8 / 99.2

Table 4.22 – Screen fraction report for grade ‘K15M’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
198.9 g	0.1 g	1.6 g	123.4 g	42.8 g	16.5 g	14.5 g
Percent	0.1 %	0.8 %	62.0 %	21.5 %	8.3 %	7.3 %

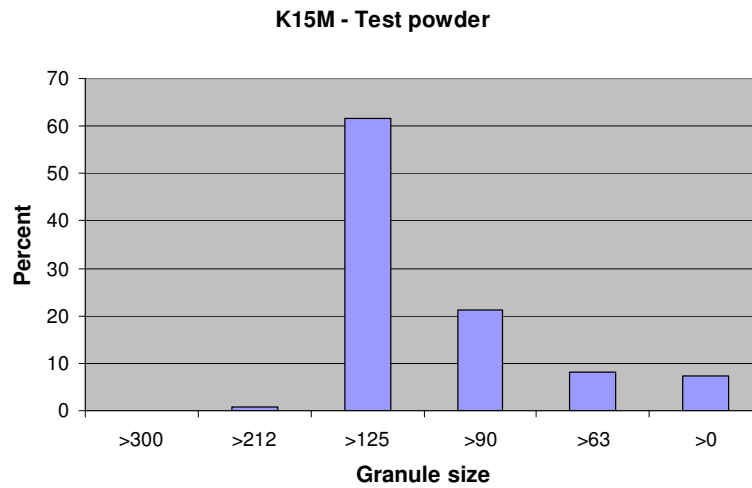


Figure 4.19 – Measured granule size distribution of test powder sample of grade K15M (apparent density of 3.12 g/cm³)

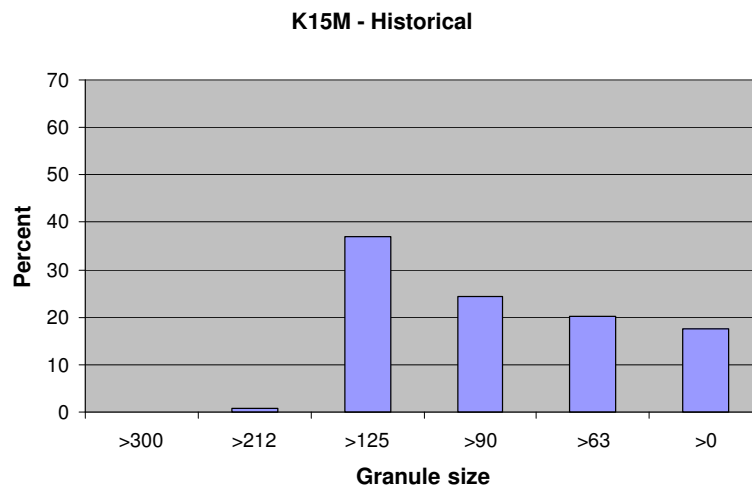


Figure 4.20 – Historical granule size distribution of grade K15M (historical apparent density of 3.19 g/cm³)

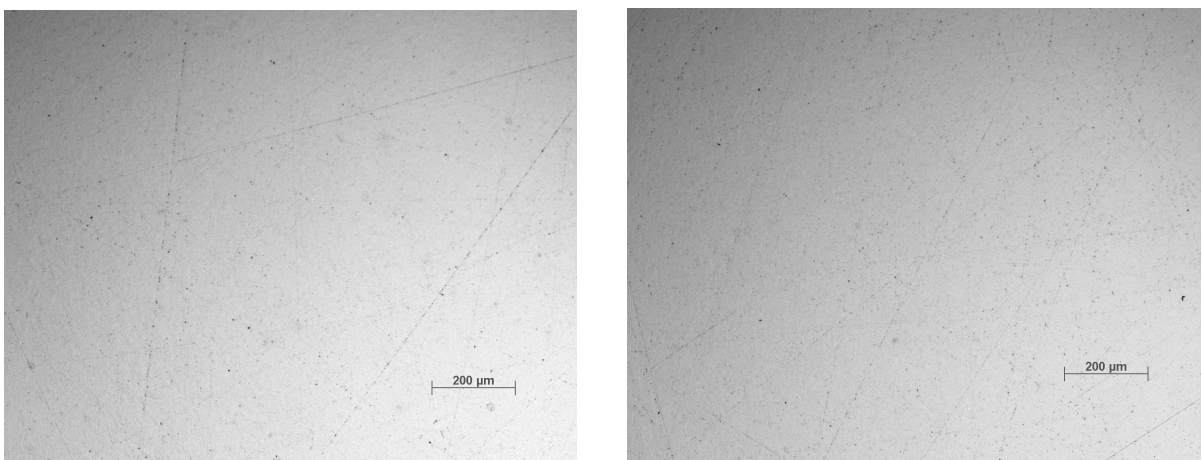


Figure 4.21 – 100x magnification image of polished cross-section of the best shrinkage (least porosity) 23 % and the worst shrinkage (most porosity) 25 % for grade K15M

4.2.8 Grade ‘MT16’ experimental data

‘MT16’ grade powder is a mining grade that has 2-3 μm grain size and high cobalt content (16.0 wt%). It is used where high toughness and wear resistance is required. It contains no mixed crystals. Pressing test sheets as well as other test results and reports can be found in Appendix 1-A8.

Table 4.23 - Grade ‘MT16’ summary

SD (Grade batch)	13.85 g/cm ³
SD (Test sample)	13.82 g/cm ³
Apparent density (Grade batch)	3.39 g/cm ³
Apparent density (Test powder sample)	3.43 g/cm ³
Volatile content (Test sample)	2.75 %
Best average green density (Measured)	7.74 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	22.4 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.24 – Pressing test results for grade ‘MT16’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	22.0 %	22.2 %	3.87	7.74	35.9 / 144
4.00	22.0 %	22.4 %	7.75	7.75	34.5 / 137
8.01	22.0 %	22.6 %	15.50	7.74	33.8 / 135

Table 4.25 – Screen fraction report for grade ‘MT16’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
157.2 g	0.3 g	10.0 g	90.4 g	25.2 g	22.4 g	8.9 g
Percent	0.2 %	6.4 %	57.5 %	16.0 %	14.2 %	5.7 %

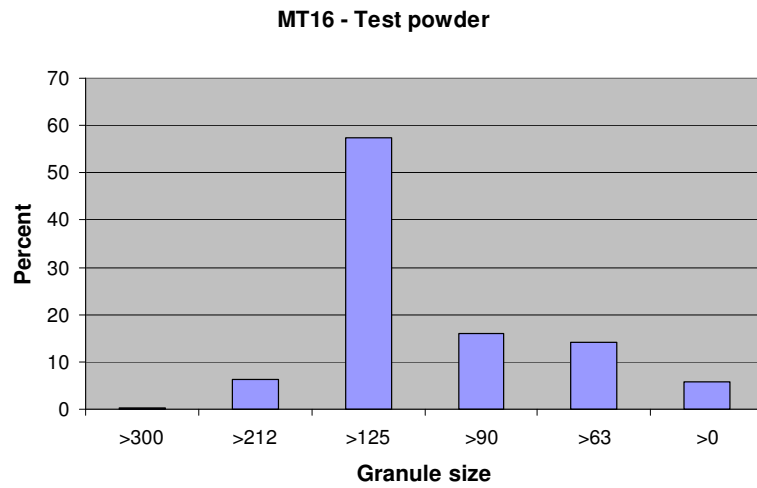


Figure 4.22 –Measured granule size distribution of test powder sample of grade MT16 (apparent density of 3.43 g/cm³)

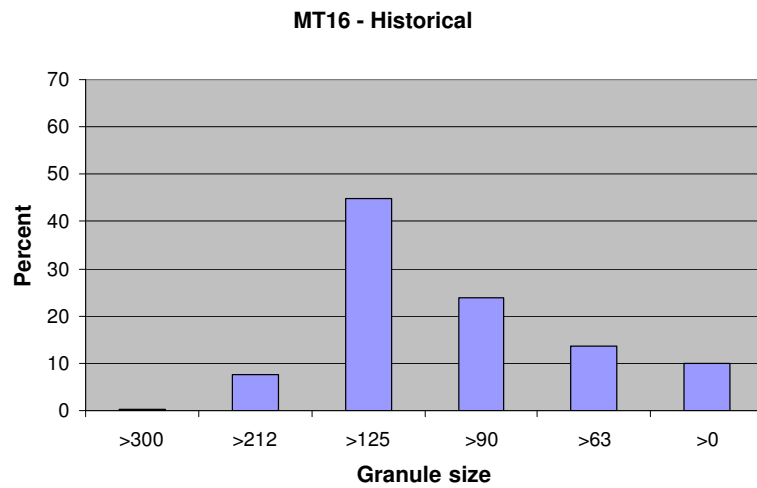


Figure 4.23 – Historical granule size distribution of grade MT16 (historical apparent density of 3.37 g/cm³)

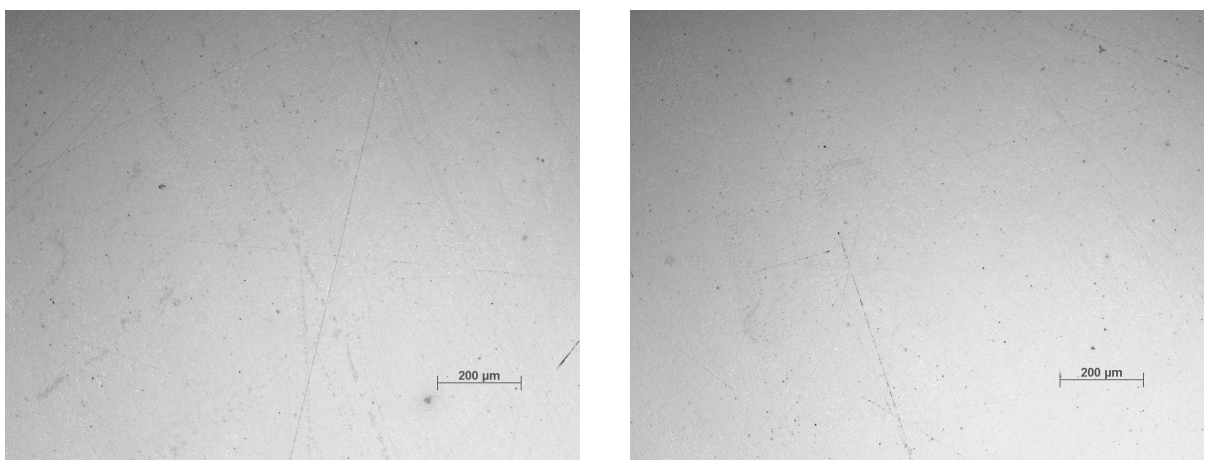


Figure 4.24 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % and the worst shrinkage (most porosity) 24 % for grade MT16

4.2.9 Grade ‘MZ15’ experimental data

‘MZ15’ grade powder is a mining grade that has 2-4 μm grain size and high cobalt content (15.2 wt%). It is used where high toughness and wear resistance is required. It contains no mixed crystals. Pressing test sheets as well as other test results and reports can be found in Appendix 1-A9.

Table 4.26 - Grade ‘MZ15’ summary

SD (Grade batch)	13.92 g/cm ³
SD (Test sample)	13.93 g/cm ³
Apparent density (Grade batch)	3.22 g/cm ³
Apparent density (Test powder sample)	3.37 g/cm ³
Volatile content (Test sample)	2.42 %
Best average green density (Measured)	7.78 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	22.4 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.27 – Pressing test results for grade ‘MZ15’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	22.0 %	22.5 %	3.89	7.78	27.0 / 108
4.00	22.0 %	22.3 %	7.78	7.78	23.7 / 94.8
8.00	22.0 %	22.3 %	15.57	7.78	23.2 / 92.8

Table 4.28 – Screen fraction report for grade ‘MZ15’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
127.0 g	0.2 g	5.8 g	74.5 g	23.6 g	17.5 g	5.4 g
Percent	0.2 %	4.6 %	58.7 %	18.6 %	13.8 %	4.3 %

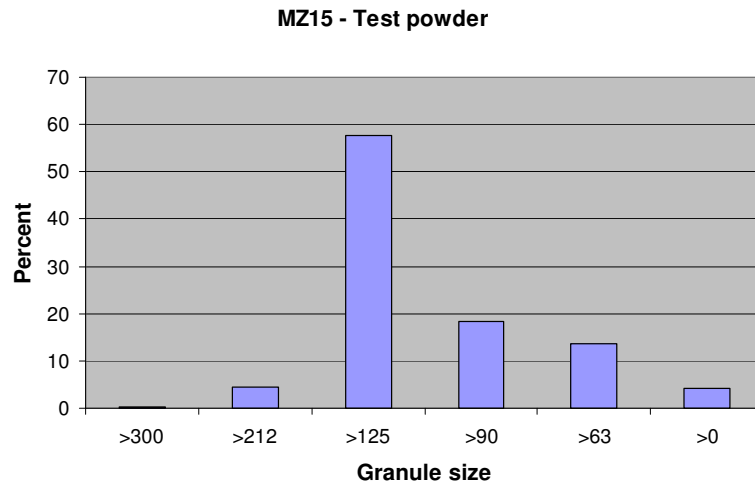


Figure 4.25 – Measured granule size distribution of test powder sample of grade MZ15 (apparent density of 3.37 g/cm³)

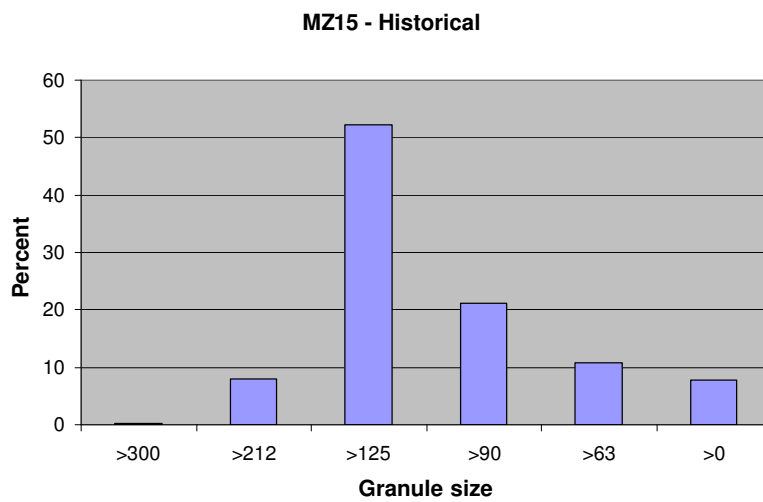


Figure 4.26 – Historical granule size distribution of grade MZ15 (historical apparent density of 3.31 g/cm³)

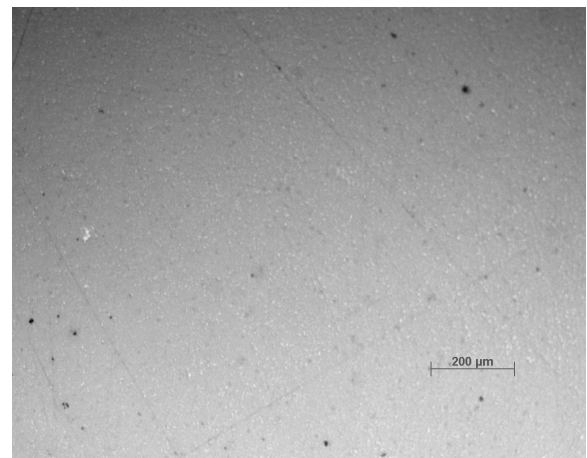
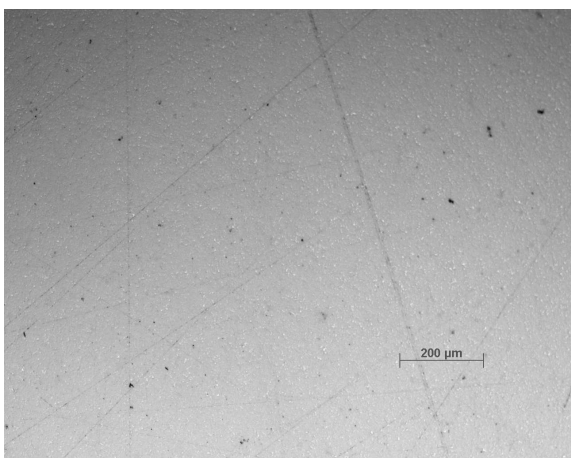


Figure 4.27 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % and the worst shrinkage (most porosity) 23 % for grade MZ15

4.2.10 Grade 'N6 / 194' experimental data

'N6 / 194' grade powder is a wear grade that has 1-3 μm grain size and relatively low cobalt content (6.0 wt%). It is used where high hardness and abrasion resistance is required. It contains no mixed crystals. Pressing test sheets, historical data as well as other test results and reports can be found in Appendix 1-A10.

Table 4.29 - Grade 'N6 / 194' summary

SD (Grade batch)	14.87 g/cm ³
SD (Test samples)	14.89 g/cm ³
Apparent density (Grade batch)	3.60 g/cm ³
Apparent density (Test powder sample)	3.62 g/cm ³
Volatile content (Test samples)	1.94 %
Best average green density (Measured)	8.14 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	22.9 %
Porosity result for 'best shrinkage' sintered components	A00 B00

Table 4.30 – Pressing test results for grade 'N6 / 194'

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.01	22.5 %	22.9 %	4.07	8.10	19.3 / 77
4.00	22.5 %	22.6 %	8.18	8.18	20.5 / 82
8.00	22.5 %	23.0 %	16.31	8.15	19.9 / 80

Table 4.31 – Screen fraction report for grade 'N6 / 194' test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
126.9 g	0.4 g	2.6 g	82.8 g	14.5 g	19.1 g	7.5 g
Percent	0.3 %	2.0 %	65.2 %	11.4 %	15.1 %	5.9 %

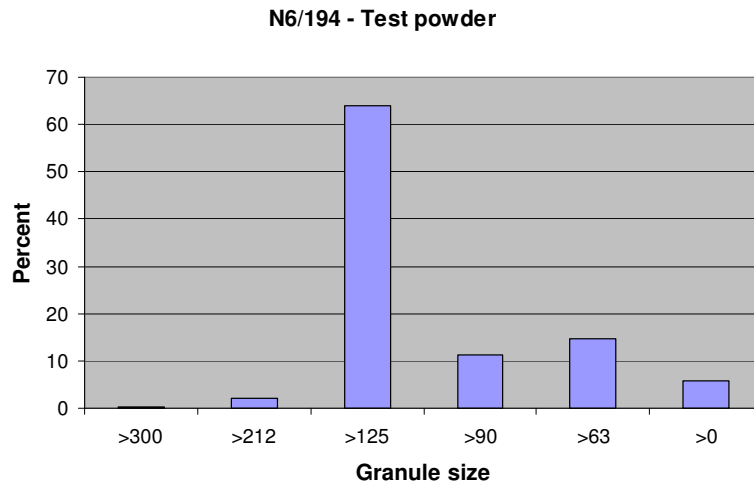


Figure 4.28 – Measured granule size distribution of test powder sample of grade N6 / 194 (apparent density of 3.62 g/cm³)

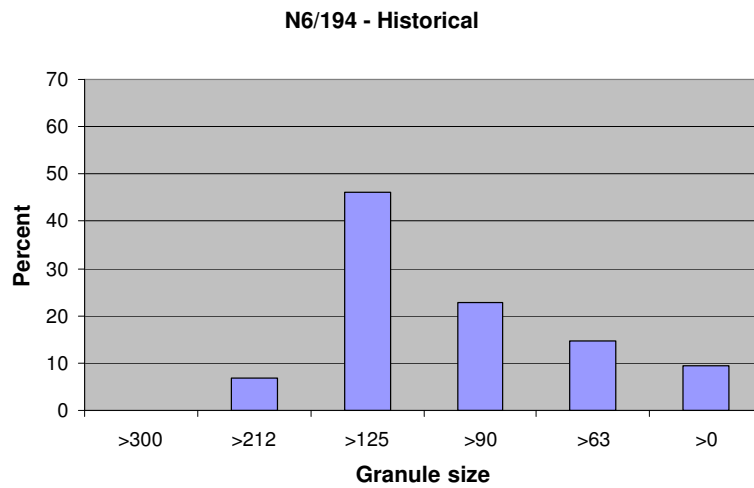


Figure 4.29 – Historical granule size distribution of grade N6 / 194 (historical apparent density of (3.72 g/cm³))

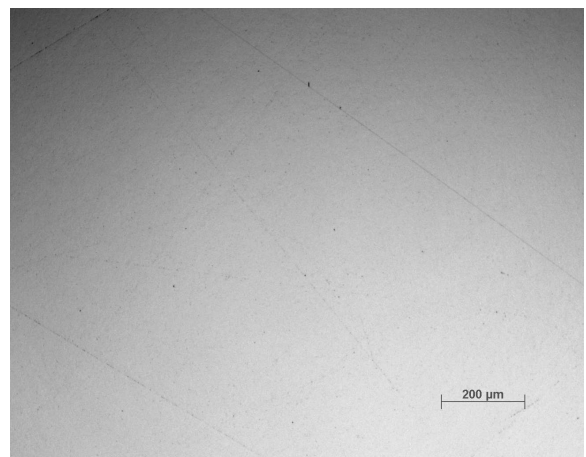
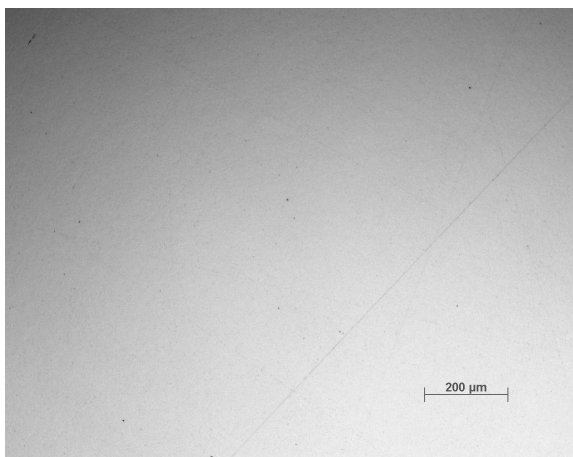


Figure 4.30 – 100x magnification image of polished section of the best shrinkage (least porosity) 22.5 % and the worst shrinkage (most porosity) 24 % for grade N6 / 194

4.2.11 Grade ‘P7’ experimental data

‘P7’ grade powder is a mining grade that has 2-6 μm grain size and relatively low cobalt content (7.5 wt%). It is used where high hardness is required. It contains no mixed crystals. Pressing test sheets as well as other test results and reports can be found in Appendix 1-A11.

Table 4.32 - Grade ‘P7’ summary

SD (Grade batch)	14.73 g/cm ³
SD (Test samples)	14.75 g/cm ³
Apparent density (Grade batch)	3.80 g/cm ³
Apparent density (Test powder samples)	3.38 g/cm ³
Volatile content (Test sample)	2.26 %
Best average green density (Measured)	8.26 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	22.2 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.33 – Pressing test results for grade ‘P7’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	22.0 %	22.6 %	4.10	8.20	21.7 / 87
4.00	22.0 %	22.3 %	8.20	8.20	19.2 / 77
8.00	22.5 %	23.0 %	16.31	8.15	19.9 / 80

Table 4.34 – Screen fraction report for grade ‘N6 / 194’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
99.1 g	0.4 g	1.2 g	25.7 g	29.7 g	20.8 g	21.3 g
Percent	0.4 %	1.2 %	25.9 %	30.0 %	21.0 %	21.5 %

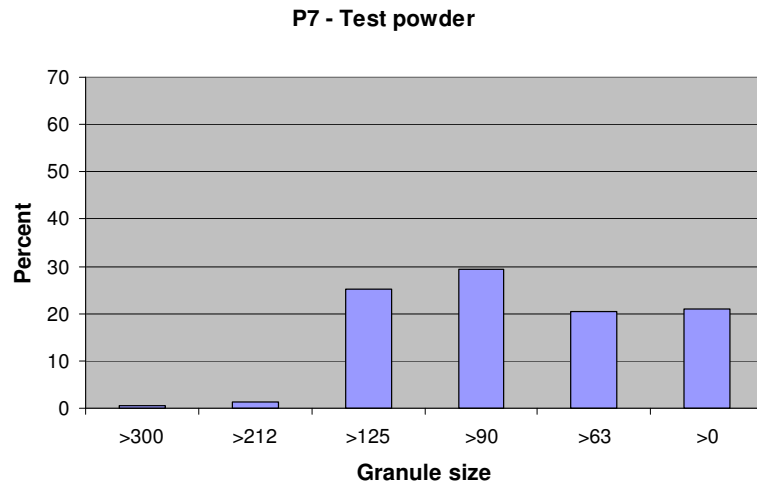


Figure 4.31 – Measured granule size distribution of test powder sample of grade P7 (apparent density of 3.38 g/cm³)

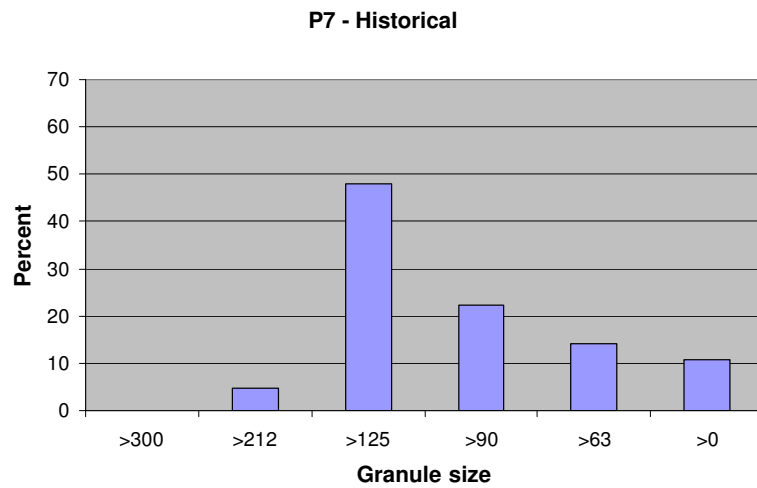


Figure 4.32 – Historical granule size distribution of grade P7 (historical apparent density of 3.72 g/cm³)

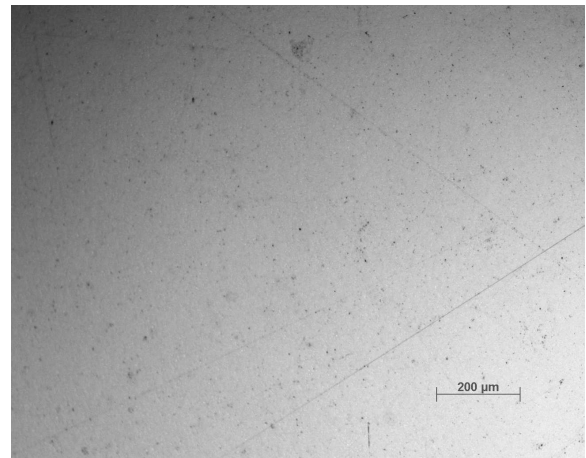
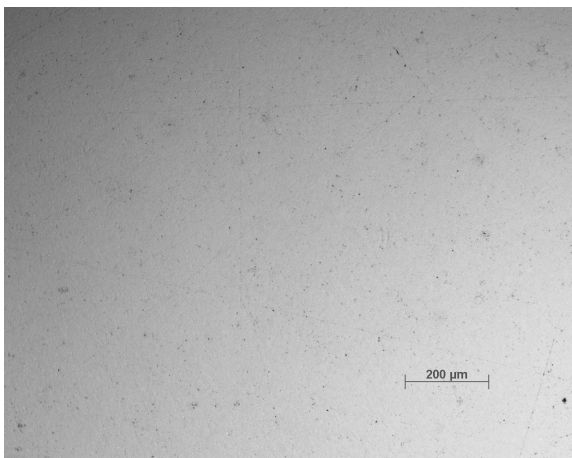


Figure 4.33 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % and the worst shrinkage (most porosity) 23 % for grade P7

4.2.12 Grade ‘P25M’ experimental data

‘P25M’ grade powder is a tool grade that has 1-2 μm grain size band and 9.5 wt% cobalt content. It is used where high hardness and toughness is required. It contains mixed crystals (TiC, TaC and NbC). Pressing test sheets, historical data as well as other test results and reports can be found in Appendix 1-A12.

Table 4.35 - Grade ‘P25M’ summary

SD (Grade batch)	12.59 g/cm ³
SD (Test samples)	12.56 g/cm ³
Apparent density (Grade batch)	3.12 g/cm ³
Apparent density (Test powder sample)	3.01 g/cm ³
Volatile content (Test sample)	2.38 %
Best average green density	7.22 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.1 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.36 – Pressing test results for grade ‘P25M’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	20.0 %	20.7 %	3.65	7.30	36.7 / 147
4.00	20.0 %	20.6 %	7.29	7.29	32.5 / 130
8.00	21.5 %	22.0 %	14.17	7.08	22.6 / 90

Table 4.37 – Screen fraction report for grade ‘P25M’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
197.0 g	0.1 g	1.7 g	92.8 g	50.0 g	33.3 g	19.1 g
Percent	0.1 %	0.9 %	47.1 %	25.4 %	16.9 %	9.7 %

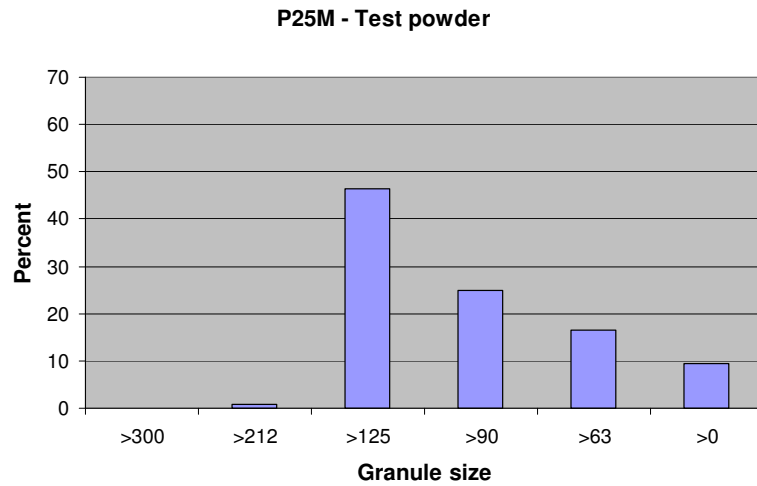


Figure 4.34 – Measured granule size distribution of test powder sample of grade P25M (apparent density of 3.01 g/cm³)

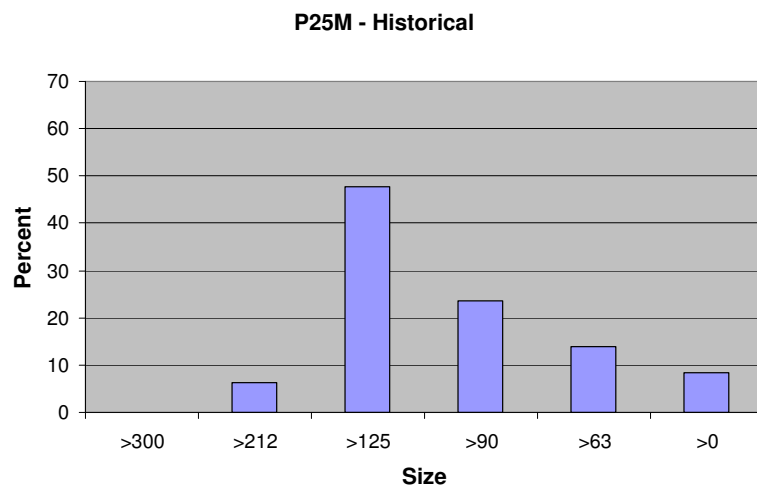


Figure 4.35 – Historical granule size distribution of grade P25M (historical apparent density of 3.14 g/cm³)

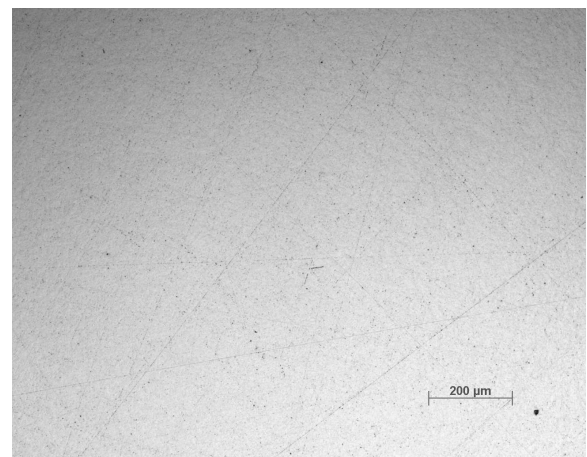
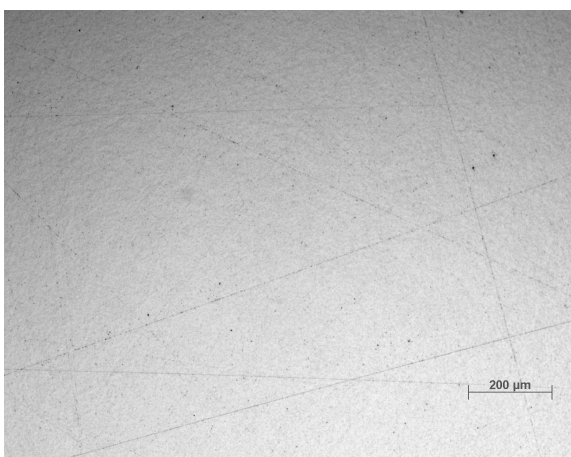


Figure 4.36 – 100x magnification image of polished section of the best shrinkage (least porosity) 20 % and the worst shrinkage (most porosity) 24 % for grade P25M

4.2.13 Grade ‘P30M’ experimental data

‘P30M’ grade powder is a tool grade that has 2-3 μm grain size and 10 wt% cobalt content. It is used for cutting inserts where a degree of toughness is required. It contains mixed crystals (TiC, TaC and NbC). Pressing test sheets, historical data as well as other test results and reports can be found in Appendix 1-A13.

Table 4.38 - Grade ‘P30M’ summary

SD (Grade batch)	12.38 g/cm ³
SD (Test samples)	12.49 g/cm ³
Apparent density (Grade batch)	3.10 g/cm ³
Apparent density (Test powder sample)	2.91 g/cm ³
Volatile content (Test samples)	2.51 %
Best average green density	7.09 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.8 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.39 – Pressing test results for grade ‘P30M’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	20 %	21.1 %	3.60	7.20	36.9 / 148
4.00	20 %	22.2 %	7.03	7.03	25.2 / 100
8.00	21 %	22.2 %	14.10	7.05	24.7 / 99

Table 4.40 – Screen fraction report for grade ‘P30M’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
211.0 g	0.4 g	36.9 g	118.6 g	33.3 g	15.6 g	6.2 g
Percent	0.2 %	17.5 %	56.2 %	15.8 %	7.4 %	2.9 %

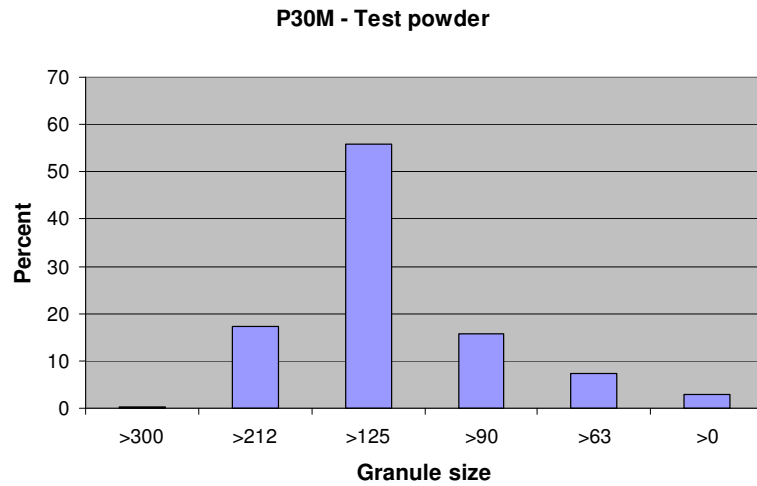


Figure 4.37 – Measured granule size distribution of test powder sample of grade P30M (apparent density of 2.91 g/cm³)

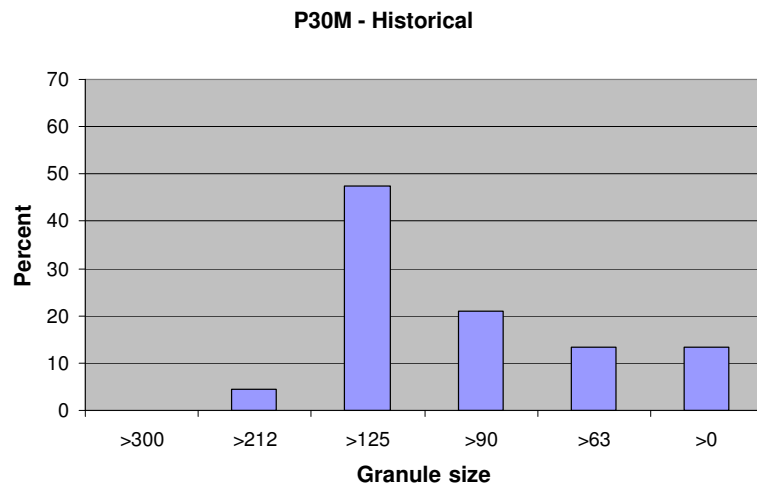


Figure 4.38 – Historical granule size distribution of grade P30M (historical apparent density of 3.09 g/cm³)

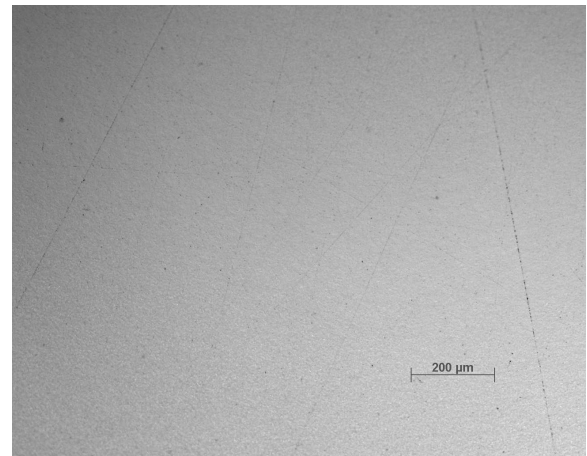
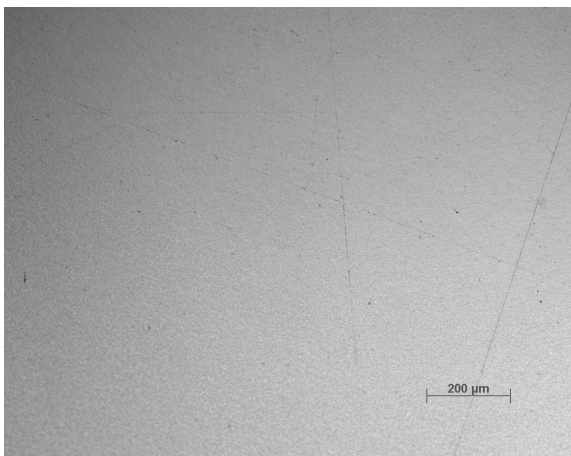


Figure 4.39 – 100x magnification image of polished section of the best shrinkage (least porosity) 20 % and the worst shrinkage (most porosity) 24 % for grade P30M

4.2.14 Grade 'P40M' experimental data

'P40M' grade powder is a tool grade that has 2-3 μm grain size and 10 wt% cobalt content. It contains mixed crystals (TiC, TaC and NbC). Pressing test sheets as well as other test results and reports can be found in Appendix 1-A14.

Table 4.41 - Grade 'P40M' summary

SD (Grade batch)	13.25 g/cm ³
SD (Test samples)	13.26 g/cm ³
Apparent density (Grade batch)	3.23 g/cm ³
Apparent density (Test powder samples)	3.09 g/cm ³
Volatile content (Test sample)	2.45 %
Best average green density	7.50 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.7 %
Porosity result for 'best shrinkage' sintered components	A00 B00

Table 4.42 – Pressing test results for grade 'P40M'

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	21.5 %	21.7 %	3.74	7.48	30.2 / 121
4.00	21.0 %	21.4 %	7.54	7.56	30.4 / 122
8.00	21.5 %	22.1 %	14.93	7.46	26.0 / 104

Table 4.43 – Screen fraction report for grade 'P40M' test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
196.8 g	0.2 g	16.8 g	108.3 g	43.0 g	20.9 g	7.6 g
Percent	0.1 %	8.5 %	55.0 %	21.9 %	10.6 %	3.9 %

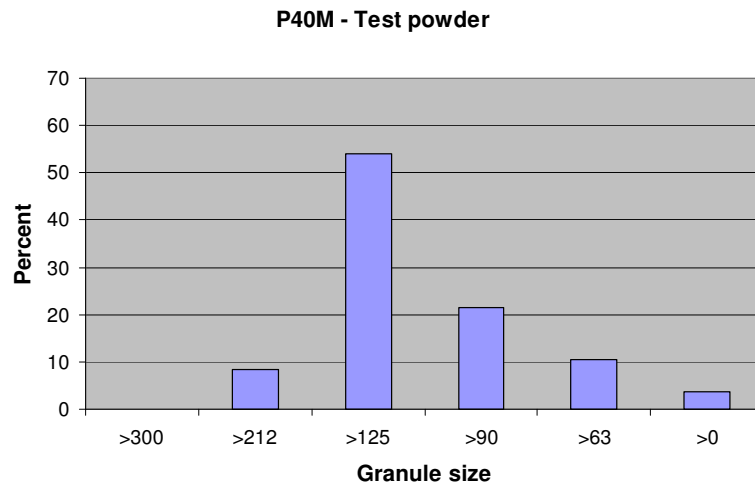


Figure 4.40 – Measured granule size distribution of test powder sample of grade P40M (apparent density of 3.09 g/cm³)

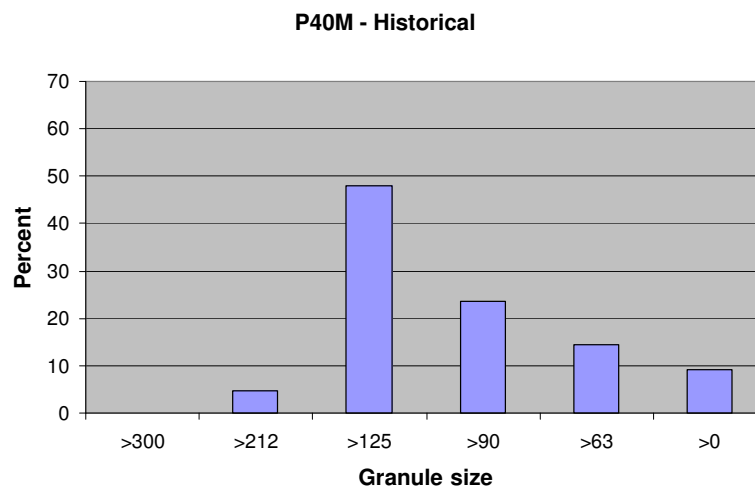


Figure 4.41 – Historical granule size distribution of grade P40M (historical apparent density of 3.26 g/cm³)

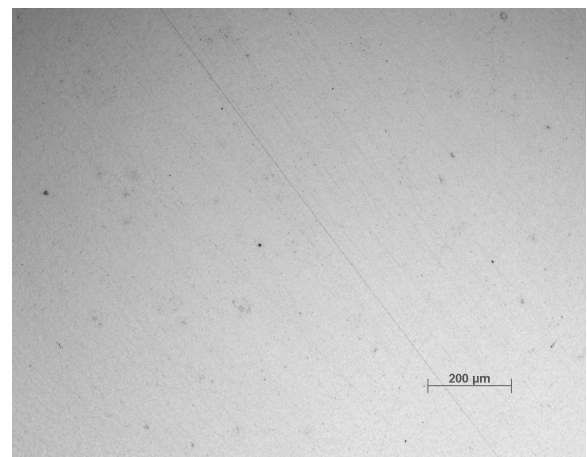
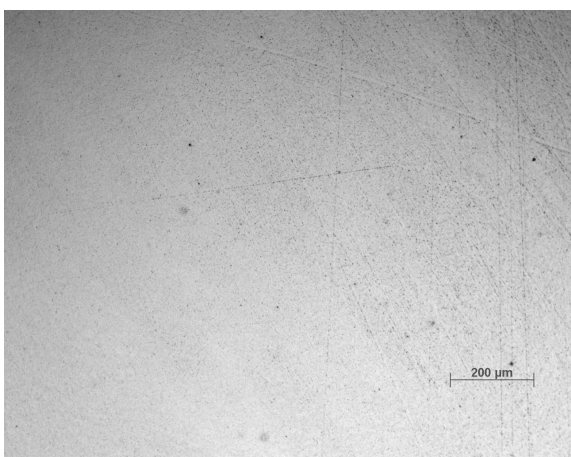


Figure 4.42 – 100x magnification image of polished section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 24 % for grade P40M

4.2.15 Grade ‘P511’ experimental data

‘P511’ grade powder is a mining grade that has 3-6 μm grain size and relatively high cobalt content (11.7 wt%). It is used where toughness is required. It contains mixed no crystals. Pressing test sheets as well as other test results and reports can be found in Appendix 1-A15.

Table 4.44 - Grade ‘P511’ summary

SD (Grade batch)	14.26 g/cm ³
SD (Test samples)	14.25 g/cm ³
Apparent density (Grade batch)	N/A
Apparent density (Test powder sample)	3.41 g/cm ³
Volatile content (Test samples)	2.89 %
Best average green density	8.01 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.6 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.45 – Pressing test results for grade ‘P511’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	22.0 %	22.0 %	3.98	7.96	22.3 / 89
4.00	22.0 %	22.2 %	7.97	7.97	20.9 / 84
8.00	20.0 %	20.7 %	16.62	8.31	29.6 / 118

Table 4.46 – Screen fraction report for grade ‘P511’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
117.5 g	0.1 g	2.7 g	63.2 g	34.8 g	14.6 g	2.1 g
Percent	0.1 %	2.3 %	53.8 %	29.6 %	12.4 %	1.8 %

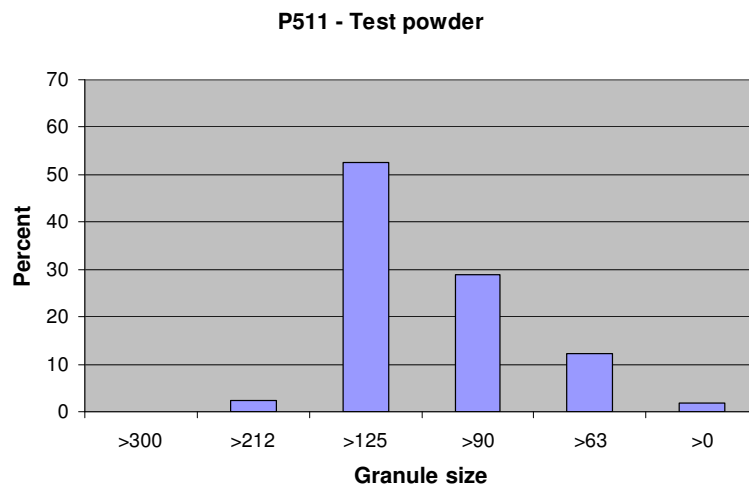


Figure 4.43 – Measured granule size distribution of test powder sample of grade P511 (apparent density of 3.41 g/cm³)

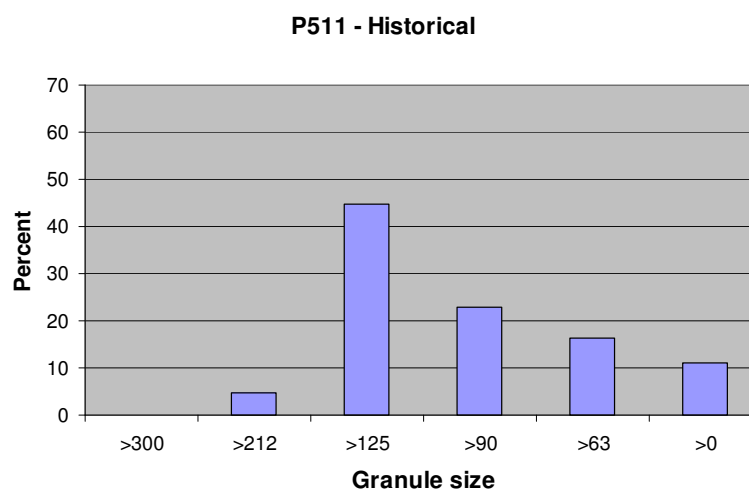


Figure 4.44 – Historical granule size distribution of grade P511 (historical apparent density of 3.53 g/cm³)

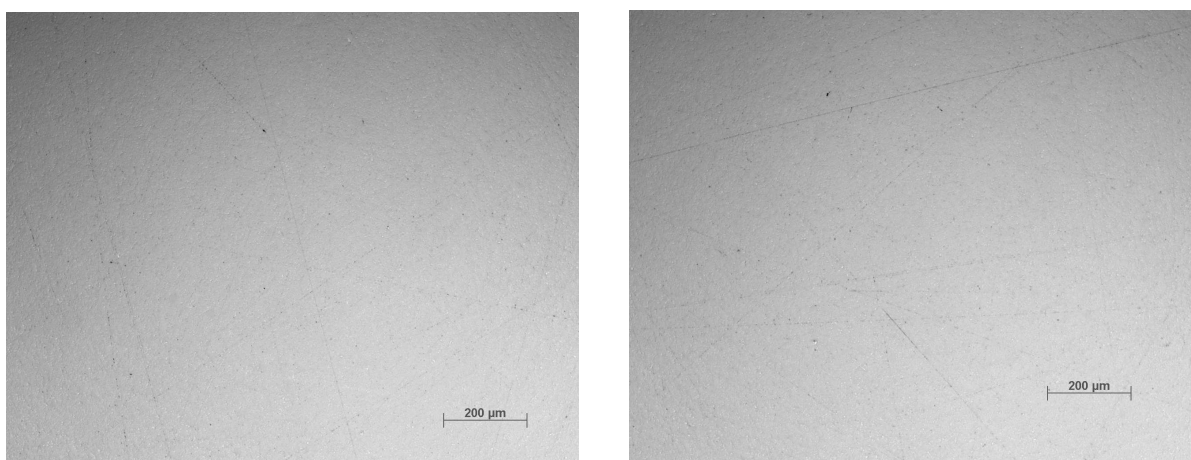


Figure 4.45 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % (left) and the worst shrinkage (most porosity) 24 % for grade P511

4.2.16 Grade ‘PBF’ experimental data

‘PBF’ grade powder is a mining grade that has 2-3 μm grain size and relatively low cobalt content (5.9 wt%). It is used where wear resistance is required. It contains no mixed crystals (straight grade). Pressing test sheets as well as other test results and reports can be found in Appendix 1-A16.

Table 4.47 - Grade ‘PBF’ summary

SD (Grade batch)	14.94 g/cm ³
SD (Test samples)	14.93 g/cm ³
Apparent density (Grade batch)	3.95 g/cm ³
Apparent density (Test powder sample)	3.86 g/cm ³
Volatile content (Test samples)	2.35 %
Best average green density	8.57 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.1 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.48 – Pressing test results for grade ‘PBF’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	20.0 %	20.2 %	4.33	8.66	27.9 / 112
4.00	20.5 %	21.2 %	8.58	8.58	21.7 / 87
8.02	21.0 %	21.9 %	16.98	8.47	17.9 / 72

Table 4.49 – Screen fraction report for grade ‘PBF’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
149.3 g	0.1 g	2.9 g	41.4 g	41.0 g	35.6 g	28.3 g
Percent	0.1 %	1.9%	27.7 %	27.5 %	23.8 %	19.0 %

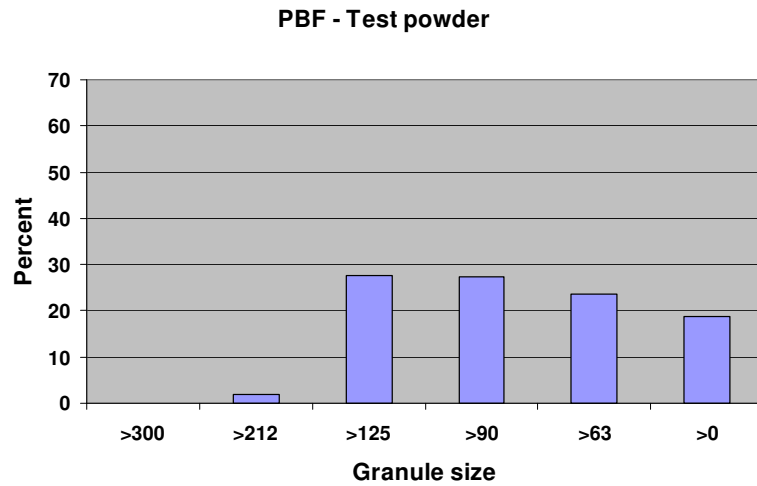


Figure 4.46 – Measured granule size distribution of test powder sample of grade PBF (apparent density of 3.86 g/cm³)

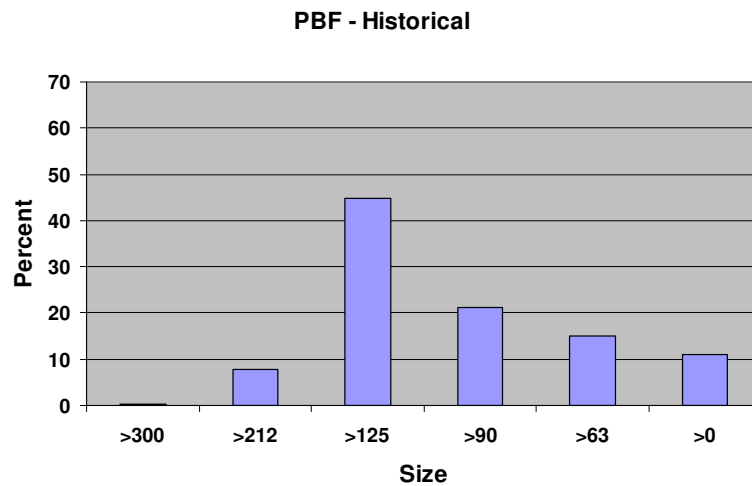


Figure 4.47 – Historical granule size distribution of grade PBF (historical apparent density of 3.70 g/cm³)

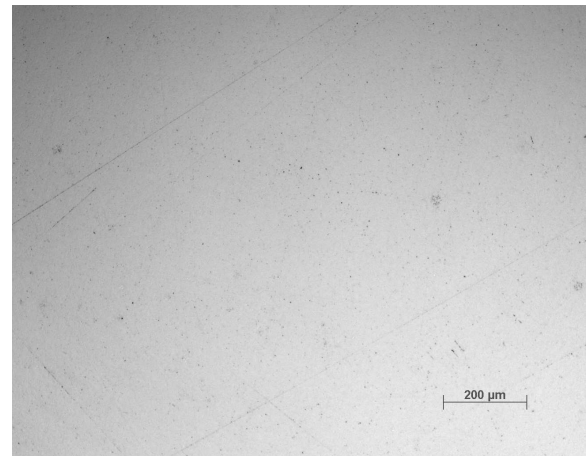
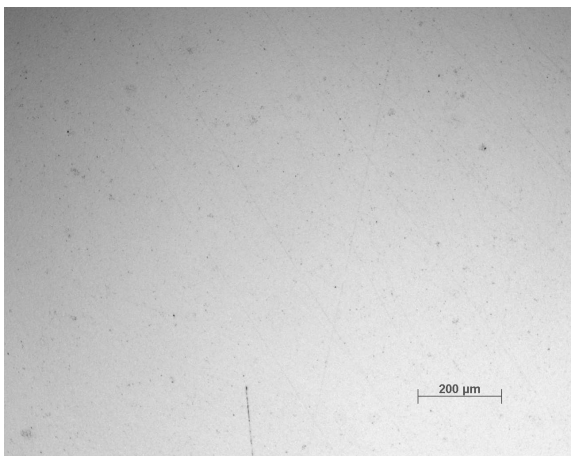


Figure 4.48 – 100x magnification image of polished section of the best shrinkage (least porosity) 21.5 % and the worst shrinkage (most porosity) 24 % for grade PBF

4.2.17 Grade ‘PBT’ experimental data

‘PBT’ grade powder is a mining grade (very similar to PBF) that has 2-4 μm grain size and relatively low cobalt content (6.0 wt%). It is used where wear resistance is required. It contains mixed no crystals. This is an important grade as it is very widely used at Powder Industries. Pressing test sheets as well as other test results and reports can be found in Appendix 1-A17.

Table 4.50 - Grade ‘PBT’ summary

SD (Grade batch)	14.96 g/cm ³
SD (Test samples)	14.98 g/cm ³
Apparent density (Grade batch)	3.80 g/cm ³
Apparent density (Test powder sample)	3.88 g/cm ³
Volatile content (Test samples)	2.17 %
Best average green density	8.68 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	20.2 %
Porosity result for ‘best shrinkage’ sintered components	A02 B02

Table 4.51 – Pressing test results for grade ‘PBT’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	20.0 %	19.8 %	4.35	8.70	29.6 / 118
4.00	20.0 %	20.3 %	8.68	8.68	28.5 / 114
8.01	20.0 %	20.5 %	17.38	8.68	26.8 / 107

Table 4.52 – Screen fraction report for grade ‘PBT’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
106.9 g	0.3 g	8.0 g	68.5 g	18.0 g	8.0 g	4.1 g
Percent	0.3 %	7.5 %	64.1 %	16.8 %	7.5 %	3.8 %

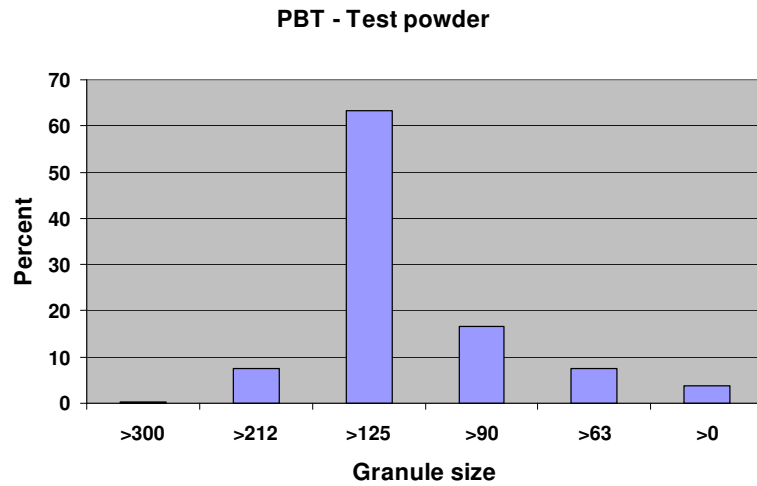


Figure 4.49 – Measured granule size distribution of test powder sample of grade PBT (apparent density of 3.88 g/cm³)

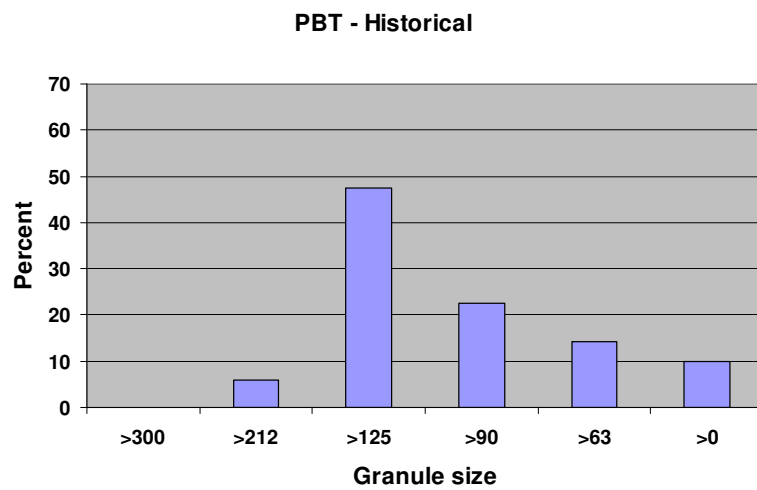


Figure 4.50 – Historical granule size distribution of grade PBT (historical apparent density of 3.84 g/cm³)

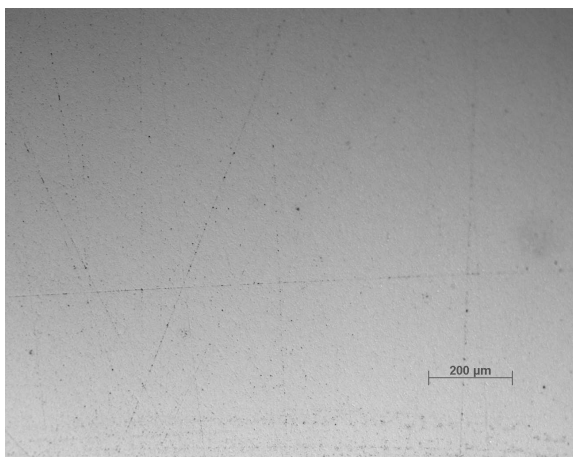


Figure 4.51 – 100x magnification image of polished section of the best shrinkage (least porosity) 20 % and the worst shrinkage (most porosity) 24 % for grade PBT

4.2.18 Grade ‘PC20’ experimental data

‘PC20’ grade powder is a tool grade that has 1-3 μm grain size and relatively low cobalt content (6.0 wt%). It is used where high hardness is required. It contains mixed crystals (TiC, TaC and NbC). Pressing test sheets as well as other test results and reports can be found in Appendix 1-A18.

Table 4.53 - Grade ‘PC20’ summary

SD (Grade batch)	14.66 g/cm ³
SD (Test sample)	14.56 g/cm ³
Apparent density (Grade batch)	3.71 g/cm ³
Apparent density (Test powder sample)	3.55 g/cm ³
Volatile content (Test sample)	2.55 %
Best average green density (Measured)	8.35 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.0 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.54 – Pressing test results for grade ‘PC20’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	21.0 %	20.9 %	4.17	8.34	34.1 / 136
4.00	21.0 %	20.9 %	8.36	8.36	35.9 / 144
8.00	21.0 %	21.2 %	16.70	8.35	36.5 / 146

Table 4.55 – Screen fraction report for grade ‘PC20’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
148.4 g	0.1 g	3.0 g	71.6 g	37.4 g	22.1 g	14.2 g
Percent	0.1 %	2.0 %	48.3 %	25.2 %	14.9 %	9.6 %

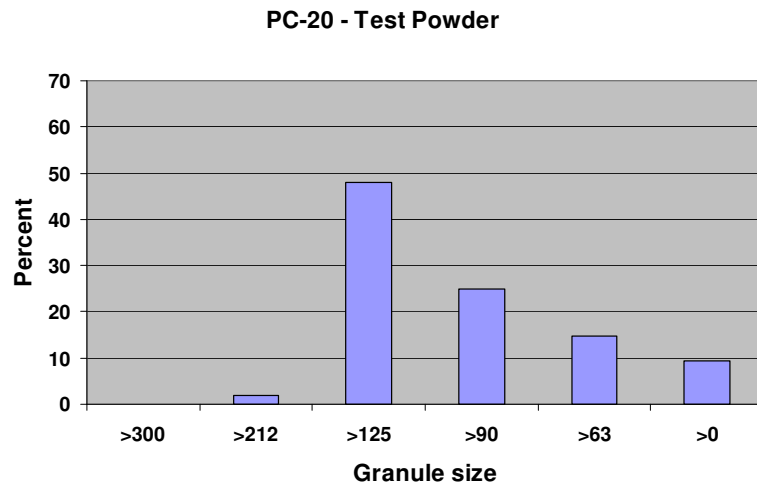


Figure 4.52 – Measured granule size distribution of test powder sample of grade PC20 (apparent density of 3.55 g/cm³)

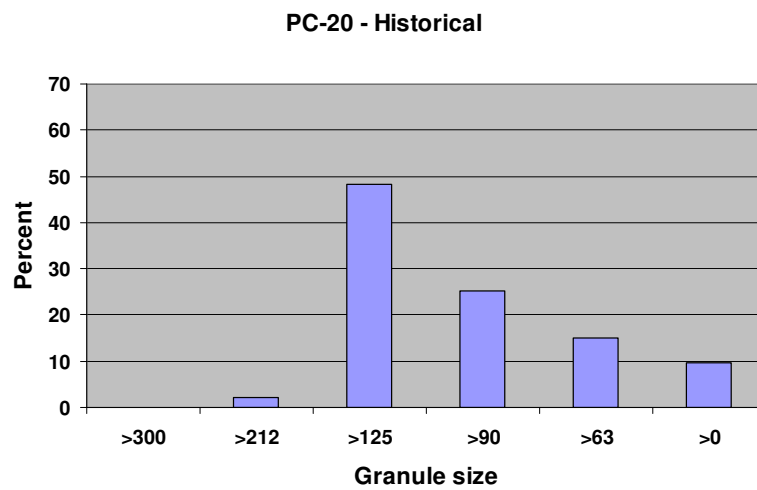


Figure 4.53 – Historical granule size distribution of grade PC20 (apparent density of 3.71 g/cm³)

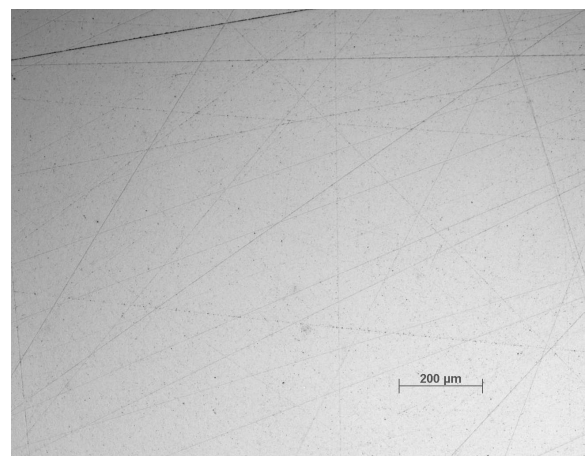
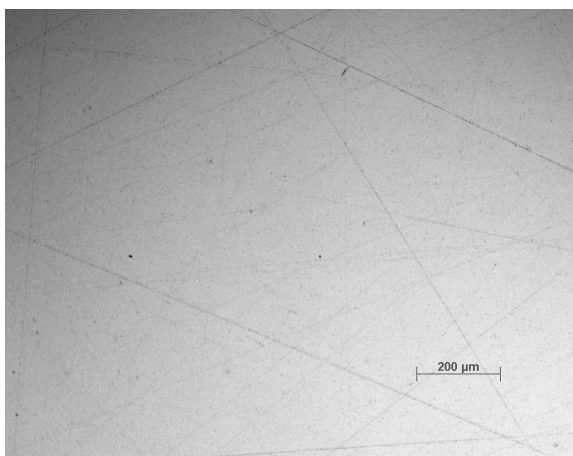


Figure 4.54 – 100x magnification image of polished section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 23 % for grade PC20

4.2.19 Grade 'PI15' experimental data

'PI15' grade powder is a tool grade that has 2-3 μm grain size band and relatively low cobalt content (6.0 wt%). It is used in the manufacture of cutting inserts. It contains mixed crystals (TiC, TaC and NbC). Pressing test sheets, historical data as well as other test results and reports can be found in Appendix 1-A19.

Table 4.56 - Grade 'PI15' summary

SD (Grade batch)	14.02 g/cm ³
SD (Test samples)	14.08 g/cm ³
Apparent density (Grade batch)	3.46 g/cm ³
Apparent density (Test powder sample)	3.40 g/cm ³
Volatile content (Test samples)	2.30 %
Best average green density	7.98 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.6 %
Porosity result for 'best shrinkage' sintered components	A00 B00

Table 4.57 – Pressing test results for grade 'PI15'

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.01	21.0 %	21.5%	4.01	7.98	30.8 / 123
4.00	21.0 %	21.5 %	7.98	7.98	28.6 / 114
8.00	21.0 %	21.7 %	15.96	7.98	28.5 / 114

Table 4.58 – Screen fraction report for grade 'PI15' test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
153.3 g	0.1 g	0.7 g	86.2 g	33.4 g	23.6 g	9.3 g
Percent	0.1 %	0.5 %	56.2 %	21.8 %	15.4 %	6.1 %

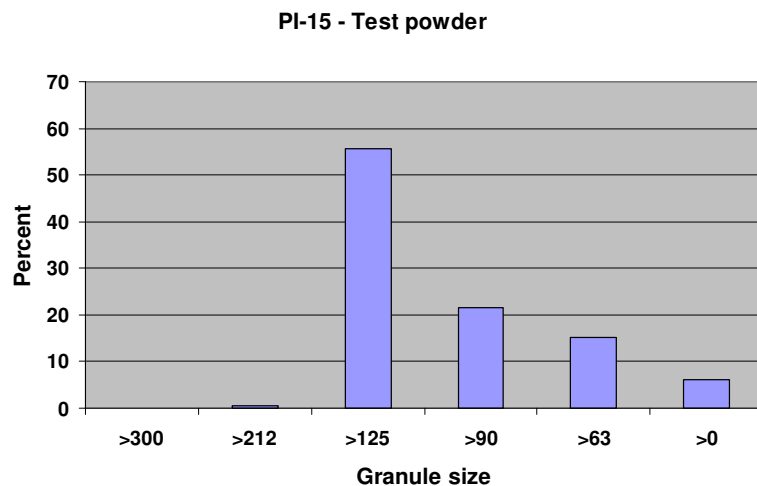


Figure 4.55 – Measured granule size distribution of test powder sample of grade PI15 (apparent density of 3.40 g/cm³)

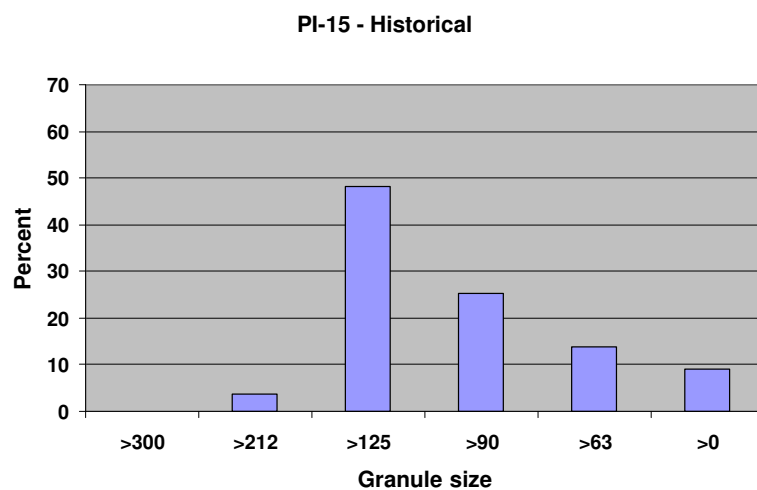


Figure 4.56 – Historical granule size distribution of grade PI15 (historical apparent density of 3.52 g/cm³)

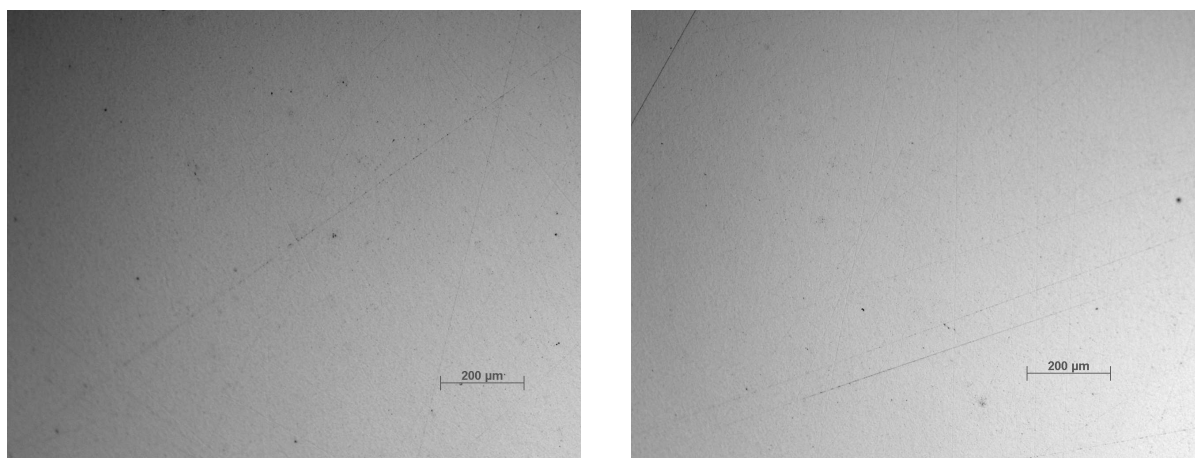


Figure 4.57 – 100x magnification image of polished section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 24 % for grade PI15

4.2.20 Grade 'PI25' experimental data

'PI-25' grade powder is a tool grade that has 2-3 μm grain size and relatively low cobalt content (6.6 wt%). It is used where high hardness is required and is used to make products such as cutting inserts. It contains mixed crystals (TiC, TaC and NbC). Pressing test sheets, historical data as well as other test results and reports can be found in Appendix 1-A20.

Table 4.59 - Grade 'PI25' summary

SD (Grade batch)	14.11 g/cm ³
SD (Test sample)	14.08 g/cm ³
Apparent density (Grade batch)	3.48 g/cm ³
Apparent density (Test powder sample)	3.37 g/cm ³
Volatile content (Test sample)	2.23 %
Best average green density (Measured)	7.94 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	21.7 %
Porosity result for 'best shrinkage' sintered components	A00 B00

Table 4.60 – Pressing test results for grade 'PI25'

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	21.0 %	21.1 %	4.01	8.02	34.0 / 136
4.01	21.0 %	21.6 %	8.00	7.98	27.7 / 111
8.00	22.0 %	22.4 %	15.68	7.84	21.8 / 87

Table 4.61 – Screen fraction report for grade 'PI25' test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
168.1 g	0.1 g	3.6 g	82.7 g	45.6 g	23.7 g	12.4 g
Percent	0.1 %	2.1 %	49.2 %	27.1 %	14.1 %	7.4 %

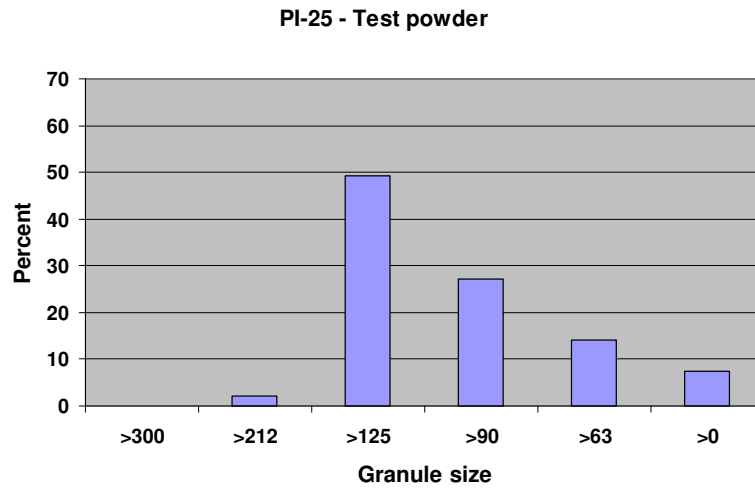


Figure 4.58 – Measured granule size distribution of test powder sample of grade PI25 (apparent density of 3.37 g/cm³)

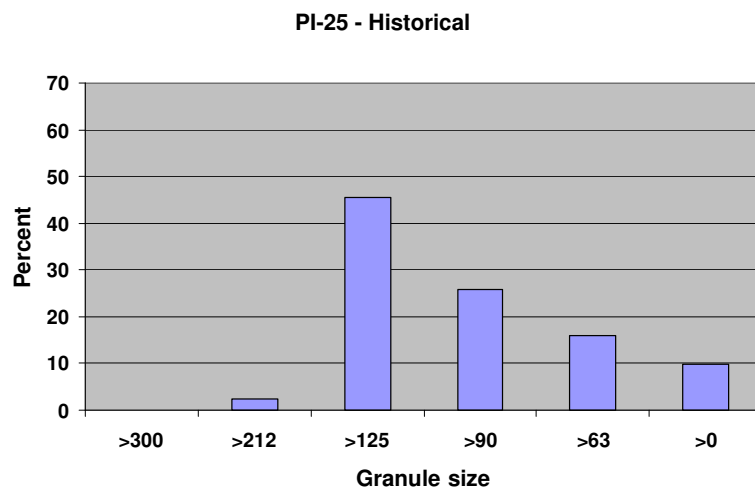


Figure 4.59 – Historical granule size distribution of grade PI25 (historical apparent density of 3.53 g/cm³)

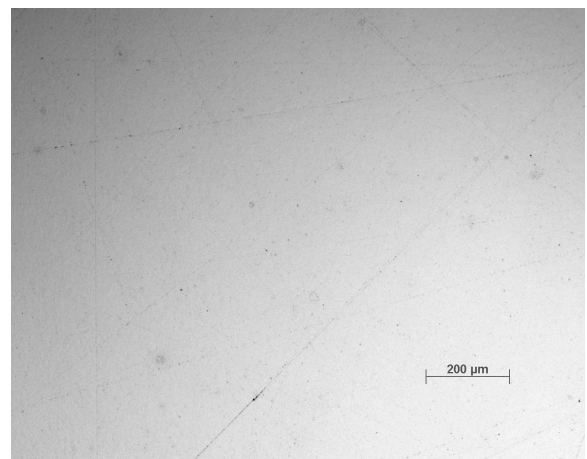
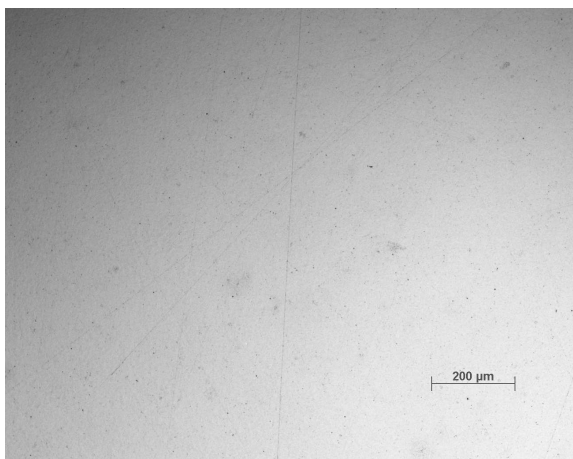


Figure 4.60 – 100x magnification image of polished section of the best shrinkage (least porosity) 21 % and the worst shrinkage (most porosity) 24 % for grade PI25

4.2.21 Grade ‘PI-40’ experimental data

‘PI-40’ grade powder is a tool grade that has 2-4 μm grain size band and 8.5 wt% cobalt content. It is used to manufacture cutting inserts where a degree of toughness is required. It contains mixed crystals (TiC, TaC and NbC). Pressing test sheets as well as other test results and reports can be found in Appendix 1-A21

Table 4.62 - Grade ‘PI40’ summary

SD (Grade batch)	13.80 g/cm ³
SD (Test sample)	13.81 g/cm ³
Apparent density (Grade batch)	3.30 g/cm ³
Apparent density (Test powder sample)	3.27 g/cm ³
Volatile content (Test sample)	2.29 %
Best average green density (Measured)	7.71 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	22.4 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.63 – Pressing test results for grade ‘PI40’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	22.0 %	22.6 %	3.85	7.70	27.4 / 110
4.00	22.0 %	22.2 %	7.73	7.73	25.7 / 103
8.00	22.0 %	22.5 %	15.40	7.70	24.2 / 98

Table 4.64 – Screen fraction report for grade ‘PI40’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
133.5 g	0.1 g	5.5 g	97.0 g	18.6 g	8.3 g	4.0 g
Percent	0.1 %	4.1 %	72.7 %	13.9 %	6.2 %	3.0 %

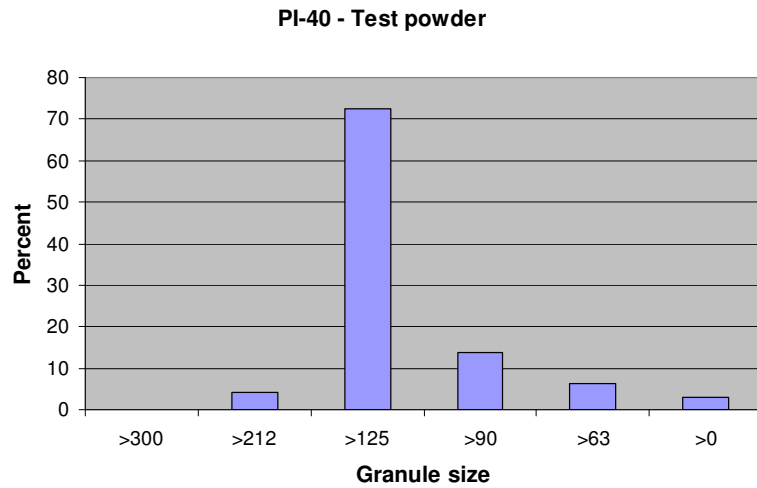


Figure 4.61 – Measured granule size distribution of test powder sample of grade PI40 (apparent density of 3.27 g/cm³)

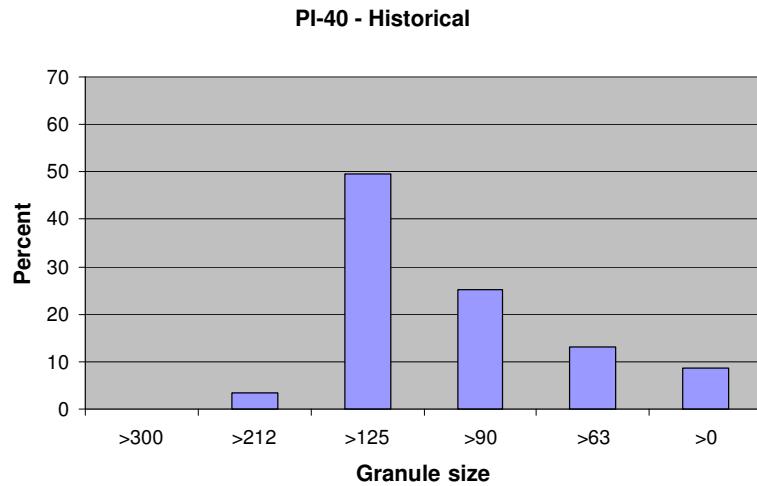


Figure 4.62 – Historical granule size distribution of grade PI40 (historical apparent density 3.40 g/cm³)

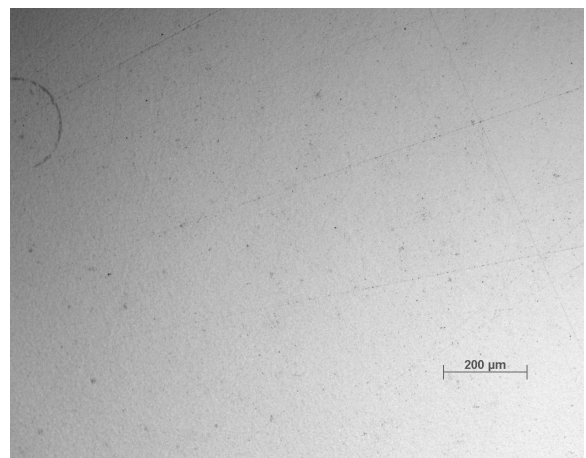
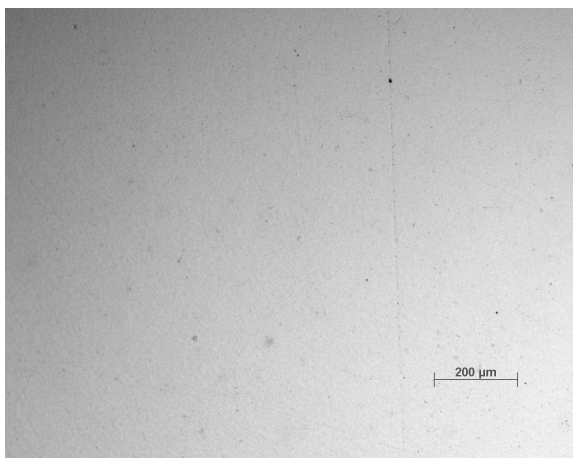


Figure 4.63 – 100x magnification image of polished section of the best shrinkage (least porosity) 22 % and the worst shrinkage (most porosity) 25 % for grade PI40

4.2.22 Grade ‘Q3’ experimental data

‘Q3’ grade powder is a mining grade that has 3-6 μm grain size and very high cobalt content (23.0 wt%). It is used where very high toughness is required. It contains no mixed crystals. Pressing test sheets, historical data as well as other test results and reports can be found in Appendix 1-A22.

Table 4.67 - Grade ‘Q3’ summary

SD (Grade batch)	13.18 g/cm ³
SD (Test sample)	13.26 g/cm ³
Apparent density (Grade batch)	3.04 g/cm ³
Apparent density (Test powder sample)	3.13 g/cm ³
Volatile content (Test sample)	2.20 %
Best average green density (Measured)	7.26 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	22.1 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.68 – Pressing test results for grade ‘Q3’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
4.00	22.5 %	23.1 %	7.26	7.26	23.4 / 94
8.00	22.5 %	23.1 %	14.52	7.26	23.0 / 92
-	-	-	-	-	-

Table 4.69 – Screen fraction report for grade ‘Q3’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
149.7 g	2.3 g	53.9 g	77.3 g	11.2 g	3.1 g	1.9 g
Percent	1.5 %	36.0 %	51.6 %	7.5 %	2.1 %	1.3 %

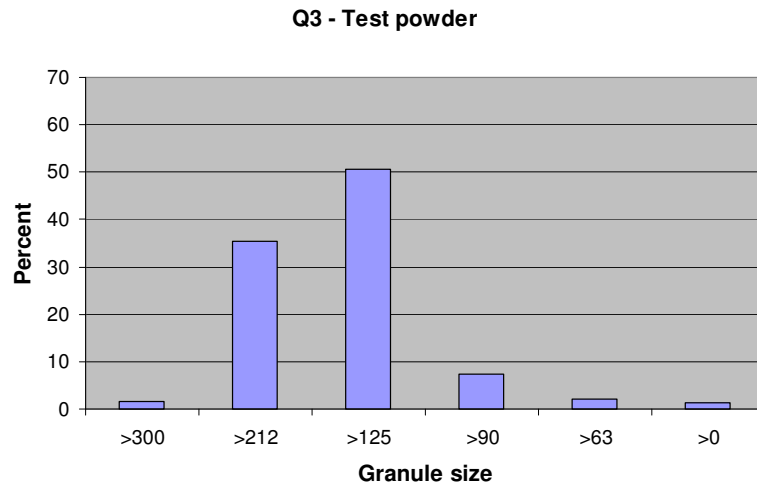


Figure 4.64 – Measured granule size distribution of test powder sample of grade Q3 (apparent density of 3.13 g/cm³)

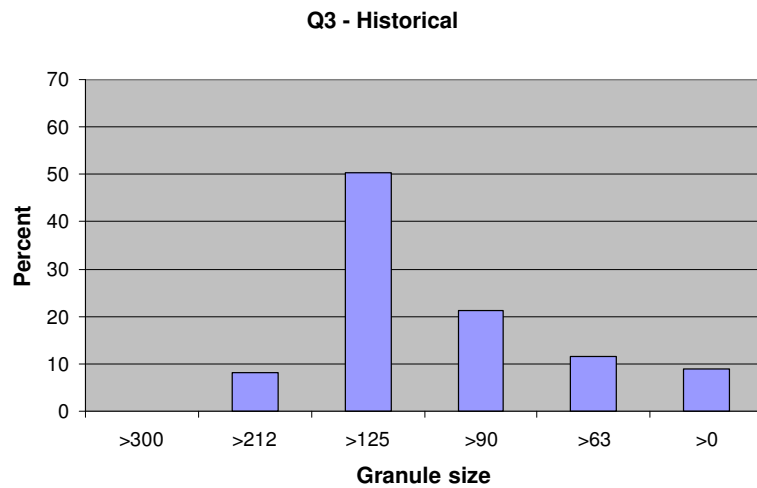


Figure 4.65 – Historical granule size distribution of grade Q3 (historical apparent density of g/cm³)

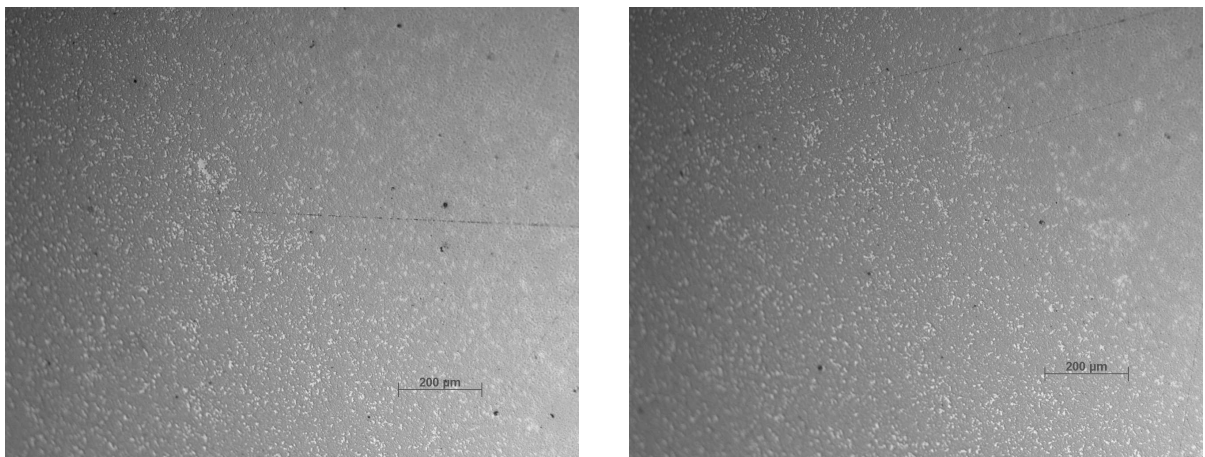


Figure 4.66 – 100x magnification image of polished section of the best shrinkage (least porosity) 22.5 % and the worst shrinkage (most porosity) 24 % for grade Q3

4.2.23 Grade ‘S9’ experimental data

‘S9’ grade powder is a tool grade that has 1-3 μm grain size and 10 wt% cobalt content. It is a very mixed grade as it is made up of mainly left-over powders from various other tool grades and can be call a general tool grade. It contains mixed crystals (TiC, TaC and NbC). Pressing test sheets, historical data as well as other test results and reports can be found in Appendix 1-A23.

Table 4.70 - Grade ‘S9’ summary

SD (Grade batch)	12.38 g/cm ³
SD (Test samples)	12.43 g/cm ³
Apparent density (Grade batch)	3.34 g/cm ³
Apparent density (Test powder sample)	3.20 g/cm ³
Volatile content (Test samples)	2.33 %
Best average green density	7.27 g/cm ³
Average best shrinkage for grade (Evaluated test pieces)	20.4 %
Porosity result for ‘best shrinkage’ sintered components	A00 B00

Table 4.71 – Pressing test results for grade ‘S9’

Green thickness (mm)	Best compact	Actual shrinkage (overall)	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
2.00	19.0 %	19.9 %	3.66	7.32	35.8 / 143
4.01	19.0 %	20.3 %	7.33	7.31	31.8 / 127
8.01	20% (S)	21.0 %	14.41	7.19	25.7 / 103

Table 4.72 – Screen fraction report for grade ‘S9’ test powder (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
167.9 g	1.6 g	20.5 g	64.2 g	40.3 g	25.1 g	16.2 g
Percent	0.1 %	12.2 %	38.2 %	24.0 %	15.0 %	9.7 %

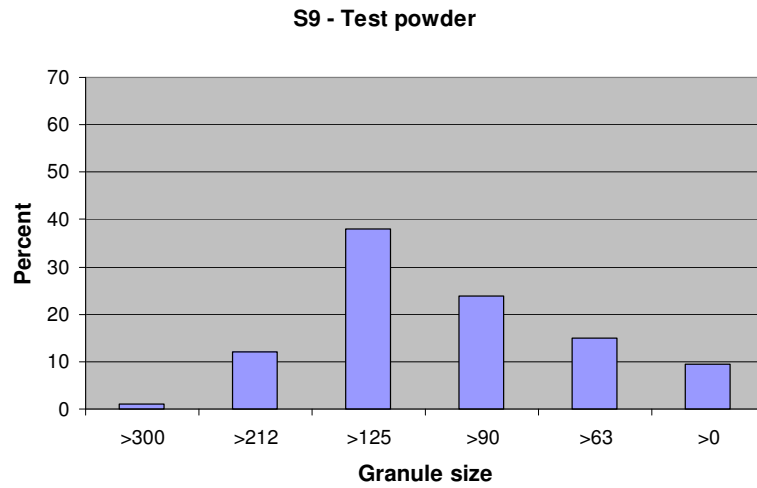


Figure 4.67 – Measured granule size distribution of test powder sample of grade S9 (apparent density of 3.20 g/cm³)

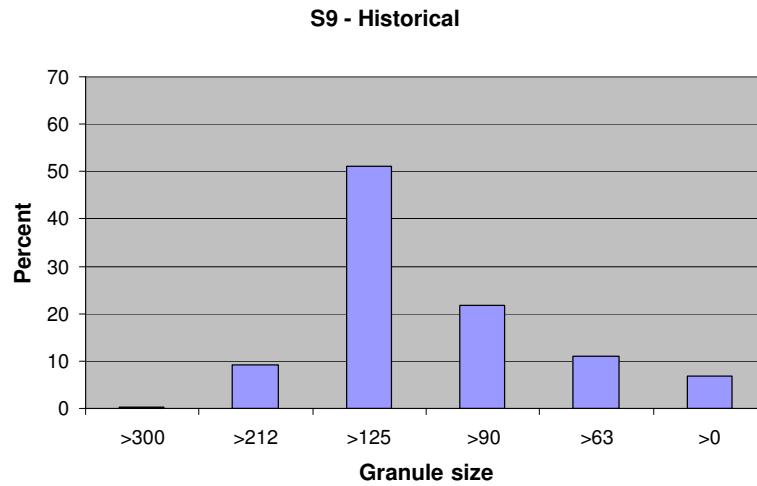


Figure 4.68 – Historical granule size distribution of grade S9 (historical apparent density of 3.25 g/cm³)

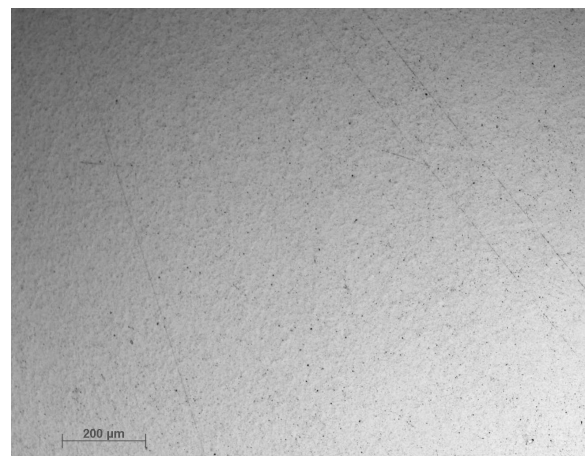
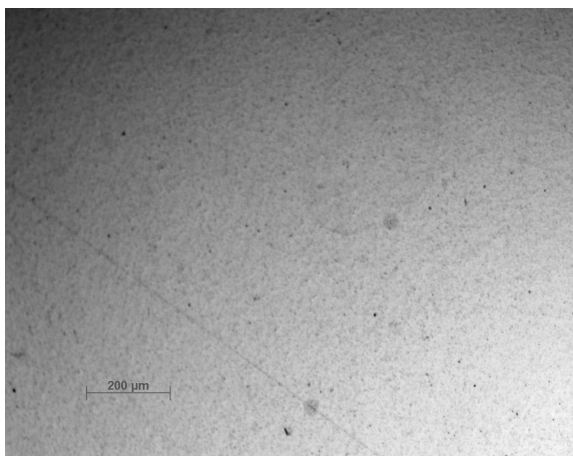


Figure 4.69 – 100x magnification image of polished section of best shrinkage (least porosity) 19 % and the worst shrinkage (most porosity) 23 % for grade S9

4.3 Positive insert test data

Four tool grades were selected to press positive inserts with an angle of 20 degrees (see section 2.2.3 & 3.1.4). The grades used in the tests were : CR10, PI-25, P40M and K15M. The pressing tests were then repeated with the same powders with a negative insert pressing. The purpose here was to assess if the geometry of the inserts has any effect on the ideal green density and thus the ideal shrinkage.

Below are tables showing the best pressed compacts for each of the four grades of tool grade powder. The following must be noted:

- The ‘best target’ shrinkage is the theoretical target shrinkage as calculated in section 3.1.5 (powder mass calculation). The actual shrinkage was not measured as the tests were purely comparative (i.e. ‘positive insert’ shrinkage vs. ‘negative insert’ shrinkage).
- The toolset used was designed with a 25.5% shrinkage. The target shrinkages in this set of experiments were calculated as shown in section 3.1.5 but using 26% as the reference.
- In the instance of the ‘CR10’ test, the best results for the two types of inserts were shrinkages of 26% (see table 4.73 & table 4.74). In the negative insert testing done in earlier experiments, the best result was 24%. In the tests done here, a new sample of CR10 powder was obtained and after physical property analysis, it was found that the apparent density was very different to the test powder used in the earlier experiments. (earlier apparent density was 3.26 g/cm³, the apparent density of the new CR10 sample used here was 3.12 g/cm³).
- The apparent density has an effect on the ideal shrinkage and this is discussed in section 4.4.4. The result of 26% shrinkage for the CR10 grade obtained here was checked with the conclusions reached in Chapter 5 and it was found to be correct.
- The ‘best target shrinkages’ were used as used as a guide for calculating a target pressing weight. For an accurate comparison of negative and positive inserts, the obtained green densities were compared. These densities were calculated from known powder weight and known green volume of the die cavity.
- The original pressing test sheets can be found in Appendix 2.

Table 4.73 – Best pressing results for negative inserts

Powder grade	Green thickness (mm)	Best target shrinkage	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
CR10	4.00	26.0 %	7.43	7.43	28.4 / 114
PI-25	4.00	21.5 %	8.05	8.05	22.7 / 90
P40M	4.01	22.5 %	7.35	7.33	27.5 / 110
K15M	3.99	24.0 %	6.93	6.94	30.6 / 122

Table 4.74 – Best pressing results for positive inserts

Powder grade	Green thickness (mm)	Best target shrinkage	Green weight (g)	Green density (g/cm ³)	Recorded force & pressure (kN / MPa)
CR10	6.14	26.0 %	10.15	7.44	29.8 / 119
PI-25	6.28	21.0 %	6.57	7.93	-
P40M	6.28	22.5 %	6.08	7.34	-
K15M	6.28	24.0 %	5.77	6.97	-

4.4 Summary and discussion of results

4.4.1 Pressing and inspection of green

As explained in the previous chapters, of the grades of powder currently being used at Powder Industries were tested in a series of pressing experiments – in total 23 different powders. The main difference in characterising each grade is cobalt content, grain size and mixed crystal content. The pressing experiments were carried out using the hydraulic CNC press in conjunction with the SNUN tooling – resulting in a square shaped component (as described in section 3). For each grade, three sets of compacts were pressed with shrinkages varying from 20% to 25% and where a grade displayed hard pressing; 21% to 26%. The differentiation between the sets of compacts is the compact thicknesses (2mm, 4mm and 8mm) with all of the compacts having the same cross-sectional area. This was done to check how compact size changes affect the ideal green density.

It was found that for a particular grade, the ideal shrinkage was the same for the three different compact thicknesses. The fact that the thickness of the compacts did not affect the ideal shrinkage indicates that all the selected “height to width” ratios were within the ratios which lead to almost uniform density distribution [15]. The average of the shrinkages of the three different compact sizes for a grade was then used to determine an average ideal shrinkage and ideal green density for a grade.

After all of the grades had been tested, a further set of pressing experiments were conducted to investigate if a positive angle on an insert changes the shrinkage or ideal density of a compact. Four tool grade powders were selected as positive inserts are generally cutting inserts. With each selected grade of powder, a set of negative inserts were pressed as well as a set of positive inserts. These compacts were inspected as per the usual procedure and their green densities were calculated. It was found that the ideal green densities were the same for the negative and positive insert of a grade. The fact that the design of the compact did not affect the ideal shrinkage indicates that compaction was successful even when tooling included acute angles.

Therefore, it was found that each grade of powder had a certain ideal shrinkage regardless of compact size and thickness (to within the limits of cutting insert size and shape produced at Powder Industries), while in the past, the company assumed that the same shrinkage (e.g. 20%) could be used across all grades. Each grade had a different ideal shrinkage because the green compacts of each grade had a different amount of porosity, due to different ratios of hard (carbides) to soft powders (cobalt), which affected the granulates' compressibility. Thus, the biggest factor influencing the shrinkage allowance during tool design is the choice of grade that the component is to be manufactured from.

4.4.2 Test powder physical properties

Every batch of granulated powder produced in the factory has its relevant physical properties evaluated and recorded. The test powders drawn from the stores for this investigation originated from these batches and the properties such as apparent density, screen fractions and powder flow rates could be checked. However, since the test samples used were representative of entire grades and the physical properties needed to be very accurate, each of the powder samples was re-evaluated – this would be a useful check of the accuracy of the powder production and analysis process as well as to have the actual properties of the test sample available (powder properties may vary within a batch). It must be noted that all of the test powders had been granulated.

The re-evaluation of the powders included measuring the apparent density and then screen fractioning the powder into its constituent granulate sizes (according to the company standard of granules, that are $>300\mu\text{m}$, $>212\mu\text{m}$, $>125\mu\text{m}$, $>90\mu\text{m}$, $>63\mu\text{m}$, $>0\mu\text{m}$)

All the test powder apparent densities from each of the 23 grades were measured as per company standard and recorded (see section 3.1.7). The test powder apparent density that was drawn from a batch of a particular grade was then compared to the historical apparent densities of the grade. In most cases, the apparent density of a test powder was very close to the historical apparent density of a grade. In a few cases, the values were not the same, but small variations from batch to batch ($\pm 0.1 \text{ g/cm}^3$) were considered acceptable. If an apparent density varied from a historical value, this would affect

compaction therefore, the aim was to relate the apparent density to compaction and thus to a green density.

It was found that in the majority of cases, test powder properties (apparent densities and granulate sizes and size distribution) were close to historical values and this indicated that intra batch variations are not very great and that the operators evaluating the powders were capturing powder data accurately.

However, there were exceptions where powder granule size distributions and apparent densities of the test sample did not match the historical. The exceptions were in the minority and the results obtained from the pressing tests using these powder samples are discussed in the following sections. As shown below in section 4.4.4, in the formulation of a method / equation to predict ideal shrinkage, the results from the exceptions had to be discarded.

4.4.3 Porosity of sintered components

The best and worst sintered test pieces from each grade were selected (best shrinkage and worst shrinkage), mounted, polished and inspected in the laboratory for porosity. The main reason for this was to confirm the selection of the compacts that were considered to have been pressed to the best green density. If a compact had been under-pressed, this would be apparent in the same sintered component as some kind of porosity would be seen.

The two pieces from each grade were observed and in most cases there was very little porosity even in the worst pressed components. It was observed that the sintered components (best and worst case pressing) were all of a high quality – this is mainly due to the HIP cycle during sintering. During the HIP cycle, a very high pressure is exerted on the components and a secondary compaction process occurs. Any residual porosity is removed and the result is a high quality component, even if the green compact was under-pressed. Thus, the porosity comparison of the test pieces was not a practical way to double-check the observations made when the green components were initially inspected.

4.4.4 Apparent density and ideal green density

After testing each grade of powder, an ideal green density for each powder was obtained, which is required to achieve good compaction. The green density was obtained by weighing the green test pieces while the accurate volume for the pieces was known (all pressing was done to a specific fixed green volume).

As expected, the apparent density of the powders used during pressing generally increased as the green density required for good compaction increased, as shown in Fig.

4.70

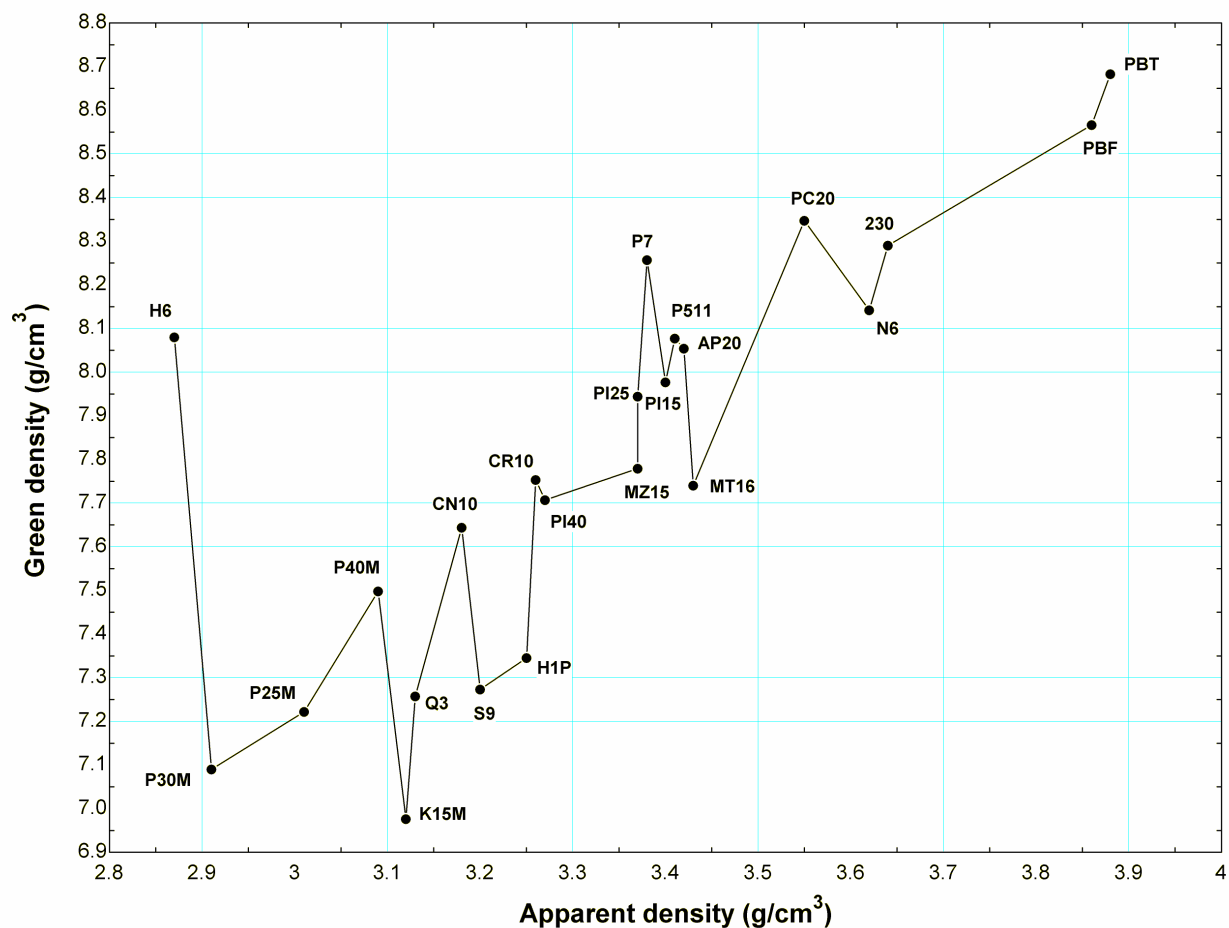


Figure 4.70 – General trend of ideal green density vs. apparent density for all powders tested (data from the tables in section 4.2)

However, the increase was not monotonic and the reason was investigated as follows:

1. Was the deviation from a monotonic increase due to different powder compositions (since composition affects density)?

2. Did the granulate size and/or size distribution vary from powder to powder (since granulate size and size distribution affect the apparent density)?
3. Other factors?

The results of the analysis were the following:

1. After analysing the powder composition (i.e. Co content, mixed crystal content and grain size) of each grade, it was found that composition had no effect on the relationship between ideal green density and apparent density.
2. It was found that variance in granulate size and size distribution was the main problem in causing the non-monotonic relationship in Fig. 4.70.
3. After further analysis, it was found that some of the powders tested could not be included in the graph and needed to be discarded from the results for the following reasons:

- Granule size distribution did not match the historical size distribution and thus the powder could not be used for comparison with other powders. If the powders are not physically similar, it is not possible to compare properties such as apparent density. Apparent density is affected by the granule size distribution. Therefore, the grades that did not match the historical values (e.g. grade P7) were omitted from the graph in Fig.4.70 The historical grain size distribution was taken as the standard and it consisted of the following:

1. Granules $> 300\mu\text{m}$ – approximately 0.3%
2. Granules $> 212\mu\text{m}$ and $< 300\mu\text{m}$ – approximately 9%
3. Granules $> 125\mu\text{m}$ and $< 212\mu\text{m}$ – approximately 46%
4. Granules $> 90\mu\text{m}$ and $< 125\mu\text{m}$ – approximately 21%
5. Granules $> 63\mu\text{m}$ and $< 90\mu\text{m}$ – approximately 13%
6. Granules $< 63\mu\text{m}$ or 'fines' approximately 9%

Figure 4.71 on the following page shows a histogram of the data given above:

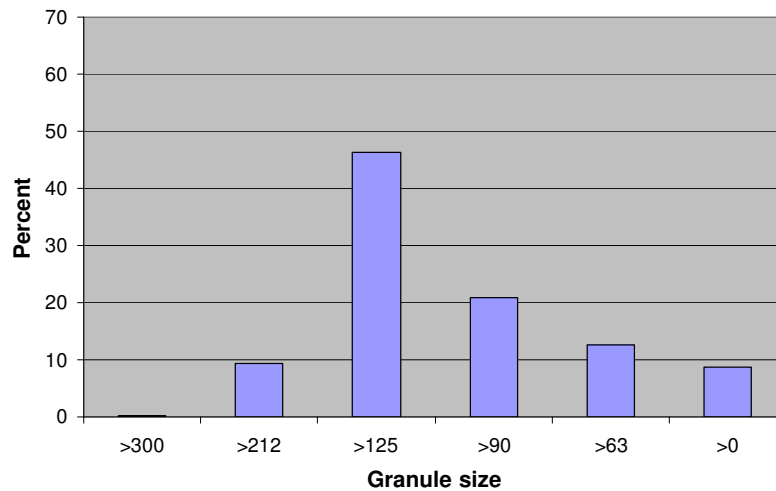


Figure 4.71 – Historical granule size distribution (standard) for all grades.

- Apparent density of the test powder from previous experiments did not match historical apparent density of that grade by a large margin. Even if the granule size distribution of the test sample matches historical values, there are factors which might have contributed to a false apparent density measurement such as the existence of hollow granules (e.g. of grade H6).
- Certain grades of powder are compactable over a large range of shrinkage and it was difficult to estimate a specific value for an ideal green density. Thus, even though the actual press tests were successful, some results had to be omitted from the graph as the ideal density range was too large. An example of this would be the grade MT16: when inspected, ‘good’ shrinkage values ranged from 20.5% to 23.5%. Grade Q3 is also another example.
- Some grades of powder are manufactured very seldom and there were not enough data available for historical evaluation. An example would be a grade in which only three batches had been manufactured (K15M) and the values for apparent density and granule size distribution were not consistent. Other batches that are very seldom manufactured are S9 and H1P.

In total, the results from 8 powders were omitted (H6, K15M, Q3, S9, H1P, MT16, N6, P7) from the “ideal green density vs. apparent density graph” (see Fig. 4.72). The results that were not omitted showed that a linear relationship can be assumed to exist between a powder’s apparent density and the green density that needs to be achieved for good compaction, namely:

$$\text{Required green density} = 2.51 + 1.6 \times \text{apparent density} \quad [4.1]$$

(with regression coefficient of $R^2 = 0.965$)

Note that the present and following equations are given using names rather than symbols to allow the practitioners to follow the arguments and apply the results, as was the purpose of this investigation.

Additional tests showed that when the correct shrinkage is used in the tooling, the shrinkage will give correctly pressed components even if it is one percentage point higher or lower than the ideal value. This means that the ideal green density can vary by $\pm 0.1 \text{ g/cm}^3$.

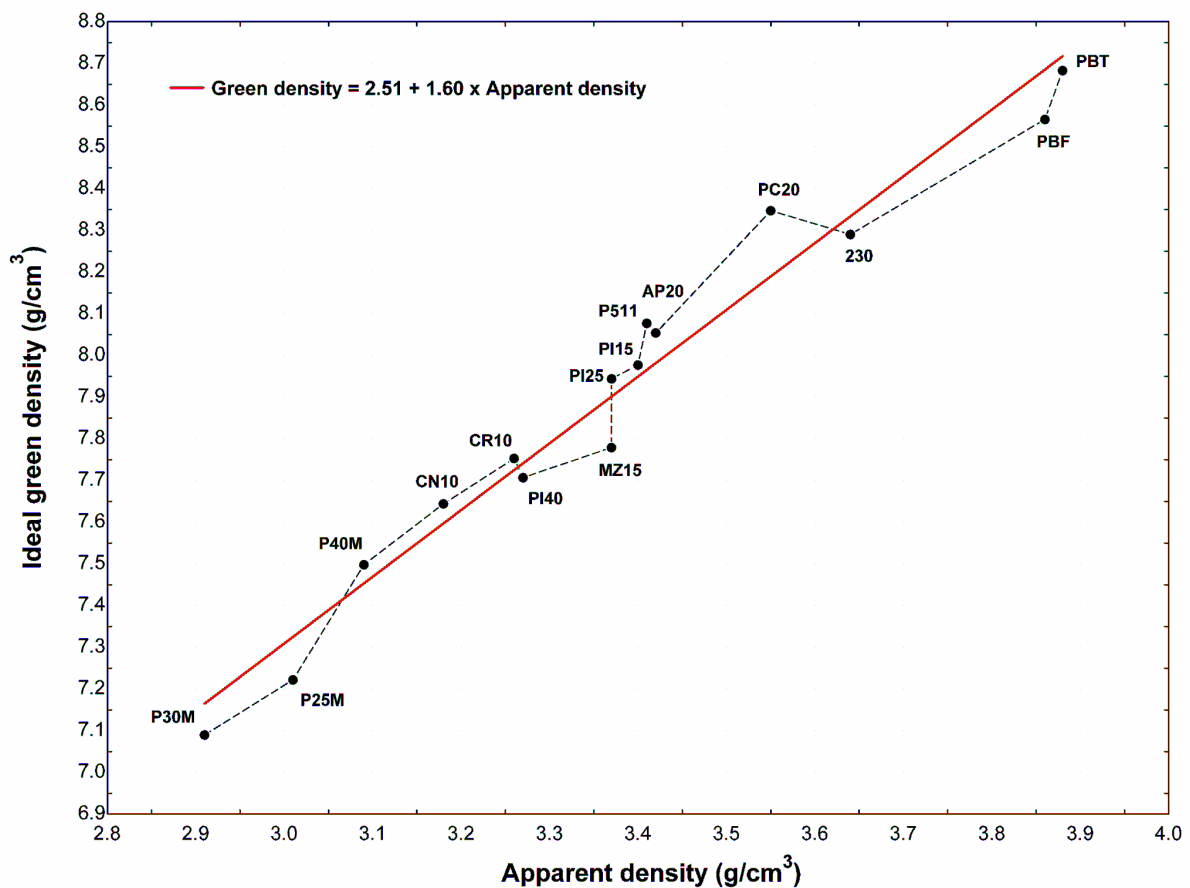


Figure 4.72 – Ideal green density vs. apparent density

The importance of equation [4.1] is that it allows to calculate the desired green density from the apparent density. If the desired green density and the mass of the powder are known, the ideal green volume can be calculated. The designer knows what the sintered volume should be. The designer also knows the sintered density and volatile content

(from powder lab reports). The ideal shrinkage can now be calculated and used when designing a new set of compaction tooling. The calculation of the ideal shrinkage is given in the following section.

4.4.5 Derivation of shrinkage equation

Now that we know the required green density (from equation [4.1]) and the required sintered density, it is possible to derive an equation to calculate the ideal shrinkage. The designer needs as inputs only the sintered density, the apparent density and the powder volatile content. The derivation for the equation is as follows:

Shrinkage can be defined as the percentage difference between a green length and sintered length:

$$\text{Linear shrinkage (\%)} = \frac{L_{\text{green}} - L_{\text{sintered}}}{L_{\text{sintered}}} \times 100$$

Simplifying:

$$\text{Linear shrinkage (\%)} = \left(\frac{L_{\text{Green}}}{L_{\text{Sintered}}} - 1 \right) \times 100 \quad [4.2]$$

Similarly, volumetric shrinkage can be defined as the percentage difference between a green volume and sintered volume :

$$\text{Volumetric shrinkage (\%)} = \left(\frac{V_{\text{green}}}{V_{\text{sintered}}} - 1 \right) \times 100 \quad [4.3]$$

As volumetric shrinkage is a cube of the linear shrinkage, we can write:

$$\left(\frac{V_{\text{green}}}{V_{\text{sintered}}} \right) = \left(\frac{L_{\text{green}}}{L_{\text{sintered}}} \right)^3 \quad \text{so} \quad \left(\frac{L_{\text{green}}}{L_{\text{sintered}}} \right) = \left(\frac{V_{\text{green}}}{V_{\text{sintered}}} \right)^{1/3} \quad \text{and substituting into [4.2] we}$$

get :

$$\text{Linear shrinkage (\%)} = \left(\left(\frac{V_{\text{green}}}{V_{\text{sintered}}} \right)^{1/3} - 1 \right) \times 100 \quad [4.4]$$

Green volume and sintered volume can be expressed as :

$$V_{green} = \frac{Mass_{green}}{Density_{green}} \quad \text{and} \quad V_{sintered} = \frac{Mass_{sintered}}{Density_{sintered}}$$

Substituting into [4.3] and remembering that $Density_{sintered} = SD$:

$$Linear\ shrinkage\ (\%) = \left(\left(\frac{Mass_{green} \times SD}{Density_{green} \times Mass_{sintered}} \right)^{1/3} - 1 \right) \times 100 \quad [4.5]$$

The mass of a green compact is equal to the sintered mass multiplied by the volatile content (volatile content being always given in industry as percentage of the mass of the powder):

$$Mass_{green} = Mass_{sintered} \times (1 + volatile(wt\%)/100)$$

So, substituting this into [4.4], we get:

$$Linear\ shrinkage\ (\%) = \left(\left(\frac{SD \times \left(1 + \frac{volatile(wt\%)}{100} \right)}{Density_{green}} \right)^{1/3} - 1 \right) \times 100 \quad [4.6]$$

From section 4.4.4 (equation [4.1]):

$$Density_{green} = 2.51 + 1.6 \times Density_{apparent}$$

Substituting this into [4.6]:

$$Linear\ shrinkage\ (\%) = \left(\left(\frac{SD \times \left(1 + \frac{volatile(wt\%)}{100} \right)}{2.51 + 1.6 \times Density_{apparent}} \right)^{1/3} - 1 \right) \times 100 \quad [4.7]$$

Since the volatile content is approximately 2.4% (historically):

$$\text{Linear shrinkage (\%)} = \left(\left(\frac{SD \times 1.024}{2.51 + 1.6 \times \text{Density}_{\text{apparent}}} \right)^{1/3} - 1 \right) \times 100 \quad [4.8]$$

4.4.6 Positive insert considerations

The base experiments of this project were performed using a ‘negative’ insert toolset. Component thickness was varied and various sizes for every grade of powder could be pressed. It was found after pressing with various tool grade powders and using a ‘positive’ insert toolset that the ideal green density and hence the shrinkage for a grade of powder is independent of insert shape. This is to within the confines of standard ISO shapes that have been tested and to the cutting inserts manufactured at Powder Industries. However, this result may not be extrapolated to components of very different shapes without previous experimentation.

4.4.7 Limitations of the ideal density vs. green density model

Different sizes of components were tested with different grades of powder. Although the sizes differed by varying a compact thickness and keeping the base area constant, this was not done for a large thickness vs. diameter ratio (known as the L/D ratio) as was stated in section 3.1.3 & 4.4.1. There is a limitation to the thickness that can be pressed if the cross-sectional area is small compared to the thickness [15], since the density distribution may no longer be uniform at large thicknesses.

4.4.8 Validating the equation – cutting insert and shim tool production

Equation [4.8] shows that linear shrinkage can be calculated from the specific gravity (SG) of the sintered component, the apparent density of the powder and the volatile content. These data are all available to the tool designer. However, it is important to note that this equation is only valid for granulated powders that have granulate size distribution approximately equal to the standard at Powder Industries (see defined standard in section 4.4.4).

The equation was tested in the manufacture of new tooling. Test pieces were pressed successfully, i.e. to the correct green density and were then sintered correctly.

The test pieces included cutting inserts that were pressed successfully for the first time. Some tooling did work before but components were pressed by applying equation [4.8] – e.g. shim components.

The equation is now in general use at Powder Industries when new tooling is being designed. However, if powder properties change in the future, the linear relationship between apparent density and green density would need to be re-evaluated.

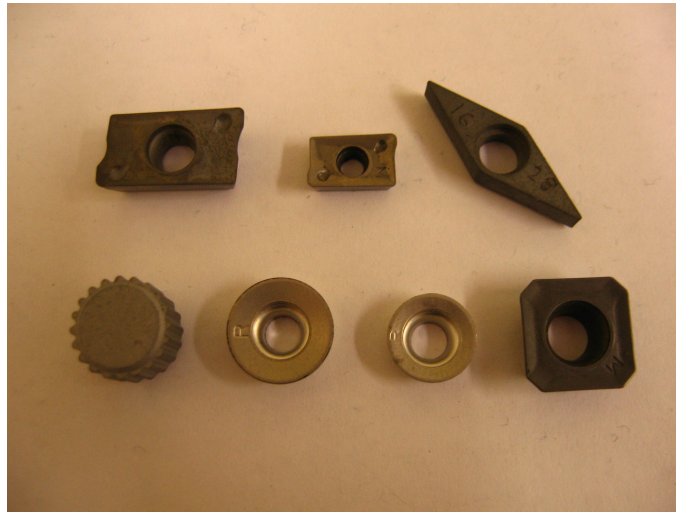


Figure 4.73 – Examples of successful new components made due to the research project. The components with central holes are examples of cutting tool inserts, the remaining component is a mining insert.

5. Conclusions

The conclusions that can be drawn from this investigation can be summarized as follows:

1. The ideal green density of a powder compact can be found experimentally by keeping the volume of the compact constant and changing the mass, while in most investigation, the opposite has been done, requiring different tooling for different green density. The method applied in this work is obviously less costly (section 3.1.3 & 3.1.5).
2. The relationship between the ideal green density of powder compacts and the apparent densities of the corresponding granulated powders is linear as long as the powders granulate mean size and size distribution is the same the standard at Powder Industries (Fig. 4.71).
3. An equation has been derived for the shrinkage of all the grades at Powder Industries. This equation can be applied to a new grade that is introduced as long as the granulate size distribution conforms to the company standard. The equation is in terms of sintered density, apparent density and volatile content. This equation has been validated by pressing (and subsequently sintering) successfully difficult to press grades, and new grades and different shapes.

$$Shrinkage(\%) = \left(\left(\frac{SD \times \left(1 + \frac{volatile(\%)}{100} \right)}{2.51 + 1.6 \times Density_{apparent}} \right)^{1/3} - 1 \right) \times 100 \quad [5.1]$$

Using volatile content as a constant (2.4%), the equation can be simplified to:

$$Shrinkage(\%) = \left(\left(\frac{SD \times 1.024}{2.51 + 1.6 \times Density_{apparent}} \right)^{1/3} - 1 \right) \times 100 \quad [5.2]$$

5.1 Recommendations for further work

The logical continuation of the present investigation would be the following:

1. The focus of this project was to improve the shrinkage consideration when designing new tooling for the powders manufactured at Powder Industries. Now that this problem has been solved, shape distortion during sintering should be investigated. After compaction, there is an inhomogeneous distribution of green density due to shape complexity, filling variations etc. and this causes the component to shrink during sintering by varying amounts in different. FEM software exists [21] that can help simulate how a compact shrinks and this allows the tool designer to modify the punch profiles in order to compensate for the variable shrinkage in specific areas of the component, however, experimental work should be carried out to validate the simulations.
2. In future, it may be possible that an important constituent of the powder will be changed (as in the case of PEG replacing paraffin wax). In order to know in advance how this may affect the shrinkage equation [5.2], test powders should be made up changing some constituents and the tests described in this report should be repeated in order to anticipate possible change in the ideal density vs. apparent density relationship.

6. References

- 1) R. Stockhill, Powder Industries, private communication
- 2) C.L. Martin, D. Bouvard, *Study of the cold compaction of composite powders by the discrete element method*, Acta Materialia, 2003, Vol. 51, pg 373-386
- 3) R. Panelli, F. Ambrozio Filho, *Compaction equation and it's use to describe powder consolidation behaviour*, Powder Metallurgy, 1998, Vol. 41 No.2, pg 131-133
- 4) T. Kraft, H. Riedel, P. Gumbsch, *Computer simulation as a tool in PM material science and technology*, 16th International Plansee Seminar 2005, Vol. 2, pg 9-23
- 5) A. Upadhyaya, R.M. German, *Control of distortion during liquid phase sintering*, 14th International Plansee seminar 1997, Vol, pg 68-65
- 6) C.L. Martin, *Elasticity, fracture and yielding of cold compacted metal powders*, Journal of the mechanics and physics of solids, 2004, Vol. 52, pg 1691-1717
- 7) T. Kraft, *Optimizing press tool shapes by numerical simulation of compaction and sintering – application to a hard metal cutting insert*, Modelling and Simulation in Materials Science and Engineering, 2003, Vol. 11, pg 381-400
- 8) T.Kraft, B. Henrich, C. Bierwisch, M. Moseler, H. Riedel, *Modelling and simulation of processing steps in powder technology using particle dynamics*, 16th International Plansee Seminar 2005, Vol. 2, Plenary lectures and PM hard materials, pg 69-81
- 9) O. Alvain, P. Doremus, D. Bouvard, *Modeling the compaction of a WC-Co powder grade : characterization and simulation*, EuroPM seminar 1999, Pressing technology presentation, pg 111-118
- 10) Dr K. Rodiger, Dr H. van den Berg, Dr K Dreyer, D. Kassel, S. Orths, *Near-net-shaping in the hardmetal industry*, EuroPM seminar 1999, Pressing technology presentation, pg 99-102
- 11) T. Kraft, O. Coube, H. Riedel, *Numerical simulation of pressing and sintering in the ceramic and hardmetal industry*, Recent developments in Computer Modelling of Powder Metallurgy Processes, NATO Advanced Research Workshop, Series III: Computer and Systems Science, 2001, Vol. 176, A. Zavaliangos, A. Laptev (eds.), IOS Press, Amsterdam, Netherlands, pg 181-190

- 12) O. Coube, R. Huber, R. Kusters, A. Plankensteiner, M. Magin, *Numerical simulation of die compaction and sintering of hardmetal drill tips*, 16th International Plansee Seminar 2005, Vol. 2, pg 243-255
- 13) H. Riedel, D. Meyer, J. Svoboda, H. Zipse, *Numerical simulation of die pressing and sintering – development of constitutive equations*, 13th International Plansee Seminar 1993, Vol. 2, pg 110-120
- 14) A. Petersson, J. Agren, *Sintering shrinkage of WC-Co materials with different compositions*, (Unknown journal source), Department of materials science and engineering, Royal institute of technology, Stockholm, Sweden.
- 15) F. Thummler, R. Oberacker, *Introduction to Powder Metallurgy*, The Institute of Materials, 1993
- 16) K. Dreyer, K. Rodinger, H. van den Berg, D. Kassel, S. Orths, *Powder Metallurgy and Applications of hardmetals*, DGM-Seminar “Powder Metallurgy”, 2000
- 17) H. Kolaska, P. Schulz, *Production of hardmetals – mixing, granulation and direct shaping*, No.4 of a series of lecture notes (Powder Industries library).
- 18) K.J.A. Brookes, *Hardmetals and other hard materials*, 3rd Edition, International Carbide Data, 1998
- 19) ASTM B212-83 and Metal Powder Industries Federation standard, MPIF 04-85, Standard test method for the determination of apparent density of metal powders
- 20) ISO 3369 and ASTM B311-93.
- 21) Deform-3D, ABAQUS

Appendices

- **Appendix 1** - This appendix includes the experimental data sheets of all grades tested and the powder properties that have been measured.
- **Appendix 2** – This appendix includes the pressing test sheets of the experiments done to compare ‘positive’ and ‘negative’ inserts.
- **Appendix 3** - This appendix includes the original porosity reports for all sintered test pieces from each grade that were evaluated for best and worst shrinkage. Included, are also the standard company micrographs used to evaluate the porosity. The company procedure for evaluating porosity is based on : Metallographic determination of porosity and uncombined carbon ISO 4505
Apparent porosity in cemented carbides ASTM B276

Appendix 1

A1.1 Experimental data sheets for grade '230'

A1.1.1 '230' Grade pressing test sheets – negative inserts

Powder details		Links
Date :	Item no : 4001002	Powder sheet
	Batch no : 92869	RTP
	Co % : 9.7	Spray drying
	Grain size : 2 - 6	
	PRZ reclaim? :	
	PEG content :	

Powder	230
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	14.52
App. Density	Volume 0.2547 cm ³
Press height	2.00
Weight	3.793
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.5	230 - 195	4.27	1.99	15.92	40.1	4.27	4.526	-0.186	G	1.67	13.33	4.18	
20.0	230 - 20	4.23	1.99	15.92	35.2	4.21	4.462	-0.176	G	1.65	13.29	4.12	
20.5	230 - 205	4.19	2.00	15.92	32.7	4.20	4.431	-0.160	G	1.64	13.26	4.10	
21.0	230 - 21	4.15	2.00	15.92	29.1	4.15	4.389	-0.150	B	1.63	13.21	4.06	
21.5	230 - 215	4.10	2.00	15.92	24.9	4.10	4.336	-0.140	G	1.62	13.155	4.01	
22.0	230 - 22	4.06	2.00	15.92	21.8	4.05	4.283	-0.130	G	1.61	13.18	3.96	
22.5	230 - 225	4.02	2.00	15.92	18.8	4.02	4.241	-0.120	G	1.61	13.035	3.93	
23.0	230 - 23	3.97	2.00	15.91	15.7	3.97	4.188	-0.110	G	1.62	12.985	3.88	
23.5	230 - 235	3.93	2.00	15.92	13.6	3.94	4.156	-0.100	G	1.61	12.96	3.85	

Powder	230
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	14.52
App. Density	Volume 0.5093 cm ³
Press height	4.00
Weight	7.584
Tool	25 %
	Volatile 2.56%

Powder details	Links
Date :	<u>Powder sheet</u>
Item no : 4001002	<u>RTP</u>
Batch no : 92869	<u>Spray drying</u>
Co % : 9.7 %	
Grain size : 2 - 6 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.5	230 - 195	8.54	4.00	15.92	35.1	8.55	9.160	-0.240	OK	3.32	13.28	8.39	
20.0	230 - 20	8.46	4.00	15.92	31.5	8.47	9.053	-0.200	OK	3.31	13.23	8.31	
20.5	230 - 205	8.37	4.00	15.92	27.7	8.36	8.946	-0.190	OK	3.30	13.18	8.26	
21.0	230 - 21	8.29	4.00	15.92	25.0	8.29	8.861	-0.180	OK	3.28	13.14	8.12	
21.5	230 - 215	8.20	4.00	15.91	21.7	8.20	8.764	-0.170	OK	3.27	13.10	8.04	
22.0	230 - 22	8.12	4.00	15.91	19.5	8.12	8.679	-0.160	OK	3.26	13.05	7.97	
22.5	230 - 225	8.03	4.00	15.91	16.4	8.01	8.561	-0.150	OK	3.25	13.00	7.85	
23.0	230 - 23	7.94	4.00	15.91	14.1	7.93	8.455	-0.140	OK	3.24	12.88	7.67	
23.5	230 - 235	7.85	4.02	15.90	12.0	7.84	8.348	-0.120	SS	3.24	12.94	7.77	

Powder	230
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	14.52
App. Density	Volume 1.0187 cm³
Press height	8.00
Weight	15.170
Tool	25 %
Volatile	2.56%

Powder details		Links
Date :		Powder sheet
Item no : 4001002		RTP
Batch no : 92869		Spray drying
Co % : 9.7 %		
Grain size : 2 - 6 μ		
PRZ reclaim? :		
PEG content : %		

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.5	230 - 195	17.09	7.99	15.91	34.7	17.08	18.350	-0.310	G	6.65	13.25	16.74	
20.0	230 - 20	16.92	8.00	15.91	30.0	16.92	18.167	-0.290	G	6.62	13.20	16.59	
20.5	230 - 205	16.75	8.00	15.91	26.7	16.74	17.974	-0.270	G	6.60	13.15	16.42	
21.0	230 - 21	16.58	8.00	15.91	23.9	16.58	17.792	-0.250	B	6.59	13.12	16.25	
21.5	230 - 215	16.41	8.01	15.91	21.4	16.44	17.599	-0.230	G	6.57	13.07	16.11	
22.0	230 - 22	16.24	8.00	15.90	18.4	16.22	17.395	-0.220	G	6.54	13.02	15.89	
22.5	230 - 225	16.06	8.00	15.90	16.5	16.08	17.224	-0.210	G	6.51	12.96	15.75	
23.0	230 - 23	15.89	8.00	15.90	14.3	15.89	16.999	-0.200	G	6.48	12.92	15.57	
23.5	230 - 235	15.71	8.00	15.90	12.4	15.69	16.785	-0.190	G	6.46	12.87	15.38	

A1.1.2 Grade 230 – Powder physical properties, SG and porosity reports

Table A1.1 – Historical screen fraction report for grade 230 (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
196.9 g	0.2 g	11.7 g	102.9 g	43.6 g	23.8 g	14.8 g

Table A1.2 – Historical screen fraction report for 230 (mass percent of previous table)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	5.9 %	52.3 %	22.1 %	12.1 %	7.5 %

Table A1.3 – Apparent density report of grade 230 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
91.1 g	25 cc	3.64 g/cm ³	3.70 g/cm ³

Table A1.4 – Sintered density report for grade 230 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	16.2579	15.2015	0.0689	0.9971	20.5	14.41	14.46
2	8.2080	7.7122	0.0689	0.9971	20.5	14.49	
3	4.0682	6.857	0.0689	0.9971	20.5	14.48	

Table A1.5 – Porosity report for best and worst sintered test pieces for grade 230

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
230	21%	23%	A00 B00	A00 B00	Very good	Good

A1.2 Experimental data sheets for grade 'AP20'

A1.2.1 'AP20' Grade pressing test sheets – negative inserts

Powder details		Links
Date :		Powder sheet
Item no : 4001029		RTP
Batch no : 87703		Spray drying
Co % : 10.5	%	
Grain size : 2 - 3	μ	
PRZ reclaim? :	Y	
PEG content :	%	

Powder	AP20
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	14.35
App. Density	3.190
Press height	2.00
Weight	3.732
Tool	25
	%
Volume	0.2547 cm ³
Volatile	2.10%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	AP20 - 20	4.16	2.00	15.92	40.0	4.15	4.704	-0.170	OK	1.65	13.27	4.06	
20.5	AP20 - 205	4.12	2.00	15.91	36.7	4.12	4.659	-0.160	OK	1.64	13.22	4.03	
21.0	AP20 - 21	4.08	2.00	15.91	33.5	4.08	4.614	-0.150	OK	1.63	13.18	3.99	
21.5	AP20 - 215	4.04	1.99	15.91	30.9	4.04	4.569	-0.150	B	1.62	13.14	3.94	
22.0	AP20 - 22	3.99	1.99	15.91	28.5	4.01	4.535	-0.140	OK	1.62	13.11	3.92	
22.5	AP20 - 225	3.95	1.99	15.91	24.8	3.95	4.467	-0.130	OK	1.62	13.05	3.88	
23.0	AP20 - 23	3.91	2.00	15.91	21.0	3.90	4.410	-0.120	OK	1.60	12.98	3.81	
23.5	AP20 - 235	3.86	2.00	15.91	18.5	3.87	4.365	-0.110	OK	1.60	12.94	3.78	
24.0	AP20 - 24	3.82	2.00	15.91	15.4	3.81	4.308	-0.100	OK	1.59	12.88	3.72	

Powder	AP20
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	14.35
App. Density	3.190
Press height	4.00
Weight	7.462
Tool	25
	%
	Volume 0.5093 cm ³
	Volatile 2.10%

Powder details	Links
Date :	
Item no : 4001029	<u>Powder sheet</u>
Batch no : 87703	<u>RTP</u>
Co % : 10.5	%
Grain size : 2 - 3	μ
PRZ reclaim? :	
PEG content :	%

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
20.0		AP20 - 20	8.32	4.00	15.92	36.7	8.32	9.566	-0.210	OK	3.30	13.23	8.14
20.5		AP20 - 205	8.24	4.00	15.92	33.3	8.24	9.474	-0.200	OK	3.28	13.22	8.07
21.0		AP20 - 21	8.16	4.00	15.91	30.0	8.15	9.371	-0.190	OK	3.28	13.13	7.98
21.5		AP20 - 215	8.07	4.00	15.91	27.1	8.07	9.268	-0.180	OK	3.25	13.09	7.89
22.0		AP20 - 22	7.99	4.00	15.91	24.7	7.98	9.164	-0.170	OK	3.24	13.05	7.81
22.5		AP20 - 225	7.90	4.00	15.91	22.0	7.91	9.073	-0.160	OK	3.23	13.00	7.73
23.0		AP20 - 23	7.81	3.99	15.91	19.1	7.79	8.946	-0.150	OK	3.21	12.93	7.62
23.5		AP20 - 235	7.73	4.00	15.91	17.6	7.74	8.866	-0.140	OK	3.21	12.92	7.58
24.0		AP20 - 24	7.64	4.01	15.91	15.3	7.66	8.752	-0.130	S	3.20	12.84	7.49

Powder	AP20
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	14.35
App. Density	3.190
Press height	8.00
Weight	14.925
Tool	25
	%
	Volume 1.0187 cm ³
	Volatile 2.10%

Powder details	Links
Date :	
Item no : 4001029	Powder sheet
Batch no : 87703	RTP
Co % : 10.5	% Spray drying
Grain size : 2 - 3	μ
PRZ reclaim? :	
PEG content :	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	AP20 - 20	16.65	8.01	15.91	35.5	16.67	19.170	-0.280	ok	6.60	13.19	16.32	
20.5	AP20 - 205	16.48	8.01	15.91	31.4	16.49	18.486	-0.270	ok	6.58	13.14	16.12	
21.0	AP20 - 21	16.31	8.01	15.91	29.0	16.32	18.801	-0.260	ok	6.57	13.09	15.96	
21.5	AP20 - 215	16.14	8.00	15.91	25.8	16.15	18.628	-0.250	ok	6.53	12.05	15.80	
22.0	AP20 - 22	15.97	8.00	15.91	23.4	15.96	18.420	-0.240	ok	6.50	13.01	15.61	
22.5	AP20 - 225	15.80	8.00	15.91	21.4	15.81	18.270	-0.230	ok	6.48	12.97	15.46	
23.0	AP20 - 23	15.63	8.00	15.90	18.9	15.61	17.970	-0.220	ok	6.46	12.91	15.26	
23.5	AP20 - 235	15.46	8.00	15.90	17.0	15.46	17.775	-0.210	ok	6.43	12.87	15.12	
24.0	AP20 - 24	15.28	8.01	15.90	14.7	15.27	17.602	-0.190	ok	6.43	12.81	14.93	

A1.2.2 Grade AP20 – Powder physical properties, SG and porosity reports

Table A1.6 – Historical screen fraction report for grade AP20 (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
195.9 g	0.1 g	6.4 g	99.4 g	47.0 g	24.3 g	18.8 g

Table A1.7 – Historical screen fraction report for grade AP20 (in percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	3.3 %	50.7 %	24.0 %	12.4 %	9.6 %

Table A1.8 – Apparent density report of grade AP20 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
85.4 g	25 cc	3.42 g/cm ³	3.42 g/cm ³

Table A1.9 – Sintered density report for grade AP20 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.8176	14.7876	0.0681	0.9974	19.2	14.37	14.35
2	7.9832	4.7968	0.0681	0.9974	19.2	14.36	
3	3.9471	3.7405	0.0681	0.9974	19.2	14.33	

Table A1.10 – Porosity report for best and worst sintered test pieces (midsize) for AP20

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
AP20	21.5 %	24 %	A00 B00	A00 B00	Good, slight instances of A&B	Instances of A&B

A1.3 Experimental data sheets for grade 'CN10'

A1.3.1 'CN10' Grade pressing test sheets – negative inserts

Powder details		Links
Date :	16.02.2006	
Item no :	4001003	<u>Powder sheet</u>
Batch no :	63467	<u>RTP</u>
Co % :	6.7	% <u>Spray drying</u>
Grain size :	< 1	μ
PRZ reclaim? :		%
PEG content :		%

Powder	CN10
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	14.41
App. Density	3.32
Press height	2.00
Weight	3.768
Tool	25
	%
	Volume 0.2547 cm ³
	Volatile 2.66%

Equivalent Tool %	Pressing (Green)								Sintered			
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
22.0	CN10-21	4.03	2.00	15.93	75.4	4.01	4.843	-0.270	H	1.63	13.08	3.90
22.5	CN10-215	3.99	2.01	15.93	63.3	3.99	4.807	-0.230	H	1.63	13.005	3.88
23.0	CN10-22	3.95	2.00	15.92	54.3	3.94	4.747	-0.20	OK	1.62	12.95	3.83
23.5	CN10-225	3.90	2.01	15.92	47.0	3.90	4.687	-0.180	OK	1.61	12.90	3.79
24.0	CN10-23	3.86	2.00	15.93	41.3	3.85	4.638	-0.170	B	1.60	12.86	3.74
24.5	CN10-235	3.81	2.00	15.93	36.3	3.81	4.578	-0.160	OK	1.60	12.82	3.71
25.0	CN10-24	3.77	2.00	15.93	32.2	3.77	4.4530	-0.150	OK	1.60	12.77	3.66
25.5	CN10-245	3.72	2.00	15.92	27.4	3.72	4.470	-0.140	OK	1.59	12.71	3.61
26.0	CN10-25	3.68	2.01	15.92	24.1	3.70	4.446	-0.120	OK	1.59	12.68	3.58

Powder CN10	
Tool set	SNUN120412 - Phase 2 (Midsized)
Area - pressed	250.1
S Height	3.20
SG	14.41
App. Density	3.32
Press height	4.00
Weight	7.534
Tool	25
	%
	Volume 0.5093 cm ³
	Volatile 2.66%

Powder details	
Date :	Links
Item no : 4001003	<u>Powder sheet</u>
Batch no : 63467	<u>RTP</u>
Co % : 6.7	%
Grain size : < 1	μ
PRZ reclaim? :	
PEG content :	%

Equivalent Tool %	Pressing (Green)										Sintered			
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight		
22.0	CN10-20	8.06	4.01	15.92	68.8	8.07	9.801	-0.320	H	3.24	13.045	7.85		
22.5	CN10-205	7.98	4.00	15.92	61.0	7.98	9.704	-0.300	H	3.23	13.00	7.77		
23.0	CN10-21	7.89	4.00	15.92	52.3	7.88	9.582	-0.280	OK	3.21	12.97	7.68		
23.5	CN10-215	7.80	4.00	15.92	46.8	7.80	9.473	-0.260	OK	3.21	12.90	7.59		
24.0	CN10-22	7.71	4.00	15.92	40.2	7.72	9.376	-0.240	B	3.21	12.85	7.4950		
24.5	CN10-225	7.62	4.00	15.92	33.9	7.61	9.254	-0.220	OK	3.19	12.795	7.40		
25.0	CN10-23	7.53	4.00	15.91	29.6	7.53	9.145	-0.200	OK	3.18	12.74	7.33		
25.5	CN10-235	7.44	4.01	15.91	24.2	7.44	9.035	-0.170	OK	3.18	12.67	7.24		
26.0	CN10-24	7.35	4.02	15.91	19.4	7.34	8.914	-0.140	SS	3.17	12.59	7.13		

Powder	CN10
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	14.41
Volume	1.0187 cm ³
App. Density	3.32
Volatile	2.66%
Press height	8.00
Weight	15.070
Tool	25 %

Powder details	Links
Date :	
Item no : 4001003	Powder sheet
Batch no : 63467	RTP
Co % : 6.7 %	Spray drying
Grain size : < 1 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
21.0	CN10-21	16.47											
21.5	CN10-215	16.30	7.99	15.93	82.7	16.28	19.821	-0.530	H	6.51	13.08	15.84	
22.0	CN10-22	16.13	8.00	15.92	74.1	16.15	19.638	-0.496	H	6.49	13.04	15.73	
22.5	CN10-225	15.96	8.00	15.92	63.1	15.46	19.431	-0.450	H	6.48	12.98	15.53	
23.0	CN10-23	15.78	7.99	15.92	55.4	15.77	19.260	-0.430	H	6.44	12.94	15.35	
23.5	CN10-235	15.61	8.00	15.92	49.2	15.63	19.017	-0.400	H	6.42	12.91	15.21	
24.0	CN10-24	15.43	8.00	15.92	40.4	15.43	18.798	-0.370	SH	6.41	12.84	15.03	
24.5	CN10-245	15.25	8.00	15.92	34.2	15.25	18.579	-0.340	OK	6.39	12.79	14.86	
25.0	CN10-25	15.07	8.02	15.92	28.8	15.08	18.372	-0.290	B	6.38	12.715	14.69	
25.5		14.89	8.01	15.91	24.5	14.88	18.140	-0.270	OK	6.35	12.665	14.50	

A1.3.2 Grade CN10 – Powder physical properties, SG and porosity reports

Table A1.11 – Historical screen fraction report for grade CN10

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
195.1 g	0.0 g	0.1 g	67.6 g	75.4 g	36.1 g	15.9 g

Table A1.12 – Historical screen fraction report for grade CN10 (granule size percentage)

>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
0.0 %	0.1 %	34.7 %	38.7 %	18.5 %	8.2 %

Table A1.13 – Apparent density report of grade CN10 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
79.6 g	25 cc	3.18 g/cm ³	3.29 g/cm ³

Table A1.14 – Sintered density report for grade CN10 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.033	14.0602	0.0685	0.9968	21.7	14.39	14.36
2	7.5859	7.1277	0.0685	0.9968	21.7	14.36	
3	3.7454	3.5536	0.0685	0.9968	21.7	14.34	

Table A1.15 – Porosity report for best and worst sintered test pieces (midsize) for CN10

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
CN10	24 %	26 %	A00 B00	A02 B00	Good	OK

A1.4 Experimental data sheets for grade 'CR10'

A1.4.1 'CR10' Grade pressing test sheets – negative inserts

Powder details	
Date :	Links
Item no : 4001004	Powder sheet
Batch no : 74704	RTP
Co % : 9.5	%
Grain size : <1	μ
PRZ reclaim? :	%
PEG content :	%

Powder	CR10
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	14.45
App. Density	3.39
Press height	2.00
Weight	3.787
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
21.0	C10 - 21	4.14											
21.5	C10 - 215	4.10	2.00	15.93	71.1	4.11	4.872	-0.240	H	1.65	13.09	3.98	
22.0	C10 - 22	4.05	2.00	15.92	59.2	4.04	4.801	-0.220	H	1.64	13.04	3.91	
22.5	C10 - 25	4.01	2.00	15.92	53.8	4.01	4.753	-0.200	H	1.63	13.00	3.88	
23.0	C10 - 23	3.97	2.00	15.92	48.4	3.97	4.706	-0.180	H	1.62	12.95	3.85	
23.5	C10 - 235	3.92	2.00	15.92	41.1	3.92	4.647	-0.160	H	1.61	12.88	3.80	
24.0	C10 - 24	3.88	2.00	15.92	35.3	3.87	4.587	-0.140	S	1.60	12.82	3.74	
24.5	C10 - 245	3.83	2.02	15.92	29.4	3.84	4.540	-0.100	OK	1.61	12.75	3.72	
25.0	C10 - 25	3.79	2.00	15.91	26.0	3.77	4.469	-0.100	OK	1.59	12.71	3.66	

Powder	CR10
Tool set	SNUN120412 - Phase 3 (Large)
Area - pressed	250
S Height	6.40
SG	14.45
App. Density	3.39
Press height	8.00
Weight	15.146
Tool	25
	%
	Volume 1.0187 cm ³
	Volatile 2.89%

Powder details	Links
Date :	
Item no : 4001004	Powder sheet
Batch no : 74704	RTP
Co % : 9.5	%
Grain size : <1	μ
PRZ reclaim? :	
PEG content :	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
21.0	CR10 - 21	16.55											
21.5	CR10 - 22	16.38	8.00	15.93	64.5	16.36	19.593	-0.476	H	6.52	13.02	15.87	
22.0	CR10 - 23	16.21	8.00	15.92	59.2	16.23	19.413	-0.400	H	6.57	13.00	15.74	
22.5	CR10 - 24	16.04	7.99	15.92	53.2	16.07	19.246	-0.390	H	6.48	12.95	15.58	
23.0	CR10 - 25	15.86	8.00	15.92	47.0	15.86	19.077	-0.370	H	6.45	12.895	15.38	
23.5	CR10 - 26	15.68	8.00	15.92	40.5	15.66	18.837	-0.350	SH	6.42	12.84	15.18	
24.0	CR10 - 27	15.51	7.98	15.92	36.1	15.52	18.644	-0.340	B	6.40	12.81	15.02	
24.5	CR10 - 28	15.33	8.00	15.92	31.2	15.33	18.333	-0.310	OK	6.36	12.74	14.86	
25.0	CR10 - 29	15.15	7.99	15.92	27.2	15.14	18.071	-0.290	OK	6.34	12.68	14.65	

25.5 CR10-25 14.97 8.01 15.91 22.9 14.95 17.785 -0.250 OK 6.32 12.615 14.49

A1.4.2 Grade CR10 – Powder physical properties, SG and porosity reports

Table A1.16 – Historical screen fraction report for grade CR10 (granule size)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
198.4 g	0.1 g	1.5 g	69.4 g	60.0 g	37.9 g	29.7 g

Table A1.17 – Screen fraction report for grade CR10 (mass percent of previous table)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.03 %	0.8 %	35.0 %	30.2 %	19.1 %	15.0 %

Table A1.18 – Apparent density report of grade CR10 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
81.6 g	25 cc	3.26 g/cm ³	3.28 g/cm ³

Table A1.19 – Sintered density report for grade CR10 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.0387	14.0778	0.0706	0.9977	17	14.55	14.56
2	7.5751	7.1268	0.0706	0.9977	17	14.56	
3	3.7410	3.5555	0.0706	0.9977	17	14.57	

Table A1.20 – Porosity report for best and worst sintered test pieces (midsize) for CR10

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
CR10	24 %	25 %	A00 B00	A00 B00	Very good	good

A1.5 Experimental data sheets for grade 'H1P'

A1.5.1 'H1P' Grade pressing test sheets – negative inserts

Powder details		Links
Date :		Powder sheet
Item no : 4001005		RTP
Batch no : 61673		Spray drying
Co % : 6.0	%	
Grain size : < 1	μ	
PRZ reclaim? :		
PEG content :	%	

Powder	H1P
Tool set	SNUN120412 - Phase 1 (Small)
Area - pressed	248
S Height	1.60
SG	12.89
Volume	0.2547 cm ³
App. Density	3.24
Volatile	2.50%
Press height	2.00
Weight	3.365
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
21.0	H1P-20	3.68	2.00	15.91	48.5	3.68	4.650	-0.18	B	1.68	13.10	3.59	
21.5	H1P-205	3.64	2.00	15.91	43.1	3.65	4.599	-0.16	G	1.67	13.05	3.55	
22.0	H1P-21	3.60	2.00	15.91	36.6	3.59	4.536	-0.14	G	1.66	12.99	3.50	
22.5	H1P-215	3.56	2.00	15.91	30.90	3.56	4.486	-0.12	G	1.65	12.94	3.46	
23.0	H1P-22	3.52	2.01	15.91	25.9	3.52	4.435	-0.100	G	1.65	12.88	3.42	
23.5	H1P-225	3.48	2.00	15.91	25.2	3.50	4.410	-0.100	G	1.64	12.87	3.40	
24.0	H1P-23	3.45	2.01	15.90	20.5	3.45	4.359	-0.09	C	1.63	12.82	3.36	
24.5	H1P-235	3.41	2.00	15.90	17.3	3.41	4.308	-0.09	SS	1.63	12.77	3.31	
25.0	H1P-24	3.37	2.00	15.90	15.8	3.37	4.258	-0.09	SS	1.63	12.74	3.28	

4.72
4.67
4.62
4.57
4.52
4.47
4.41

Good

SS

Powder H1P	
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250
S Height	3.20
SG	12.89
App. Density	3.24
Press height	4.00
Weight	6.729
Tool	25
	%
	Volume 0.509 cm ³
	Volatile 2.50%

Powder details	
Date:	2.02.2006
Item no :	4001005
Batch no :	61673
Co % :	6.0
Grain size :	< 1
PRZ reclaim? :	
PEG content :	
	Links
	Powder sheet
	RTP
	Spray drying

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
21.0	H1P-20	7.35	4.02	15.92	48.7	7.37	9.286	-0.22	B	3.34	15.16	7.26	
21.5	H1P-205	7.28	4.02	15.92	40.50	7.27	9.185	-0.21	Oh	3.30	13.03	7.09	
22.0	H1P-21	7.20	4.06	15.91	34.2	7.18	9.059	-0.20	Oh	3.28	12.98	6.98	
22.5	H1P-215	7.12	4.00	15.91	31.3	7.12	9.008	-0.18	Oh	3.27	12.96	6.95	
23.0	H1P-22	7.05	4.00	15.91	26.6	7.05	8.926	-0.16	Oh	3.26	12.89	6.87	
23.5	H1P-225	6.97	4.00	15.90	24.0	6.99	8.819	-0.15	Oh	3.26	12.86	6.81	
24.0	H1P-23	6.89	4.01	15.90	19.50	6.88	8.692	-0.13	Oh	3.24	12.79	6.71	
24.5	H1P-235	6.81	4.00	15.90	16.6	6.80	8.579	-0.12	Oh	3.23	12.74	6.63	
25.0	H1P-24	6.73	4.00	15.90	14.71	6.72	8.465	-0.120	SS	3.21	12.70	6.55	

Best. 9.67
9.57
9.47
9.37
9.27
9.17
9.06
8.96
8.85

SS

Powder	H1P
Tool set	SNUN120412 - Phase 3 (Large)
Area - pressed	250
S Height	6.40
SG	12.89
App. Density	3.24-3.445
Press height	8.00
Weight	13.459
Tool	25
	%
	Volume 1.0187 cm³
	Volatile 2.50%

Powder details	
Date :	3 02.2006
Links	
Item no :	4001005
Batch no :	61673 74765
Co % :	6.0
Grain size :	<1
PRZ reclaim? :	%
PEG content :	%
	Spray drying
	RTP
	Powder sheet

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
20.0		H1P-20	15.01	8.01	15.91	50.8	15.00	17.963	-0.34	H	6.67	13.17	14.67
20.5		H1P-205	14.86	8.01	15.91	44.2	14.86	17.784	-0.32	H	6.64	13.13	14.50
21.0		H1P-21	14.71	8.01	15.91	40.0	14.71	17.604	-0.30	H	6.62	13.09	14.40
21.5		H1P-215	14.56	8.01	15.91	34.1	14.59	17.366	-0.28	H	6.60	13.05	14.27
22.0		H1P-22	14.41	8.01	15.91	29.5	14.42	17.246	-0.26	SH	6.57	12.99	14.10
22.5		H1P-225	14.25	8.01	15.91	25.4	14.25	17.054	-0.24	B	6.56	12.96	13.94
23.0		H1P-23	14.10	8.00	15.91	21.9	14.09	16.863	-0.22	B	6.54	12.92	13.82
23.5		H1P-235	13.94	8.00	15.92	18.3	13.92	16.648	-0.20	SS	6.51	12.85	13.63
24.0		H1P-24	13.78	8.00	15.92	14.76	13.75	16.338	-0.18	SS	6.49	12.82	13.46

A1.5.2 Grade H1P – Powder physical properties, SG and porosity reports

Table A1.21 – Historical screen fraction report for grade H1P (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
197.8 g	0.1 g	8.2 g	91.8 g	47.7 g	31.2 g	18.8 g

Table A1.22 – Historical screen fraction report for grade H1P (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	4.2 %	46.4 %	24.1 %	15.8 %	9.5 %

Table A1.23 – Apparent density report of grade H1P test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
81.3 g	25 cc	3.25 g/cm ³	3.21 g/cm ³

Table A1.24 – Sintered density report for grade H1P test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	14.4068	13.3621	0.0683	0.9974	18.9	12.91	12.89
2	6.9811	6.5096	0.0683	0.9974	18.9	12.90	
3	3.5012	3.2980	0.0683	0.9974	18.9	12.86	

Table A.25 – Porosity report for best and worst sintered test pieces (midsize) for H1P

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
H1P	21 %	25 %	A00 B00	A00 B02	Very good	OK

A1.6 Experimental data sheets for grade 'H6'

A1.6.1 'H6' Grade pressing test sheets – negative inserts

Powder details		Links
Date :	Item no : 4001006	<u>Powder sheet</u>
	Batch no : 82320	<u>RTP</u>
	Co % : 5.7	% <u>Spray drying</u>
	Grain size : 1 - 1.5	μ
	PRZ reclaim? :	%
	PEG content :	%

Powder	H6
Tool set	SNUN120412 - Phase 1 (Small)
Area - pressed	248
S Height	2.53
SG	14.95
App. Density	3.03
Press height	3.16
Weight	6.157
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	H6-20	6.87											
20.5	H6-205	6.80											
21.0	H6-21	6.73											
21.5	H6-215	6.66	3.17	15.94	113.7	6.68	9.099	-0.420	1	2.60	13.12	6.80	
22.0	H6-22	6.59	3.17	15.94	102.0	6.58	8.990	-0.39	H	2.57	13.07	6.42	
22.5	H6-225	6.52	3.16	15.94	93.0	6.51	8.881	-0.37	H	2.55	12.04	6.34	
23.0	H6-23	6.45	3.16	15.94	86.2	6.46	8.799	-0.350	OL	2.54	12.99	6.30	
23.5	H6-235	6.38	3.15	15.94	82.3	6.40	8.731	-0.350	OL	2.54	12.00	6.24	
24.0	H6-24	6.30	3.14	15.93	75.0	6.30	8.622	-0.350	OL	2.51	12.92	6.16	
24.5	H6-245	6.23	3.13	15.93	65.5	6.22	8.486	-0.33	OL	2.50	12.87	6.07	
25.0	H6-25	6.16	3.16	15.93	55.5	6.16	8.405	-0.28	OL	2.51	12.79	6.00	
25.5	H6-255	6.09	3.16	15.93	50.2	6.10	8.349	-0.26	SS	2.50	12.74	5.94	

A1.6.2 Grade H6 – Powder physical properties, SG and porosity reports

Table A1.26 – Screen fraction report for grade H6 (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
196.5 g	0.2 g	9.5 g	91.6 g	45.2 g	28.8 g	21.2 g

Table A1.27 – Screen fraction report for grade H6 (mass percent of previous table)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	4.8 %	46.6 %	23.0 %	14.7 %	10.8 %

Table A1.28 – Apparent density report of grade H6 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
71.8 g	25 cc	2.87 g/cm ³	3.41 g/cm ³

Table A1.29 – Sintered density report for grade H6 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	12.2684	11.5154	0.0685	0.9974	18.7	14.90	14.90
2	6.0650	5.7277	0.0685	0.9974	18.7	14.91	
3	6.2442	5.8941	0.0685	0.9974	18.7	14.88	

Table A1.30 – Porosity report for best and worst sintered test pieces (midsize) for H6

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
H6	23.5 %	25.5 %	A00 B00	A00 B00	Very good	Very good

A1.7 Experimental data sheets for grade 'K15M'

A1.7.1 'K15M' Grade pressing test sheets – negative inserts

Powder details		Links
Date :		
Item no : 4001009		<u>Powder sheet</u>
Batch no : 66531		<u>RTP</u>
Co % : 7.3	%	<u>Spray drying</u>
Grain size : 1 - 2	μ	
PRZ reclaim? :		
PEG content :	%	

Powder	K15M	
Tool set	SNUN120412 - Phase 1 (Small size)	
Area - pressed	250	
S Height	1.60	
SG	12.77	Volume 0.2547 cm ³
App. Density	3.225	Volatile 2.48%
Press height	2.00	
Weight	3.333	
Tool	25	%

Equivalent Tool %	Pressing (Green)								Sintered			
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
21.0	K15M - 21	3.64	2.00	15.92	50.5	3.64	4.419	-0.200	H	1.66	13.125	3.55
21.5	K15M - 215	3.61	2.00	15.92	45.2	3.62	4.395	-0.180	H	1.64	13.09	3.53
22.0	K15M - 22	3.57	2.00	15.92	38.6	3.57	4.346	-0.160	OK	1.64	13.025	3.49
22.5	K15M - 225	3.53	2.00	15.92	32.3	3.52	4.286	-0.140	OK	1.63	12.995	3.45
23.0	K15M - 23	3.49	2.00	15.92	28.1	3.49	4.237	-0.130	(B)	1.62	12.925	3.41
23.5	K15M - 235	3.45	2.00	15.91	25.0	3.46	4.201	-0.120	OK	1.63	12.885	3.38
24.0	24K15M -	3.41	2.00	15.92	19.8	3.40	4.140	-0.110	OK	1.62	12.83	3.31
24.5	K15M - 245	3.37	2.00	15.91	16.5	3.36	4.079	-0.100	OK	1.60	12.775	3.28
25.0	K15M - 25	3.33	2.00	15.91	14.7	3.33	4.043	-0.100	S	1.60	12.735	3.24

Powder	K15M			
Tool set	SNUN120412 - Phase 2 (Midsize)			
Area - pressed	250.1			
S Height	3.20			
SG	12.77	Volume	0.5093 cm ³	
App. Density	3.225	Volatile	2.48%	
Press height	4.00			
Weight	6.665			
Tool	25			%

Powder details		Links
Date:	17.02.2006.	
Item no :	4001009	<u>Powder sheet</u>
Batch no :	66531	<u>RTP</u>
Co % :	7.3	% <u>Spray drying</u>
Grain size :	1 - 2	μ
PRZ reclaim? :		
PEG content :		%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
21.0	K15M - 21	7.28	4.00	15.92	47.5	7.28	8.985	-0.260	H	3.29	13.09	7.10	
21.5	K15M - 215	7.21	4.00	15.91	41.6	7.21	8.898	-0.240	H	3.27	13.055	7.03	
22.0	K15M - 22	7.13	4.00	15.91	35.7	7.14	8.812	-0.220	OK	3.25	13.02	6.96	
22.5	K15M - 225	7.06	4.00	15.91	31.0	7.07	8.737	-0.200	OK	3.25	12.96	6.89	
23.0	K15M - 23	6.98	4.00	15.91	26.2	6.98	8.638	-0.180	B	3.24	12.905	6.80	
23.5	K15M - 235	6.90	4.01	15.91	21.7	6.90	8.539	-0.150	OK	3.23	12.85	6.73	
24.0	24K15M -	6.82	4.00	15.91	18.4	6.81	8.428	-0.140	OK	3.23	12.79	6.64	
24.5	K15M - 245	6.74	4.00	15.91	15.9	6.75	8.342	-0.130	OK	3.22	12.745	6.57	
25.0	K15M - 25	6.67	4.01	15.90	13.2	6.67	8.255	-0.120	SS	3.20	12.69	6.48	

A1.7.2 Grade K15M – Powder physical properties, SG and porosity reports

Table A1.31 – Historical screen fraction report for grade K15M

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
193.2 g	0.1 g	1.7 g	71.4 g	47.3 g	39.1 g	33.8 g

Table A1.32 – Historical screen fraction report for grade K15M (mass percent)

>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
0.1 %	0.9 %	36.9 %	24.5 %	20.2 %	17.5 %

Table A1.33 – Apparent density report of grade K15M test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
77.9 g	25 cc	3.12 g/cm ³	3.19 g/cm ³

Table A1.34 – Sintered density report for grade K15M test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	13.7959	12.7901	0.0676	0.9974	18.8	12.82	12.82
2	6.8955	6.4275	0.0676	0.9974	18.8	12.84	
3	3.4392	3.2389	0.0676	0.9974	18.8	12.80	

Table A1.35 – Porosity report for best and worst sintered test pieces (midsize) for K15M

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
K15M	23.0 %	25.0 %	A02 B02	A02 B02	Acceptable	Acceptable

A1.8 Experimental data sheets for grade 'MT16'

A1.8.1 'MT16' Grade pressing test sheets – negative inserts

Powder details		Links
Date :		<u>Powder sheet</u>
Item no : 4001007		<u>RTP</u>
Batch no :		<u>Spray drying</u>
Co % : 16.0	%	
Grain size : 2 - 3	μ	
PRZ reclaim? :	%	
PEG content :	%	

Powder	MT16
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	13.85
Volume	0.2547 cm ³
App. Density	Volatile 2.70%
Press height	2.00
Weight	3.623
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0 ²⁴	MT16 - 20	3.70 4.12	2.00	15.92	22.3	3.69	4.017	-0.120	OK	1.66	12.86	3.57	
19.5 ²³⁵	MT16 - 205	3.73 4.08	2.00	15.92	25.8	3.75	4.082	-0.130	OK	1.60	12.92	3.64	
20.0	MT16 - 21	4.04	2.00	15.92	53.6	4.04	4.443	-0.200	F1	1.65	13.23	3.93	
20.5	MT16 - 215	4.00	2.00	15.92	48.7	3.99	4.388	-0.190	F1	1.64	13.19	3.88	
21.0	MT16 - 22	3.96	2.00	15.92	45.1	3.96	4.345	-0.180	OK	1.64	13.16	3.85	
21.5	MT16 - 225	3.92	2.00	15.92	40.3	3.91	4.290	-0.170	OK	1.63	13.12	3.80	
22.0	MT16 - 23	3.88	2.00	15.92	35.9	3.87	4.235	-0.160	OK	1.67	13.06	3.76	
22.5	MT16 - 235	3.84	2.00	15.92	32.7	3.84	4.192	-0.150	OK	1.62	13.03	3.73	
23.0	MT16 - 24	3.79	2.00	15.92	28.4	3.78	4.126	-0.140	OK	1.61	12.97	3.67	

Powder	MT16
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	13.85
Volume	0.5093 cm ³
App. Density	Volatile 2.70%
Press height	4.00
Weight	7.244
Tool	25 %

Powder details		Links
Date :	Item no : 4001007	Powder sheet
	Batch no : 93577	RTP
	Co % : 16.0	Spray drying
	Grain size : 2 - 3	μ
	PRZ reclaim? :	
	PEG content :	%

Equivalent Tool %	Pressing (Green)								Sintered			
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
19.0	MT16 - 20	8.24	3.99	15.93	65.1	8.23	9.131	-0.310	H	3.32	13.33	8.01
19.5	MT16 - 205	8.16	3.99	15.93	61.9	8.17	9.053	-0.210	H	3.29	13.31	7.95
20.0	MT16 - 21	8.08	3.99	15.93	54.7	8.07	8.593	-0.300	OK	3.28	13.26	7.85
20.5	MT16 - 215	8.00	3.99	15.93	49.9	8.00	8.865	-0.280	OK	3.27	13.21	7.79
21.0	MT16 - 22	7.92	4.00	15.93	44.2	7.94	8.798	-0.250	OK	3.26	13.16	7.72
21.5	MT16 - 225	7.84	4.01	15.92	37.3	7.82	8.687	-0.220	OK	3.25	13.09	7.61
22.0	MT16 - 23	7.75	4.00	15.92	34.2	7.75	8.587	-0.210	OK	3.25	13.04	7.54
22.5	MT16 - 235	7.67	4.00	15.92	31.1	7.68	8.510	-0.200	OK	3.23	13.01	7.47
23.0	MT16 - 24	7.59	4.01	15.92	27.9	7.60	8.432	-0.180	OK	3.22	12.95	7.40

Powder MT16			
Tool set SNUN120412 - Phase 3 (Largesize)			
Area - pressed	250.1		
S Height	6.40		
SG	13.85	Volume	1.0187 cm ³
App. Density		Volatile	2.70%
Press height	8.00		
Weight	14.490		
Tool	25		%

Powder details		Links
Date :		
Item no :	4001007	Powder sheet
Batch no :	93577	RTP
Co % :	16.0	% Spray drying
Grain size :	2 - 3	μ
PRZ reclaim? :		
PEG content :		%

Equivalent Tool %	Pressing (Green)									Sintered			
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0	MT16 - 20	16.48	8.00		64.5	16.46		-0.410	H	6.63	13.29	16.01	
19.5	MT16 - 205	16.32	7.99	15.93	60.4	16.33	18.201	-0.400	H	6.61	13.26	15.99	
20.0	MT16 - 21	16.16	7.99	15.92	54.1	16.17	18.034	-0.380	OK	6.60	13.22	15.73	
20.5	MT16 - 215	16.00	8.00	15.92	49.4	16.02	17.877	-0.360	OK	6.58	13.18	15.58	
21.0	MT16 - 22	15.84	8.00	15.92	42.8	15.83	17.687	-0.340	OK	6.55	13.11	15.39	
21.5	MT16 - 225	15.67	8.00	15.92	38.3	15.69	17.520	-0.320	OK	6.53	13.07	15.26	
22.0	MT16 - 23	15.51	8.01	15.92	33.8	15.50	17.330	-0.300	OK	6.50	13.02	15.08	
22.5	MT16 - 235	15.34	8.00	15.91	31.4	15.35	17.151	-0.290	OK	6.48	12.98	14.96	
23.0	MT16 - 24	15.17	8.01	15.91	27.7	15.19	17.039	-0.270	OK	6.47	12.93	14.77	

A1.8.2 Grade MT16 – Powder physical properties, SG and porosity reports

Table A1.36 – Screen fraction report for grade MT16 (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
197.9 g	0.34 g	15.0 g	88.9 g	47.2 g	26.8 g	19.6 g

Table A1.37 – Screen fraction report for grade MT16 (mass percent of previous table)

>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
0.2 %	7.6 %	44.9 %	23.9 %	13.5 %	9.9 %

Table A1.38 – Apparent density report of grade MT16 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
85.8 g	25 cc	3.43 g/cm ³	3.37 g/cm ³

Table A1.39 – Sintered density report for grade MT16 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.0929	14.0733	0.0681	0.9974	19.2	13.84	13.82
2	7.6168	7.1357	0.0681	0.9974	19.2	13.83	
3	3.7604	3.5568	0.0681	0.9974	19.2	13.80	

Table A1.40 – Porosity report for best and worst sintered test pieces (midsize) for MT16

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
MT16	22.0 %	24.0 %	A00 B00	A00 B00	Very good	Good

A1.9 Experimental data sheets for grade 'MZ15'

A1.9.1 'MZ15' Grade pressing test sheets – negative inserts

Powder details	
Date :	Links
Item no : 4001027	Powder sheet
Batch no : 93402	RTP
Co % : 15.2	%
Grain size : 2 - 4	μ
PRZ reclaim? <u>(y)</u>	%
PEG content :	%

Powder	MZ15
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	13.92
AVE	0.2547 cm ³
App. Density	Volatile 2.50%
Press height	2.00
Weight	3.634
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0	MZ15 - 19	4.13	2.00	15.93	49.8	4.13	4.895	-0.200	41	1.68	13.36	4.03	
19.5	MZ15 - 195	4.09	2.00	15.93	45.7	4.09	4.848	-0.190	41	1.66	13.32	3.99	
20.0	MZ15 - 20	4.05	2.00	15.93	40.8	4.04	4.788	-0.180	41	1.64	13.25	3.94	
20.5	MZ15 - 205	4.01	2.00	15.93	37.1	4.01	4.741	-0.170	OK	1.63	13.24	3.91	
21.0	MZ15 - 21	3.97	2.00	15.93	34.5	3.98	4.706	-0.160	OK	1.63	13.21	3.89	
21.5	MZ15 - 215	3.93	2.00	15.93	30.4	3.92	4.646	-0.150	(B)G	1.62	13.14	3.83	
22.0	MZ15 - 22	3.89	2.00	15.92	27.0	3.89	4.599	-0.140	(B)K	1.61	13.09	3.78	
22.5	MZ15 - 225	3.85	2.00	15.92	24.4	3.85	4.552	-0.130	OK	1.61	13.05	3.75	
23.0	MZ15 - 23	3.81	2.00	15.92	21.7	3.81	4.505	-0.120	S	1.60	12.98	3.71	

Powder	MZ15			
Tool set	SNUN120412 - Phase 2 (Midsize)			
Area - pressed	250.1			
S Height	3.20			
SG	13.92	AVE	Volume	0.5093 cm ³
App. Density			Volatile	2.50%
Press height	4.00			
Weight	7.267			
Tool	25			%

Powder details		Links
Date :		<u>Powder sheet</u>
Item no :	4001027	<u>RTP</u>
Batch no :	93402	<u>Spray drying</u>
Co % :	15.2	%
Grain size :	2 - 4	μ
PRZ reclaim? :	y	
PEG content :		%

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
	19.0	MZ15 - 19	8.26	3.99	15.93	44.8	8.25	9.931	-0.250	H	3.33	13.29	8.06
	19.5	MZ15 - 195	8.18	4.00	15.92	39.0	8.16	9.811	-0.230	H	3.31	13.23	7.98
	20.0	MZ15 - 20	8.10	4.00	15.92	35.89	8.10	9.762	-0.210	H	3.31	13.19	7.92
	20.5	MZ15 - 205	8.02	4.00	15.92	31.8	8.01	9.654	-0.190	H	3.30	13.14	7.85
	21.0	MZ15 - 21	7.94	4.00	15.92	28.6	7.94	9.557	-0.180	H	3.29	13.10	7.77
	21.5	MZ15 - 215	7.86	4.00	15.92	25.8	7.84	9.437	-0.180	OK	3.29	13.05	7.68
	22.0	MZ15 - 22	7.78	4.00	15.92	23.7	7.78	9.341	-0.170	B	3.27	13.02	7.61
	22.5	MZ15 - 225	7.69	4.00	15.92	20.9	7.68	9.221	-0.160	G	3.26	12.97	7.51
	23.0	MZ15 - 23	7.61	4.00	15.91	18.9	7.61	9.125	-0.150	G	3.25	12.93	7.45

Powder	MZ15
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	13.92
AVE	1.0187 cm ³
App. Density	Volatile 2.50%
Press height	8.00
Weight	14.535
Tool	25 %

Powder details	Links
Date :	
Item no : 4001027	Powder sheet
Batch no : 93402	RTP
Co % : 15.2 %	Spray drying
Grain size : 2 - 4 μ	
PRZ reclaim? : y	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0	MZ15 - 19	16.53	7.99	15.92	45.6	16.54	19.859	-0.350	H	6.67	13.28	16.19	
19.5	MZ15 - 195	16.37	7.99	15.92	40.1	16.39	19.691	-0.340	H	6.65	13.23	16.04	
20.0	MZ15 - 20	16.21	8.00	15.92	36.5	16.23	19.522	-0.320	H	6.63	13.18	15.87	
20.5	MZ15 - 205	16.05	7.99	15.92	32.5	16.06	19.924	-0.310	H	6.60	13.12	15.70	
21.0	MZ15 - 21	15.89	8.00	15.92	29.1	15.90	19.078	-0.290	H	6.59	13.08	15.55	
21.5	MZ15 - 215	15.72	8.00	15.92	26.3	15.74	18.850	-0.270	OK	6.58	13.04	15.40	
22.0	MZ15 - 22	15.56	8.00	15.92	23.2	15.57	18.670	-0.250	B	6.56	13.00	15.22	
22.5	MZ15 - 225	15.39	8.01	15.92	21.2	15.40	18.478	-0.230	G	6.54	12.95	15.07	
23.0	MZ15 - 23	15.22	8.00	15.91	18.9	15.23	18.286	-0.220	G	6.51	12.89	14.96	

A1.9.2 Grade MZ15 – Powder physical properties, SG and porosity reports

Table A1.41 – Historical screen fraction report for grade MZ15

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
196.5 g	0.4 g	15.6 g	102.5 g	41.7 g	21.0 g	15.3 g

Table A1.42 – Historical screen fraction report for grade MZ15 (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.2 %	7.9 %	52.2 %	21.2 %	10.7 %	7.8 %

Table A1.43 – Apparent density report of grade MZ15 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
84.2 g	25 cc	3.37 g/cm ³	3.31 g/cm ³

Table A1.44 – Sintered density report for grade MZ15 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.2493	14.2267	0.0683	0.9974	18.9	13.94	13.93
2	7.6817	7.1999	0.0683	0.9974	18.9	13.93	
3	3.7945	3.5911	0.0683	0.9974	18.9	13.93	

Table A1.45 – Porosity report for best and worst sintered test pieces (midsize) for MZ15

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
MZ15	23 %	24 %	A00 B00	A00 B00	Very good	Very good

A1.10 Experimental data sheets for grade 'N6/194'

A1.10.1 'N6/194' Grade pressing test sheets – negative inserts

Powder details		Links
Date :		<u>Powder sheet</u>
Item no : 4001001		<u>RTP</u>
Batch no : 88444		<u>Spray drying</u>
Co % : 6.0	%	
Grain size : 1 - 3	μ	
PRZ reclaim? :		
PEG content :	%	

Powder	N6 / 194
Tool set	SNUN120412 - Phase 1 (Small)
Area - pressed	250
S Height	1.60
SG	14.87
Volume	0.2547 cm ³
App. Density	3.595
Volatile	1.81%
Press height	2.00
Weight	3.856
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	N6-20	4.30	2.01	15.91	39.5	4.30	4.591	-0.140	H	1.69	13.185	4.21	
20.5	N6-205	4.26	2.00	15.91	35.4	4.26	4.548	-0.130	oh	1.67	13.15	4.18	
21.0	N6-21	4.21	2.00	15.91	30.9	4.20	4.495	-0.120	oh	1.66	13.08	4.12	
21.5	N6-215	4.17	2.00	15.91	27.9	4.17	4.452	-0.110	C	1.65	13.06	4.09	
22.0	N6-22	4.13	2.00	15.91	23.6	4.12	4.399	-0.100	B	1.64	13.025	4.03	
22.5	N6-225	4.08	2.01	15.91	19.3	4.07	4.356	-0.080	B	1.63	12.96	3.99	
23.0	N6-23	4.04	2.00	15.91	17.0	4.05	4.324	-0.080	B	1.62	12.90	3.96	
23.5	N6-235	3.99	2.00	15.91	15.9	4.01	4.292	-0.080	C	1.62	12.87	3.94	
24.0	N6-24	3.95	1.99	15.91	14.3	3.95	4.249	-0.090	C	1.61	12.83	3.87	

Powder N6 / 194			
Tool set SNUN120412 - Phase 2 (Midsize)			
Area - pressed	250		
S Height	3.20		
SG	14.87	Volume	0.5093 cm ³
App. Density	3.595	Volatile	1.81%
Press height	4.00		
Weight	7.710		
Tool	25		%

Powder details		Links
Date :		
Item no : 4001001		Powder sheet
Batch no : 88444		RTP
Co % : 6.0	%	Spray drying
Grain size : 1 - 3	μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	N6-20	8.60	4.00	15.92	38.6	8.60	9.250	-0.200	H	3.33	13.17	8.44	
20.5	N6-205	8.51	4.01	15.92	33.0	8.52	4.164	-0.170	OK	3.32	13.125	8.36	
21.0	N6-21	8.43	4.00	15.91	29.2	8.42	9.067	-0.160	OK	3.31	13.07	8.27	
21.5	N6-215	8.34	4.00	15.91	25.9 26.6	8.33	8.960	-0.150	OK	3.29	13.03	8.18	
22.0	N6-22	8.25	4.00	15.91	23.0	8.25	8.863	-0.140	B	3.28	12.985	8.10	
22.5	N6-225	8.16	4.00	15.91	20.5	8.18	8.788	-0.130	(B)	3.27	12.955	8.02	
23.0	N6-23	8.07	4.00	15.90	17.5	8.07	8.691	-0.120	B	3.26 3.25	12.91 12.85	7.92	
23.5	N6-235	7.98	3.99	15.90	15.9	7.98	8.594	-0.130	OK	3.25	12.85	7.82	
24.0	N6-24	7.89	3.99	15.90	13.7	7.90	8.497	-0.130	G	3.23	12.82	7.75	

Powder	N6 / 194		
Tool set	SNUN120412 - Phase 3 (Large)		
Area - pressed	250		
S Height	6.40		
SG	14.87	Volume	1.0187 cm ³
App. Density	3.595	Volatile	1.81%
Press height	8.00		
Weight	15.422		
Tool	25	%	

Powder details		Links
Date :		<u>Powder sheet</u>
Item no :	4001001	<u>RTP</u>
Batch no :	88444	<u>Spray drying</u>
Co % :	6.0	%
Grain size :	1 - 3	μ
PRZ reclaim? :		%
PEG content :		%

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
	20.0	N6-20	17.20	8.00	15.91	38.6	17.23	18.587	-0.300	H	6.65	13.155	16.93
	20.5	N6-205	17.03	8.00	15.91	34.4	17.05	18.425	-0.280	OK	6.63	13.11	16.74
	21.0	N6-21	16.86	8.00	15.91	29.8	16.86	18.241	-0.260	OK	6.61	13.07	16.56
	21.5	N6-215	16.68	7.99	15.91	26.1	16.69	18.057	-0.250	G	6.57	13.02	16.39
	22.0	N6-22	16.51	8.00	15.91	22.8	16.49	17.851	-0.230	B	6.54	12.965	16.18
	22.5	N6-225	16.33	8.00	15.91	19.9	16.31	17.624	-0.210	B	6.52	12.92	16.00
	23.0	N6-23	16.15	8.00	15.91	17.8	16.17	17.483	-0.190	B	6.50	12.87	15.86
	23.5	N6-235	15.97	8.00	15.91	15.2	15.97	17.289	-0.180	G	6.48	12.835	15.66
	24.0	N6-24	15.79	7.99	15.90	13.2	15.77	17.072	-0.190	OK	6.44	12.775	15.48

A1.10.2 Grade N6/194 – Powder physical properties, SG and porosity reports

Table A1.46 – Historical screen fraction report for grade N6/194 (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
197.4 g	0.2 g	13.4 g	91.0 g	45.0 g	29.0 g	18.8 g

Table A1.47 – Historical screen fraction report for grade N6/194 (mass percent)

>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
0.1 %	6.8 %	46.1 %	22.8 %	14.7 %	9.5 %

Table A1.48 – Apparent density report of grade N6/194 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
90.5 g	25 cc	3.62 g/cm ³	3.72 g/cm ³

Table A1.49 – Sintered density report for grade N6/194 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	16.1811	15.1651	0.0686	0.9981	15.3	14.89	14.89
2	8.0971	7.6224	0.0686	0.9981	15.3	14.88	
3	4.0336	3.8320	0.0686	0.9981	15.3	14.90	

Table A1.50 – Porosity report for best and worst sintered test pieces (midsize) for N6/194

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
N6/194	22.5 %	24 %	A00 B00	A00 B00	Excellent	Excellent

A1.11 Experimental data sheets for grade 'P7'

A1.11.1 'P7' Grade pressing test sheets – negative inserts

Powder details		Links
Date :	Item no : 4001014	<u>Powder sheet</u>
	Batch no : 87700	<u>RTP</u>
	Co % : 7.5	<u>Spray drying</u>
	Grain size : 3 - 4	μ
	PRZ reclaim? :	%
	PEG content :	%

Powder P7	
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	14.73
App. Density	3.800
Press height	2.00
Weight	3.840
Tool	25
	%
	Volume 0.2547 cm ³
	Volatile 2.34%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0	P7 - 19	4.37											
19.5	P7 - 195	4.32	2.00	15.91	38.7	4.32	4.687	-0.170	SH	1.67	13.22	4.22	
20.0	P7 - 20	4.28	1.99	15.91	34.9	4.28	4.644	-0.160	OK	1.66	13.16	4.17	
20.5	P7 - 205	4.24	1.99	15.91	31.6	4.24	4.686	-0.150	OK	1.65	13.13	4.13	
21.0	P7 - 21	4.20	2.00	15.91	28.2	4.20	4.642	-0.140	OK	1.64	13.085	4.09	
21.5	P7 - 215	4.15	2.00	15.91	25.3	4.16	4.598	-0.130	OK	1.64	13.03	4.06	
22.0	P7 - 22	4.11	2.00	15.91	21.7	4.10	4.543	-0.120	P	1.63	12.99	4.01	
22.5	P7 - 225	4.07	2.00	15.91	19.4	4.07	4.498	-0.110	OK	1.63	12.95	3.97	
23.0	P7 - 23	4.02	2.00	15.91	14.8	3.99	4.410	-0.100	OK	1.62	12.85	3.89	

Powder	P7
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	14.73
App. Density	3.800
Press height	4.00
Weight	7.678
Tool	25
	%
	Volume 0.5093 cm ³
	Volatile 2.34%

Powder details		Links
Date :		
Item no :	4001014	Powder sheet
Batch no :	87700	RTP
Co % :	7.5	%
Grain size :	3 - 4	μ
PRZ reclaim? :		%
PEG content :		%

Equivalent Tool %	Pressing (Green)								Sintered			
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
19.0	P7 - 19	8.73										
19.5	P7 - 195	8.65	4.00	15.91	33.9	8.66	9.782	-0.190	SH	3.37	13.17	8.45
20.0	P7 - 20	8.56	4.00	15.90	29.9	8.55	9.669	-0.180	OK	3.35	13.13	8.35
20.5	P7 - 205	8.48	4.00	15.90	27.9	8.44	9.590	-0.170	OK	3.34	13.10	8.29
21.0	P7 - 21	8.39	4.00	15.90	25.0	8.39	9.488	-0.160	OK	3.33	13.05	8.20
21.5	P7 - 215	8.30	4.00	15.90	21.9	8.29	9.375	-0.150	OK	3.30	13.00	8.10
22.0	P7 - 22	8.22	4.00	15.90	19.2	8.20	9.262	-0.140	(B)	3.30	12.94	8.01
22.5	P7 - 225	8.13	4.00	15.90	17.0	8.11	9.138	-0.130	OK	3.30	12.90	7.94
23.0	P7 - 23	8.04	4.00	15.91	8.05	8.05	9.048	-0.120	OK	3.28	12.85	7.86

15.0

Powder P7			
Tool set SNUN120412 - Phase 3 (Largesize)			
Area - pressed	250.1		
S Height	6.40		
SG	14.73	Volume	1.0187 cm ³
App. Density	3.800	Volatile	2.34%
Press height	8.00		
Weight	15.357		
Tool	25	%	

Powder details		Links
Date :		
Item no : 4001014		Powder sheet
Batch no : 87700		RTP
Co % : 7.5	%	Spray drying
Grain size : 3 - 4	μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	P7 - 20	17.13	8.00	15.91	29.1	17.13	19.457	-0.240	H	6.69	13.11	16.75	
20.5	P7 - 205	16.96	8.00	15.90	27.4	16.99	19.928	-0.230	OK	6.68	13.08	16.57	
21.0	P7 - 21	16.78	8.00	15.90	23.9	16.76	19.071	-0.220	B	6.64	13.01	16.38	
21.5	P7 - 215	16.61	8.00	15.90	22.0	16.64	18.911	-0.210	OK	6.62	12.99	16.26	
22.0	P7 - 22	16.44	8.00	15.90	19.9	16.43	18.830	-0.200	OK	6.60	12.94	16.05	
22.5	P7 - 225	16.26	8.00	15.90	17.7	16.27	18.647	-0.190	OK	6.57	12.89	15.89	
23.0	P7 - 23	16.08	8.00	15.90	15.5	16.09	18.452	-0.180	OK	6.55	12.85	15.72	
23.5	P7 - 235	15.90	8.00	15.90	13.6	15.89	18.234	-0.170	OK	6.51	12.78	15.52	
24.0	P7 - 24	15.72	7.99	15.91	12.3	15.72	18.028	-0.170	SS	6.50	12.74	15.26	

A1.11.2 Grade P7 – Powder physical properties, SG and porosity reports

Table A1.51 – Historical screen fraction report for grade P7 (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
197.2 g	0.2 g	9.1 g	94.8 g	43.8 g	28.1 g	21.3 g

Table A1.52 – Historical screen fraction report for grade P7 (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	4.6 %	48.1 %	22.2 %	14.3 %	10.8 %

Table A1.53 – Apparent density report of grade P7 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
84.6 g	25 cc	3.38 g/cm ³	3.72 g/cm ³

Table A1.54 – Sintered density report for grade P7 test pieces (best shrinkages)

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	16.0605	15.0441	0.0685	0.9968	21.7	14.76	14.75
2	8.1002	7.6219	0.0685	0.9968	21.7	14.77	
3	4.0069	3.804	0.0685	0.9968	21.7	14.72	

Table A1.55 – Porosity report for best and worst sintered test pieces (midsize) for P7

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
P7	22 %	23 %	A00 B00	A00 B00	Good	Good

A1.12 Experimental data sheets for grade 'P25M'

A1.12.1 'P25M' Grade pressing test sheets – negative inserts

Powder details	
Date :	Links
Item no : 4001010	Powder sheet
Batch no : 87699	RTP
Co % : 9.5	%
Grain size : 1 - 2	μ
PRZ reclaim? :	%
PEG content :	%

Powder	P25M
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	12.59
App. Density	3.120
Press height	2.00
Weight	3.275
Tool	25
	%
	Volume 0.2547 cm ³
	Volatile 2.12%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	P25M - 215	3.65	2.00	15.92	36.7	3.65	4.560	-0.170	Ⓘ	1.65	13.22	3.56	
20.5	P25M - 22	3.62	2.00	15.91	32.6	3.61	4.510	-0.160	OK	1.64	13.185	3.52	
21.0	P25M - 225	3.58	2.00	15.91	29.3	3.58	4.460	-0.150	OK	1.64	13.14	3.49	
21.5	P25M - 23	3.54	2.00	15.91	25.7	3.53	4.410	-0.140	OK	1.62	13.095	3.45	
22.0	P25M - 235	3.50	2.00	15.91	22.1	3.50	4.361	-0.130	OK	1.63	13.07	3.41	
22.5	P25M - 24	3.47	1.99	15.91	19.8	3.46	4.311	-0.130	OK	1.51	13.00	3.37	
23.0	P25M - 245	3.43	2.00	15.91	17.1	3.43	4.261	-0.120	S	1.61	12.93	3.33	
23.5	P25M - 25	3.39	1.99	15.91	15.6	3.40	4.224	-0.120	S	1.60	12.90	3.31	
24.0	P25M - 255	3.35	2.00	15.91	12.8	3.34	4.162	-0.110	S	1.59	12.83	3.26	

Powder	P25M
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	12.59
App. Density	3.120
Press height	4.00
Weight	6.548
Tool	25
	%
	Volume 0.5093 cm ³
	Volatile 2.12%

Powder details		Links
Date :		
Item no : 4001010		Powder sheet
Batch no : 87699		RTP
Co % : 9.5	%	Spray drying
Grain size : 1 - 2	μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	P25M - 215	7.30	4.00	15.91	32.5	7.29	9.265	-0.200	5	3.34	13.20	7.12	
20.5	P25M - 22	7.23	4.00	15.91	29.4	8.24	9.188	-0.190	OK	3.31	13.14	7.07	
21.0	P25M - 225	7.16	4.00	15.91	26.4	7.16	9.099	-0.180	OK	3.30	13.09	7.00	
21.5	P25M - 23	7.08	4.00	15.91	23.0	7.07	8.985	-0.170	OK	3.27	13.04	6.91	
22.0	P25M - 235	7.01	3.99	15.91	21.2	7.01	8.896	-0.170	OK	3.26	13.01	6.84	
22.5	P25M - 24	6.93	4.00	15.91	18.2	6.93	8.795	-0.160	OK	3.25	12.97	6.76	
23.0	P25M - 245	6.86	4.00	15.91	15.5	6.85	8.693	-0.150	S	3.24	12.91	6.69	
23.5	P25M - 25	6.78	4.00	15.90	13.0	6.77	8.579	-0.140	S	3.23	12.88	6.60	
24.0	P25M - 255	6.70	4.02	15.90	11.1	6.69	8.465	-0.120	S	3.23	12.79	6.53	

Powder	P25M
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	12.59
App. Density	3.120
Press height	8.00
Weight	13.097
Tool	25
	%
	Volume 1.0187 cm ³
	Volatile 2.12%

Powder details		Links
Date :		
Item no :	4001010	Powder sheet
Batch no :	87699	RTP
Co % :	9.5	% Spray drying
Grain size :	1 - 2	μ
PRZ reclaim? :		
PEG content :		%

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
21.5		P25M - 215	14.17	8.00	15.91	22.6	14.17	18.080	-0.250	5	6.57	13.03	13.84
22.0		P25M - 22	14.02	8.00	15.91	20.0	14.00	17.864	-0.240	5	6.54	12.98	13.67
22.5		P25M - 225	13.87	8.00	15.91	17.9	13.88	17.685	-0.230	5	6.53	12.96	13.56
23.0		P25M - 23	13.72	8.00	15.90	15.4	13.72	17.494	-0.220	5	6.50	12.89	13.40
23.5		P25M - 235	13.56	8.00	15.90	13.5	13.55	17.277	-0.210	5	6.48	12.82	13.24
24.0		P25M - 24	13.41	8.00	15.90	11.8	13.40	17.073	-0.200	5	6.44	12.78	13.09
24.5		P25M - 245	13.25	8.00	15.90	10.4	13.25	16.870	-0.190	5	6.43	12.76	12.98
25.0		P25M - 25	13.10	8.00	15.90	9.1	13.09	16.667	-0.190	5	6.40	12.71	12.79
25.5		P25M - 255	12.94	7.98	15.90	7.9	12.92	16.437	-0.200	5	6.35	12.65	12.61

A1.12.2 Grade P25M – Powder physical properties, SG and porosity reports

Table A1.56 – Historical screen fraction report for grade P25M (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
195.0 g	0.2 g	12.2 g	93.0 g	46.2 g	26.9 g	16.6 g

Table A1.57 – Screen fraction report for grade P25M (mass percent)

>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
0.1 %	6.2 %	47.7 %	23.7 %	13.8 %	8.5 %

Table A1.58 – Apparent density report of grade P25M test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
75.2 g	25 cc	3.01 g/cm ³	3.14 g/cm ³

Table A1.59 – Sintered density report for grade P25M test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	13.8477	12.8145	0.0689	0.9971	20.5	12.53	12.56
2	7.0758	6.5841	0.0689	0.9971	20.5	12.59	
3	3.5593	3.3455	0.0689	0.9971	20.5	12.55	

Table A1.60 – Porosity report for best and worst sintered test pieces (midsize) for P25M

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
P25M	20 %	24 %	A00 B00	A00 B00	Very good	OK– some A&B type

A1.13 Experimental data sheets for grade 'P30M'

A1.13.1 'P30M' Grade pressing test sheets – negative inserts

Powder details	
Date :	Links
Item no : 4001011	Powder sheet
Batch no : 33028	RTP
Co % : 10.0	%
Grain size : 2 - 3	μ
PRZ reclaim? :	%
PEG content :	%

Powder P30M	
Tool set SNUN120412 - Phase 1 (Small size)	
Area - pressed 250	
S Height 1.60	
SG 12.38	Volume 0.2547 cm ³
App. Density 3.100	Volatile 2.44%
Press height 2.00	
Weight 3.230	
Tool 25	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	P30 - 20	3.60	2.00	15.92	36.7	3.40	4.870	-0.160	B	1.63	13.24	3.51	
20.5	P30 - 205	3.57	2.00	15.91	33.3	3.56	4.816	-0.150	G	1.62	13.18	3.47	
21.0	P30 - 21	3.53	2.00	15.91	30.3	3.53	4.762	-0.140	G	1.62	13.13	3.44	
21.5	P30 - 225	3.49	2.00	15.91	26.5	3.50	4.721	-0.130	G	1.61	13.08	3.41	
22.0	P30 - 23	3.46	2.00	15.91	22.7	3.45	4.667	-0.120	G	1.60	13.04	3.37	
22.5	P30 - 235	3.42	1.99	15.91	20.3	3.42	4.613	-0.120	S	1.57	13.00	3.33	
23.0	P30 - 24	3.38	1.99	15.91	18.2	3.38	4.557	-0.120	S	1.58	12.95	3.29	
23.5	P30 - 245	3.35	2.00	15.91	15.4	3.35	4.519	-0.110	S	1.58	12.92	3.26	
24.0	P30 - 25	3.31	1.99	15.91	13.8	3.31	3.310	-0.110	S	1.58	12.86	3.22	

Powder P30M			
Tool set SNUN120412 - Phase 2 (Midsize)			
Area - pressed	250.1		
S Height	3.20		
SG	12.38	Volume	0.5093 cm ³
App. Density	3.100		Volatile
Press height	4.00		
Weight	6.305		
Tool	25		%

Powder details		Links
Date :		
Item no : 4001011		Powder sheet
Batch no : 33028		RTP
Co % : 10.0	%	Spray drying
Grain size : 2 - 3	μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	P30 - 20	7.03	4.00	15.91	25.2	7.03	7.462	-0.180	B	3.25	13.07	6.85	
20.5	P30 - 205	6.96	4.00	15.91	22.7	6.96	9.368	-0.170	C	3.24	13.04	6.77	
21.0	P30 - 21	6.89	3.99	15.91	20.7	6.90	9.288	-0.170	C	3.22	12.98	6.71	
21.5	P30 - 225	6.82	3.99	15.91	18.4	6.83	9.207	-0.160	C	3.21	12.95	6.66	
22.0	P30 - 23	6.75	3.99	15.91	15.5	6.74	9.099	-0.150	OK	3.20	12.88	6.57	
22.5	P30 - 235	6.68	4.00	15.91	13.4	6.69	9.018	-0.140	S	3.19	12.83	6.51	
23.0	P30 - 24	6.60	4.00	15.90	11.5	6.59	8.896	-0.130	S	3.18	12.78	6.41	
23.5	P30 - 245	6.53	4.00	15.90	9.6	6.53	8.802	-0.126	S	3.18	12.73	6.36	
24.0	P30 - 25	6.46	4.01	15.90	8.35	6.46	8.708	-0.110	S	3.18	12.68	6.29	

Powder	P30M
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	12.38
App. Density	3.100
Press height	8.00
Weight	12.919
Tool	25 %
	Volume 1.0187 cm ³
	Volatile 2.44%

Powder details	Links
Date :	
Item no : 4001011	Powder sheet
Batch no : 33028	RTP
Co % : 10.0 %	Spray drying
Grain size : 2 - 3 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent		Pressing (Green)										Sintered		
Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight		
21.0	P30 - 21	14.12	8.00	15.91	24.7	14.10	18.888	-0.260	5	6.52	13.05	13.75		
21.5	P30 - 215	13.97	8.00	15.91	22.4	13.97	18.657	-0.250	OK	6.51	13.01	13.62		
22.0	P30 - 22	13.83	8.00	15.91	20.1	13.84	18.573	-0.240	OK	6.49	12.99	13.48		
22.5	P30 - 225	13.68	8.00	15.91	17.6	13.67	18.299	-0.230	OK	6.45	12.94	13.34		
23.0	P30 - 23	13.53	8.00	15.91	15.2	13.51	18.072	-0.220	OK	6.42	12.87	13.16		
23.5	P30 - 235	13.38	8.00	15.90	13.2	13.38	17.884	-0.210	OK	6.41	12.82	13.02		
24.0	P30 - 24	13.23	8.00	15.90	11.7	13.21	17.604	-0.200	OK	6.38	12.77	12.87		
24.5	P30 - 245	13.07	8.00	15.90	10.4	13.10	17.431	-0.190	OK	6.38	12.73	12.76		
25.0	P30 - 25	12.92	8.00	15.90	9.2	12.91	17.178	-0.190	5	6.35	12.68	12.59		

A1.13.2 Grade P30M – Powder physical properties, SG and porosity reports

Table A1.61 – Historical screen fraction report for grade P30M (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
197.6 g	0.1 g	8.9 g	94.0 g	41.5 g	26.7 g	26.5 g

Table A1.62– Historical screen fraction report for grade P30M (mass percent)

>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
0.1 %	4.5 %	47.6 %	21.0 %	13.5 %	13.4 %

Table A1.63 – Apparent density report of grade P30M test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
72.8 g	25 cc	2.91 g/cm ³	3.09 g/cm ³

Table A1.64 – Sintered density report for grade P30M test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	13.7444	12.711	0.068	0.9974	18.8	12.45	12.49
2	6.7832	6.3094	0.068	0.9974	18.8	12.49	
3	3.5096	3.2980	0.068	0.9974	18.8	12.52	

Table A1.65 – Porosity report for best and worst sintered test pieces (midsize) for P30M

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
P30M	20 %	24 %	A00 B00	A00 B00	Very good	Good

A1.14 Experimental data sheets for grade 'P40M'

A1.14.1 'P40M' Grade pressing test sheets – negative inserts

Powder details		Links
Date :		Powder sheet
Item no : 4001012		RTP
Batch no : 79832		Spray drying
Co % : 10.0	%	
Grain size : 2 - 3	μ	
PRZ reclaim? :	%	
PEG content :	%	

Powder	P40M
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	13.25
Volume	0.2547 cm ³
App. Density	3.225
Volatile	2.28%
Press height	2.00
Weight	3.452
Tool	25
	%

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
	20.0	P40M - 215	3.85	2.00	15.92	42.0	3.85	4.783	-0.180	H	3.32	13.15	7.48
	20.5	P40M - 22	3.81	2.00	15.92	37.5	3.81	4.733	-0.170	H	3.31	13.12	7.43
	21.0	P40M - 225	3.77	2.00	15.92	33.1	3.76	4.671	-0.160	G	3.30	13.08	7.36
	21.5	P40M - 23	3.73	2.00	15.91	30.2	3.74	4.634	-0.150	G (B)	3.28	13.05	7.31
	22.0	P40M - 235	3.69	2.00	15.91	26.4	3.69	4.584	-0.140	G	3.27	13.01	7.21
	22.5	P40M - 24	3.65	2.00	15.91	22.9	3.65	4.534	-0.130	G OK	3.26	12.96	7.13
	23.0	P40M - 245	3.61	2.00	15.91	19.8	3.61	4.485	-0.120	S	3.23	12.89	7.04
	23.5	P40M - 25	3.57	2.00	15.91	17.2	3.57	4.435	-0.110	S	3.23	12.85	6.98
	24.0	P40M - 255	3.53	2.01	15.91	15.4	3.55	4.410	-0.100	S	3.21	12.79	6.88

Powder	P40M
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	13.25
App. Density	3.225
Press height	4.00
Weight	6.902
Tool	25
	%
	Volume 0.5093 cm ³
	Volatile 2.28%

Powder details	Links
Date :	
Item no : 4001012	<u>Powder sheet</u>
Batch no : 79832	<u>RTP</u>
Co % : 10.0	%
Grain size : 2 - 3	μ
PRZ reclaim? :	
PEG content : :	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	P40M - 215	7.70	4.00	15.91	35.5	7.67	9.600	-0.210	OK	3.32	13.15	7.48	
20.5	P40M - 22	7.62	4.00	15.91	32.3	7.62	9.500	-0.200	OK	3.31	13.12	7.43	
21.0	P40M - 225	7.54	3.99	15.91	30.4	7.54	9.401	-0.200	I	3.30	13.08	7.36	
21.5	P40M - 23	7.47	3.99	15.91	28.1	7.49	9.338	-0.190	OK	3.28	13.05	7.31	
22.0	P40M - 235	7.39	3.99	15.92	25.2	7.39	9.238	-0.190	OK	3.27	13.01	7.21	
22.5	P40M - 24	7.31	3.99	15.92	22.0	7.30	9.126	-0.180	OK	3.26	12.96	7.13	
23.0	P40M - 245	7.23	4.00	15.91	18.6	7.22	9.013	-0.170	OK	3.23	12.89	7.04	
23.5	P40M - 25	7.15	4.00	15.91	16.3	7.15	8.914	-0.160	OK	3.23	12.85	6.98	
24.0	P40M - 255	7.07	4.00	15.91	14.4	7.06	8.801	-0.150	S	3.21	12.79	6.88	

Powder	P40M
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	13.25
App. Density	3.225
Press height	8.00
Weight	13.806
Tool	25
	%
	Volume 1.0187 cm ³
	Volatile 2.28%

Powder details	Links
Date :	
Item no : 4001012	Powder sheet
Batch no : 79832	RTP
Co % : 10.0	% Spray drying
Grain size : 2 - 3	μ
PRZ reclaim? :	%
PEG content : :	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
21.5	P40M - 215	14.93	8.00	15.92	26.0	14.93	18.633	-0.260	B	6.58	13.01	14.59	
22.0	P40M - 22	14.78	8.00	15.91	23.4	14.78	18.445	-0.250	OK	6.57	12.97	14.44	
22.5	P40M - 225	14.62	8.00	15.91	20.6	14.60	18.221	-0.240	OK	6.53	12.92	14.25	
23.0	P40M - 23	14.46	8.00	15.91	18.7	14.49	18.059	-0.230	OK	6.52	12.88	14.14	
23.5	P40M - 235	14.30	8.00	15.91	16.3	14.30	17.859	-0.220	OK	6.47	12.82	13.96	
24.0	P40M - 24	14.13	8.00	15.91	13.9	14.12	17.634	-0.210	OK	6.44	12.77	13.79	
24.5	P40M - 245	13.97	8.00	15.91	12.2	13.95	17.409	-0.200	OK	6.43	12.73	13.62	
25.0	P40M - 25	13.81	8.01	15.90	11.3	13.83	17.235	-0.190	OK	6.41	12.69	13.52	
25.5	P40M - 255	13.64	7.99	15.90	9.8	13.66	17.047	-0.190	SS	6.40	12.63	13.34	

A1.14.2 Grade P40M – Powder physical properties, SG and porosity reports

Table A1.66 – Historical screen fraction report for grade P40M (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
196.3 g	0.1 g	9.5 g	94.2 g	46.4 g	28.1 g	17.9 g

Table A1.67 – Historical screen fraction report for grade P40M (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	4.8 %	48.0 %	23.6 %	14.3 %	9.1 %

Table A1.68 – Apparent density report of grade P40M test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
77.2 g	25 cc	3.09 g/cm ³	3.26 g/cm ³

Table A1.69 – Sintered density report for grade P40M test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	14.5934	13.5640	0.0681	0.9974	18.8	13.26	13.26
2	7.4335	6.9425	0.0681	0.9974	18.8	13.26	
3	3.6752	4.668	0.0681	0.9974	18.8	13.26	

Table A1.70 – Porosity report for best and worst sintered test pieces (midsize) for P40M

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
P40M	21 %	24 %	A00 B00	A00 B00	Very good	Very good

A1.15 Experimental data sheets for grade 'P511'

A1.15.1 'P511' Grade pressing test sheets – negative inserts

Powder details		Links
Date :	Item no : 4001013	<u>Powder sheet</u>
	Batch no : 93458	<u>RTP</u>
	Co % : 11.7	% <u>Spray drying</u>
	Grain size : 3 - 6	μ
	PRZ reclaim? :	%
	PEG content : :	%

Powder	P511
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	14.26
App. Density	Volume 0.2547 cm ³
Press height	2.00
Weight	3.725
Tool	25
	%

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
	20.0	P511 - 20	4.15	2.00	15.91	34.5	4.16	4.704	-0.150	OK	1.68	13.17	4.13
	20.5	P511 - 205	4.11	1.99	15.91	31.8	4.10	4.648	-0.150	OK	1.67	13.14	4.09
	21.0	P511 - 21	4.07	1.99	15.91	28.3	4.06	4.591	-0.140	OK	1.66	13.07	4.05
	21.5	P511 - 215	4.03	2.00	15.91	26.5	4.05	4.568	-0.130	OK	1.66	13.05	4.00
	22.0	P511 - 22	3.99	2.00	15.91	22.3	3.98	4.512	-0.120	OK	1.65	13.00	3.96
	22.5	P511 - 225	3.94	2.00	15.91	17.5	3.94	4.455	-0.110	OK	1.65	12.94	3.95
	23.0	P511 - 23	3.90	2.00	15.90	17.0	3.90	4.410	-0.100	OK	1.65	12.87	4.88
	23.5 195	P511 - 235	4.19 3.86	2.00	15.91	38.3	4.19	4.738	-0.160	OK	1.69 1.6869	13.22	4.84
	24.0 19	P511 - 24	4.24 3.81	2.00	15.91	42.4	4.24	4.744	-0.170	OK	1.70	13.27	3.80

Powder	P511
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	14.26
App. Density	Volume 0.5093 cm ³
Press height	4.00
Weight	7.449
Tool	25 %
	Volatile 2.57%

Powder details	Links
Date:	<u>Powder sheet</u>
Item no : 4001013	<u>RTP</u>
Batch no : 93458	<u>Spray drying</u>
Co % : 11.7 %	
Grain size : 3 - 6 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
	20.0	P511 - 20	8.31	3.99	15.91	30.8	8.31	9.454	-0.196	OK	3.35	13.15	8.11
	20.5	P511 - 205	8.23	3.99	15.91	28.5	8.23	9.363	-0.180	OK	3.33	13.11	8.01
	21.0	P511 - 21	8.14	3.99	15.91	25.5	8.13	9.249	-0.176	OK	3.32	13.05	7.93
	21.5	P511 - 215	8.06	4.00	15.91	22.7	8.07	9.170	-0.160	OK	3.31	13.01	7.87
	22.0	P511 - 22	7.97	4.00	15.91	20.9	7.97	9.067	-0.150	8	3.30	12.97	7.76
	22.5	P511 - 225	7.89	3.99	15.90	19.3	7.91	8.977	-0.150	OK	3.28	12.92	7.71
	23.0	P511 - 23	7.80	3.99	15.90	17.1	7.79	8.863	-0.150	OK	3.26	12.87	7.60
	23.5	P511 - 235	7.71	3.99	15.90	15.1	7.72	8.772	-0.148	OK	3.26	12.82	7.52
	24.0	P511 - 24	7.63	4.06	15.90	12.8	7.61	8.658	-0.130	OK	3.24	12.76	7.42
19.5			8.40	4.00	15.91	14.9	8.41	9.551	-0.200		3.37	13.20	8.20

Powder P511			
Tool set SNUN120412 - Phase 3 (Largesize)			
Area - pressed	250.1		
S Height	6.40		
SG	14.26	Volume	1.0187 cm ³
App. Density		Volatile	2.57%
Press height	8.00		
Weight	14.900		
Tool	25	%	

Powder details		Links
Date :		
Item no : 4001013		Powder sheet
Batch no : 93458		RTP
Co % : 11.7	%	Spray drying
Grain size : 3 - 6	μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
20.0		P511 - 20	16.62	8.00	15.90	29.6	16.62	18.876	-0.240	(B)	6.70	13.11	16.20
20.5		P511 - 205	16.45	8.00	15.90	27.0	16.46	18.695	-0.230	G	6.67	13.07	16.04
21.0		P511 - 21	16.29	8.00	15.90	24.8	16.30	18.524	-0.226	G	6.65	13.04	15.90
21.5		P511 - 215	16.12	8.00	15.90	22.4	16.11	18.319	-0.210	G	6.62	12.98	15.71
22.0		P511 - 22	15.95	8.00	15.90	20.3	15.95	18.126	-0.200	G	6.61	12.94	15.56
22.5		P511 - 225	15.78	8.00	15.90	18.5	15.77	17.888	-0.190	G	6.58	12.89	15.38
23.0		P511 - 23	15.60	7.99	15.90	16.5	15.59	17.672	-0.190	G	6.54	12.85	15.19
23.5		P511 - 235	15.43	8.00	15.90	14.6	15.41	17.457	-0.180	G	6.53	12.80	15.02
24.0		P511 - 24	15.25	7.99	15.90	13.3	15.25	17.253	-0.180	G	6.50	12.76	14.86

A1.15.2 Grade P511 – Powder physical properties, SG and porosity reports

Table A1.71 – Historical screen fraction report for grade P511 test sample (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
197.1 g	0.2 g	9.6 g	88.0 g	45.3 g	32.1 g	22.0 g

Table A1.72 – Historical screen fraction report for grade P511 (mass percent)

>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
0.1 %	4.9 %	44.6 %	23.0 %	16.3 %	11.2 %

Table A1.73 – Apparent density report of grade P511 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
85.2 g	25 cc	3.41 g/cm ³	3.53 g/cm ³

Table A1.74 – Sintered density report for grade P511 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.5793	14.5577	0.0683	0.9974	18.9	14.26	14.25
2	7.8684	7.3863	0.0683	0.9974	18.9	14.26	
3	3.8868	3.6828	0.0683	0.9974	18.9	14.24	

Table A1.75 – Porosity report for best and worst sintered test pieces (midsize) for P511

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
P511	22 %	24 %	A00 B00	A00 B00	Excellent	Excellent

A1.16 Experimental data sheets for grade 'PBF'

A1.16.1 'PBF' Grade pressing test sheets – negative inserts

Powder details	
Date :	Links
Item no : 4001016	<u>Powder sheet</u>
Batch no : 72954	<u>RTP</u>
Co % : 5.9	%
Grain size : 2 - 3	μ
PRZ reclaim? :	%
PEG content :	%

Powder	PBF
Tool set	SNUN120412 - Phase 1 (Small)
Area - pressed	250.1
S Height	1.60
SG	14.94
App. Density	3.945
Press height	2.00
Weight	3.887
Tool	25
	%
	Volume 0.2547 cm ³
	Volatile 2.14%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PBF-20	4.33	2.00	15.92	27.9	4.33	4.195	-0.140	SS	1.67	13.225	4.22	
20.5	PBF-205	4.29	2.00	15.92	24.9	4.30	4.166	-0.130	G	1.66	13.18	4.19	
21.0	PBF-21	4.25	2.00	15.92	21.7	4.25	4.127	-0.120	OK	1.64	13.135	4.15	
21.5	PBF-215	4.20	2.00	15.92	18.2	4.21	4.088	-0.110	OK	1.63	13.07	4.10	
22.0	PBF-22	4.16	2.00	15.92	15.2	4.16	4.049	-0.110	SS	1.62	13.025	4.05	
22.5	PBF-225	4.12	2.00	15.92	13.3	4.13	4.030	-0.100	SS	1.62	12.975	4.01	
23.0	PBF-23	4.07	2.00	15.92	11.6	4.06	3.971	-0.100	SS	1.62	12.925	3.95	
23.5	PBF-235	4.02	2.00	15.92	10.3	4.01	3.922	-0.100	SS	1.61	12.87	3.90	
24.0	PBF-24	3.98	1.99	15.92	10.1	3.98	3.883	-0.100	SS	1.61	12.855	3.88	

Powder	PBF
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	14.94
App. Density	3.945
Press height	4.00
Weight	7.772
Tool	25 %
	Volume 0.5093 cm ³
	Volatile 2.14%

Powder details	Links
Date :	
Item no : 4001016	<u>Powder sheet</u>
Batch no : 72954	<u>RTP</u>
Co % : 5.9 %	<u>Spray drying</u>
Grain size : 2 - 3 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PBF-20	8.67	4.00	15.91	25.0	8.66	8.552	-0.170	OK	3.31	13.175	8.49	
20.5	PBF-205	8.58	4.00	15.91	21.7	8.68	8.413	-0.160	B	3.30	13.135	8.39	
21.0	PBF-21	8.49	4.00	15.91	19.5	8.49	8.325	-0.150	OK	3.29 3.28	13.08 13.055	8.31	
21.5	PBF-215	8.41	3.99	15.91	17.5	8.42	8.256	-0.150	OK	3.28	13.055	8.23	
22.0	PBF-22	8.32	4.00	15.91	14.5	8.32	8.168	-0.140	OK	3.27	12.995	8.14	
22.5	PBF-225	8.23	4.00	15.91	12.8	8.23	8.080	-0.130	SS	3.26	12.94	8.04	
23.0	PBF-23	8.14	4.00	15.91	11.1	8.14	7.991	-0.120	S	3.25	12.89	7.96	
23.5	PBF-235	8.05	4.00	15.91	10.2	8.06	7.913	-0.120	S	3.22	12.85	7.88	
24.0	PBF-24	7.96	4.00	15.91	9.11	7.99	7.854 7.854	-0.120	S	3.22	12.81	7.81	

Powder	PBF
Tool set	SNUN120412 - Phase 3 (Large)
Area - pressed	248
S Height	6.40
SG	14.94
App. Density	3.945
Press height	8.00
Weight	15.545
Tool	25 %
	Volume 1.0187 cm ³
	Volatile 2.14%

Powder details	
Date :	Links
Item no : 4001016	Powder sheet
Batch no : 72954	RTP
Co % : 5.9 %	Spray drying
Grain size : 2 - 3 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PBF-20	17.34											
20.5	PBF-205	17.16	7.99	15.91	21.4	17.16	17.012	-0.240	OK	6.61	13.115	16.78	
21.0	PBF-21	16.99	8.02	15.91	17.9	16.98	16.775	-0.19	B	6.60	13.035	16.59	
21.5	PBF-215	16.81	8.00	15.91	16.2	16.81	16.547	-0.200	OK	6.58	13.00	16.43	
22.0	PBF-22	16.64	8.00	15.90	14.8	16.65	16.439	-0.210	OK	6.55	12.97	16.28	
22.5	PBF-225	16.46	7.99	15.90	12.7	16.46	16.232	-0.200	SS	6.53	12.91	16.08	
23.0	PBF-23	16.28	7.99	15.91	11.5	16.30	16.074	-0.200	S	6.51	12.88	15.93	
23.5	PBF-235	16.10	7.98	15.90	10.2	16.09	15.886	-0.200	S	6.48	12.825	15.72	
24.0	PBF-24	15.92	7.98	15.91	9.0	15.94	15.728	-0.190	S	6.47	12.78	15.58	

A1.16.2 Grade PBF – Powder physical properties, SG and porosity reports

Table A1.76 – Historical screen fraction report for grade PBF test (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
197.5 g	0.3 g	15.6 g	88.5 g	41.7 g	29.8 g	21.7 g

Table A1.77 – Historical screen fraction report for grade PBF (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	7.9 %	44.8 %	21.1 %	15.1 %	11.0 %

Table A1.78 – Apparent density report of grade PBF test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
96.5 g	25 cc	3.86 g/cm ³	3.70 g/cm ³

Table A1.79 – Sintered density report for grade PBF test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	16.7678	15.7175	0.0685	0.9968	21.7	14.94	14.93
2	8.4630	7.9673	0.0685	0.9968	21.7	14.95	
3	4.1973	3.9852	0.0685	0.9968	21.7	14.91	

Table A1.80 – Porosity report for best and worst sintered test pieces (midsize) for PBF

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
PBF	20.5 %	24.0 %	A00 B00	A00 B00	Very good	Very good

A1.17 Experimental data sheets for grade 'PBT'

A1.17.1 'PBT' Grade pressing test sheets – negative inserts

Powder details		Links
Date :	Item no : 4001015	<u>Powder sheet</u>
	Batch no : 86847	<u>RTP</u>
	Co % : 6.0	<u>Spray drying</u>
	Grain size : 2 - 4	μ
	PRZ reclaim? :	%
	PEG content :	%

Powder	PBT
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	14.96
Volume	0.2547 cm ³
App. Density	3.76
Volatile	2.20%
Press height	2.00
Weight	3.894
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PBT-20	4.34	2.00	15.90	29.6	4.35	4.691	-0.1	15	1.69	13.19	4.25	
20.5	PBT-205	4.30	2.01	15.90	23.0	4.30	4.637	-0.07	UG	1.69	13.09	4.20	
21.0	PBT-21	4.26	2.00	15.90	20.2	4.26	4.594	-0.07	UG	1.68	13.07	4.17	
21.5	PBT-215	4.21	2.00	15.89	18.0	4.21	4.529	-0.07	SS	1.68	13.06	4.11	
22.0	PBT-22	4.17	2.00	15.89	15.9	4.17	4.486	-0.07	SS	1.67	12.99	4.06	
22.5	PBT-225	4.12	1.99	15.89	14.7	4.13	4.443	-0.07	SS	1.70	12.975	4.04	
23.0	PBT-23	4.08	1.99	15.89	12.0	4.07	4.389	-0.07	SS	1.65	12.945	3.98	
23.5	PBT-235	4.03	1.986	15.90	12.8	4.03	4.324	-0.10	SS	1.65	12.925	3.94	
24.0	PBT-24	3.99	2.01	15.90	9.0	4.00	4.292	-0.05	SS	1.65	12.845	3.91	

Powder	PBT
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250
S Height	3.20
SG	14.96
	Volume 0.5093 cm ³
App. Density	3.76
	Volatile 2.20%
Press height	4.00
Weight	7.787
Tool	25 %

Powder details		Links
Date:	3/2/2006	
Item no :	4001015	Powder sheet
Batch no :	86847	RTP
Co % :	6.0 %	Spray drying
Grain size :	2 - 4 μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PBT-20	8.68	4.00	15.90	28.5	8.68	9.481	-0.14	B	3.35	13.16	8.51	
20.5	PBT-205	8.60	4.00	15.90	24.9	8.59	9.382	-0.13	G	3.34	13.12	8.42	
21.0	PBT-21	8.51	4.00	15.90	22.2	8.51	9.284	-0.13	G	3.32	13.085	8.34	
21.5	PBT-215	8.42	4.00	15.90 14.9	19.9	8.41	9.175	-0.13	SS	3.31	13.045	8.25	
22.0	PBT-22	8.33	4.00	15.90	17.3	8.33	9.099	-0.12	SS	3.30	13.00	8.16	
22.5	PBT-225	8.24	4.00	15.89	13.3	8.26	9.001	-0.09	SS	3.29	12.925	8.07	
23.0	PBT-23	8.15	4.00	15.90	11.6	8.15	8.902	-0.09	SS	3.27	12.875	7.97	
23.5	PBT-235	8.06	4.00	15.89	10.8	8.09	8.826	-0.09	SS	3.26	12.855	7.92	
24.0	PBT-24	7.97	3.99	15.89	9.1	7.99	8.717	-0.09	SS	3.25	12.815	7.81	

Powder	PBT
Tool set	SNUN120412 - Phase 3 (Midsize)
Area - pressed	250
S Height	6.40
SG	14.96
App. Density	3.76
Press height	8.00
Weight	15.575
Tool	25
	%
	Volume 1.0187 cm ³
	Volatile 2.20%

Powder details	Links
Date :	
Item no : 4001015	<u>Powder sheet</u>
Batch no : 86847	<u>RTP</u>
Co % : 6.0	% <u>Spray drying</u>
Grain size : 2 - 4	μ
PRZ reclaim? :	
PEG content :	%

Equivalent	Pressing (Green)										Sintered		
	Tool %	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
20.0		PBT-20	17.37	8.01	15.88	26.8	17.38	17.370	-0.20	B/S	6.69	13.13	16.99
20.5		PBT-205	17.20	8.00	15.88	24.2	17.19	17.959	-0.20	V/S U/L	6.67	13.09	16.82
21.0		PBT-21	17.02	7.98	15.88	21.6	17.02	17.782	-0.20	V/S U/L	6.62	13.06	16.64
21.5		PBT-215	16.85	7.99	15.88	19.3	16.86	17.604	-0.18	S/S	6.61	13.01	16.48
22.0		PBT-22	16.67	8.00	15.89	17.3	16.69	17.406	-0.18	S/S	6.58	12.98	16.32
22.5		PBT-225	16.49	8.00	15.90	14.4	16.48	17.986	-0.18	S/S	6.56	12.935	16.15
23.0		PBT-23	16.31	8.02	15.90	12.0	16.32	17.768	-0.15	S/S	6.54	12.88	15.99
23.5		PBT-235	16.13	8.01	15.89	10.50	16.13	17.702	-0.14	S/S	6.51	12.835	15.76
24.0		PBT-24	15.95	8.00	15.89	9.40	15.95	17.504	-0.14	S/S	6.50	12.79	15.65

A1.17.2 Grade PBT – Powder physical properties, SG and porosity reports

Table A1.81 – Screen fraction report for grade PBT test sample (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
196.8 g	0.2 g	11.6 g	93.2 g	44.6 g	27.9 g	19.3 g

Table A1.82 – Screen fraction report for grade PBT (mass percent of previous table)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	5.9 %	47.4 %	22.6 %	14.2 %	9.8 %

Table A1.83 – Apparent density report of grade PBT test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
97.0 g	25 cc	3.88 g/cm ³	3.84 g/cm ³

Table A1.84 – Sintered density report for grade PBT test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	16.8208	15.7667	0.0681	0.9974	19.2	14.95	14.98
2	7.9802	7.5173	0.0681	0.9974	19.2	14.99	
3	4.2544	4.0394	0.0681	0.9974	19.2	14.99	

Table A1.85 – Porosity report for best and worst sintered test pieces (midsize) for PBT

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
PBT	20 %	24 %	A02 B02	A02 B02	Acceptable	Acceptable

A1.18 Experimental data sheets for grade 'PC20'

A1.18.1 'PC20' Grade pressing test sheets – negative inserts

Powder details		Links
Date :	Item no : 4001007	<u>Powder sheet</u>
	Batch no : 6977	<u>RTP</u>
	Co % : 6.0	<u>Spray drying</u>
	Grain size : 1 - 3	μ
	PRZ reclaim? :	%
	PEG content :	%

Powder	PC20
Tool set	SNUN120412 - Phase 1 (Small)
Area - pressed	250
S Height	1.60
SG	14.66
Volume	0.2547 cm ³
App. Density	3.705
Volatile	2.43%
Press height	2.00
Weight	3.825
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0	PC20-19	4.35	2.00	15.91	61.6	4.36	4.771	-0.19	f1	1.73	13.33	4.25	
19.5	PC20-195	4.31	2.00	15.91	54.0	4.30	4.716	-0.17	f1	1.70	13.28	4.20	
20.0	PC20-20	4.27	2.00	15.92	47.5	4.27	4.672	-0.150	oh	1.68	13.235	4.17	
* 20.5	PC20-205	4.22	2.01	15.91	38.7	4.22	4.618	-0.120	oh	1.66	13.145	4.11	
21.0	PC20-21	4.18	2.00	15.91	34.1	4.17	4.563	-0.110	B	1.67	13.10	4.06	
21.5	PC20-215	4.14	2.00	15.91	30.4	4.14	4.519	-0.100	oh	1.67	13.085	4.03	
22.0	PC20-22	4.09	2.00	15.91	27.1	4.08	4.454	-0.100	oh	1.65	13.04	3.98	
22.5	PC20-225	4.05	1.99	15.91	26.68	4.06	4.410	-0.100	ss	1.64	13.04	3.95	
23.0	PC20-23	4.01	1.99	15.90	21.3	4.00	4.356	-0.100	ss	1.63	12.98	3.90	

Powder	PC20
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250
S Height	3.20
SG	14.66
App. Density	3.705
Press height	4.00
Weight	7.648
Volume	0.5093 cm ³
Volatile	2.43%
Tool	25 %

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0	PC20-19	8.70	4.00	15.90	63.2	8.71	9.528	-0.27	H	3.36	13.32	8.49	
19.5	PC20-195	8.61	4.00	15.90	54.7	8.62	9.441	-0.25	H	3.34	13.275	8.41	
20.0	PC20-20	8.53	4.00	15.90	49.7	8.53	9.353	-0.23	OK	3.35	13.23	8.32	
20.5	PC20-205	8.44	4.00	15.89	42.2	8.43	9.243	-0.21	OK	3.32	13.16	8.22	
21.0	PC20-21	8.36	4.00	15.90	35.9	8.36	9.156	-0.19	B	3.32	13.135	8.14	
21.5	PC20-215	8.27	4.00	15.89	31.3	8.27	9.057	-0.17	OK	3.30	13.09	8.05	
22.0	PC20-22	8.19	4.01	15.90	26.9	8.20	8.981	-0.14	OK	3.29	13.035	7.97	
22.5	PC20-225	8.10	4.00	15.89	22.4	8.10	8.882	-0.120	SS	3.29	12.98	7.90	
23.0	PC20-23	8.01	4.00	15.89	19.5	8.01	8.783	-0.120	SS	3.27	12.95	7.80	

Powder	PC20
Tool set	SNUN120412 - Phase 3 (Large)
Area - pressed	250
S Height	6.40
SG	14.66
Volume	1.0187 cm ³
App. Density	3.705
Volatile	2.43%
Press height	8.00
Weight	15.297
Tool	25 %

Powder details	Links
Date :	
Item no : 4001007	<u>Powder sheet</u>
Batch no : 6977	<u>RTP</u>
Co % : 6.0 %	<u>Spray drying</u>
Grain size : 1 - 3 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0	PC20ii-20	17.40	7.99	15.91	67.5	17.40	18.964	-0.43	H	6.68	13.30	17.00	
19.5	PC20ii-205	17.23	7.99	15.90	58.5	17.22	18.768	-0.40	H	6.65	13.265	16.82	
20.0	PC20ii-21	17.06	7.99	15.90	51.8	17.07	18.593	-0.37	Oh	6.64	13.225	16.67	
20.5	PC20ii-215	16.89	8.00	15.90	44.5	16.90	18.419	-0.34	Oh	6.62	13.165	16.51	
21.0	PC20ii-22	16.72	8.00	15.90	36.5	16.70	18.212	-0.31	B	6.60	13.12	16.30	
21.5	PC20ii-225	16.55	8.00	15.90	32.5	16.56	18.038	-0.28	Ok	6.58	13.07	16.17	
22.0	PC20ii-23	16.37	8.01	15.90	26.7	16.37	17.895	-0.24	SS	6.57	13.035	16.01	
22.5	PC20ii-235	16.20	8.00	15.89	23.20	16.18	17.688	-0.22	SS	6.54	12.955	15.81	
23.0	PC20ii-24	16.02	8.01	15.90	19.8	16.02	17.491	-0.20	SS	6.52	12.925	15.63	

A1.18.2 Grade PC20 – Powder physical properties, SG and porosity reports

Table A1.86 – Historical screen fraction report for grade PC20 (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
148.4 g	0.1 g	3.0 g	71.6 g	37.4 g	22.1 g	14.2 g

Table A1.87 – Historical screen fraction report for grade PC20 (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	2.0 %	48.3 %	25.2 %	14.9 %	9.6 %

Table A1.88 – Apparent density report of grade PC20 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
88.7 g	25 cc	3.55 g/cm ³	3.71 g/cm ³

Table A1.89 – Sintered density report for grade PC20 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	16.3068	15.2596	0.0683	0.9974	18.9	14.58	14.56
2	8.2236	7.7292	0.0683	0.9974	18.9	14.58	
3	4.0648	3.8539	0.0683	0.9974	18.9	14.52	

Table A1.90 – Porosity report for best and worst sintered test pieces (midsize) for PC20

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
PC20	21 %	23 %	A00 B00	A00 B00	Good	Good

A1.19.1 'PI-15' Grade pressing test sheets – negative inserts

A1.19.1 'PI-15' Grade pressing test sheets – negative inserts

[illegible]

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PI15-2	4.08	2.01	15.92	40.7	4.08	4.648	-0.160	H	1.68	13.21	3.99	
20.5	PI15-205	4.04	2.00	15.92	35.0	4.03	4.591	-0.150	H	1.67	13.15	3.94	
21.0	PI15-21	4.00	2.01	15.92	30.8	4.01	4.557	-0.130	S	1.65	13.12	3.91	
21.5	PI15-215	3.95	2.00	15.91	26.9	3.94	4.489	-0.130	OK	1.64	13.065	3.85	
22.0	PI15-22	3.91	2.00	15.91	23.9	3.90	4.432	-0.130	OK	1.63	13.025	3.81	
22.5	PI15-225	3.87	1.99	15.91	21.3	3.87	4.387	-0.130	OK	1.62	13.00	3.77	
23.0	PI15-23	3.83	1.99	15.92	19.1	3.83	4.342	-0.100	OK	1.62	12.96	3.73 ⁽³⁾	
23.5	PI15-235	3.79	2.00	15.91	16.8	3.80	4.308	-0.120	OK	1.62	12.925	3.70	
24.0	PI15-24	3.74	1.99	15.91	14.3	3.74	4.251	-0.120	SS	1.60	12.87	3.65	

Powder	PI-15
Tool set	SNUN120412 - Phase 2 (Midsized)
Area - pressed	250
S Height	3.20
SG	14.02
App. Density	3.46
Press height	4.00
Weight	7.311
Tool	25 %
Volume	0.5093 cm ³
Volatiles	2.39%

Powder details	Links
Date :	<u>Powder sheet</u>
Item no : 4001018	<u>RTP</u>
Batch no : 91483	<u>Spray drying</u>
Co % : 6.0 %	
Grain size : 2 - 3 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PI15-20	8.15	4.00	15.91	36.6	8.15	9.346	-0.220	H	3.22	13.195	7.96	
20.5	PI15-205	8.07	4.01	15.91	32.6	8.07	9.254	-0.190	H	3.30	13.135	7.88	
21.0	PI15-21	7.99	4.00	15.91	28.6	7.98	9.151	-0.180	B	3.29	13.10	7.80	
21.5	PI15-215	7.91	4.00	15.91	25.7	7.91	9.060	-0.170	OK	3.28	13.055	7.73	
22.0	PI15-22	7.82	4.00	15.91	22.1	7.81	8.945	-0.160	OK	3.26	13.00	7.67	
22.5	PI15-225	7.74	3.99	15.91	20.3	7.74	8.854	-0.160	OK	3.26	12.975	7.56	
23.0	PI15-23	7.66	4.00	15.91	17.5	7.66	8.762	-0.150	OK	3.25	12.915	7.48	
23.5	PI15-235	7.57	4.00	15.91	14.3	7.56	8.648	-0.140	OK	3.24	12.86	7.39	
24.0	PI15-24	7.49	4.00	15.91	12.8	7.49	8.556	-0.130	SS	3.22	12.82	7.31	

Powder	PI-15
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250
S Height	6.40
SG	14.02
App. Density	3.46
Press height	8.00
Weight	14.624
Tool	25
	%
	Volume 1.0187 cm ³
	Volatile 2.39%

Powder details	Links
Date :	
Item no : 4001018	<u>Powder sheet</u>
Batch no : 91483	<u>RTP</u>
Co % : 6.0	% <u>Spray drying</u>
Grain size : 2 - 3	μ
PRZ reclaim? :	
PEG content :	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PI15-20	16.31	8.00	15.91	37.8	16.32	18.646	-0.310	L	6.61	3.17	15.96	
20.5	PI15-205	16.15	8.00	15.91	32.6	16.16	18.430	-0.290	H	6.60	13.115	15.79	
21.0	PI15-21	15.98	8.00	15.91	28.5	15.96	18.203	-0.270	(B)	6.58	13.065	15.61	
21.5	PI15-215	15.82	8.01	15.91	24.6	15.81	18.019	-0.240	OK	6.55	13.02	15.45	
22.0	PI15-22	15.65	8.00	15.91	22.1	15.67	17.848	-0.230	OK	6.53	12.99	15.30	
22.5	PI15-225	15.48	8.00	15.91	19.3	15.49	17.666	-0.220	OK	6.50	12.93	15.14	
23.0	PI15-23	15.31	8.00	15.91	17.1	15.32	17.483	-0.210	OK	6.48	12.885	14.97	
23.5	PI15-235	15.14	8.00	15.91	14.9	15.16	17.312	-0.200	OK	6.43	12.79	14.82	
24.0	PI15-24	14.97	8.00	15.91	12.7	14.97	17.118	-0.190	SS	6.43	12.79	14.63	

A1.19.2 Grade PI-15 – Powder physical properties, SG and porosity reports

Table A1.91 – Historical screen fraction report for grade PI-15 test sample (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
196.1 g	0.1 g	7.3 g	94.4 g	49.5 g	27.1 g	17.7 g

Table A1.92 – Historical screen fraction report for grade PI-15 (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.04 %	3.72 %	48.1 %	25.2 %	13.8 %	9.0 %

Table A1.93 – Apparent density report of grade PI-15 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
84.9 g	25 cc	3.40 g/cm ³	3.52 g/cm ³

Table A1.94 – Sintered density report for grade PI-15 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.6136	14.5756	0.0685	0.9974	18.7	14.07	14.08
2	7.8866	7.3972	0.0685	0.9974	18.7	14.10	
3	3.9169	3.7077	0.0685	0.9974	18.7	14.08	

Table A1.95 – Porosity report for best and worst sintered test pieces (midsize) for PI-15

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
PI-15	21 %	23 %	A00 B00	A00 B00	Very good	Very good

A1.20.1 'PI-25' Grade pressing test sheets – negative inserts

Powder	PI-25
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	14.11
App. Density	3.475
Press height	2.00
Weight	3.666
Tool	25 %
	Volume 0.2547 cm ³
	Volatile 2.01%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PI25-20	4.09	1.99	15.92	45.5	4.08	4.60	-0.190	OK	1.67	13.28	3.99	
20.5	PI25-205	4.05	2.00	15.92	39.3	4.05	4.614	-0.170	OK	1.65	13.23	3.95	
21.0	PI25-21	4.01	2.00	15.92	34.0	4.01	4.568	-0.150	(B)	1.65 1.64	13.14 13.14	3.91	
21.5	PI25-215	3.97	2.00	15.92	29.6	3.96	4.511	-0.140	OK	1.64	13.14	3.87	
22.0	PI25-21	3.92	2.00	15.91	25.9	3.93	4.466	-0.130	OK	1.63	13.07	3.82	
22.5	PI25-225	3.88	2.00	15.91	21.9	3.87	4.409	-0.120	OK	1.64	13.015	3.78	
23.0	PI25-23	3.84	2.00	15.91	17.6	3.83	4.352	-0.110	SS	1.62	12.94	3.74	
23.5	PI25-235	3.80	2.00	15.91	15.1	3.80	4.307	-0.100	S	1.61	12.90	3.69	
24.0	PI25-24	3.75	2.00	15.91	12.5	3.74	4.239	-0.100	S	1.61	12.86	3.63	

Powder	PI-25
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	14.11
App. Density	3.475
Press height	4.00
Weight	7.331
Tool	25
	%
	Volume 0.5093 cm ³
	Volatile 2.01%

Powder details	Links
Date :	
Item no : 4001019	Powder sheet
Batch no : 87701	RTP
Co % : 6.6	% Spray drying
Grain size : 2 - 3	μ
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PI25-20	8.18	4.00	15.92	39.2	8.18	9.345	-0.220	OK	3.32	13.215	7.98	
20.5	PI25-205	8.09	4.00	15.92	33.7	8.10	9.253	-0.200	OK	3.31	13.18	7.90	
21.0	PI25-21	8.01	4.01	15.92	27.7	8.00	9.150	-0.170	B	3.29	13.11	7.82	
21.5	PI25-215	7.93	4.00	15.92	24.7	7.92	9.048	-0.160	OK	3.28	13.05	7.72	
22.0	PI25-21	7.85	4.00 4.05	15.91	22.5	7.86	8.968	-0.150	OK	3.27	13.025	7.68	
22.5	PI25-225	7.76	4.00	15.90	19.7	7.77	8.876	-0.140	OK	3.25	12.98	7.59	
23.0	PI25-23	7.68	4.00	15.90	16.6	7.66	8.762	-0.130	SS	3.24	12.91	7.48	
23.5	PI25-235	7.59	4.00	15.90	14.4	7.59	8.659	-0.130	S	3.24	12.88	7.42	
24.0	PI25-24	7.51	4.00	15.90	12.9	7.51	8.568	-0.130	S	3.22	12.815	7.33	

Powder	PI-25
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	14.11
App. Density	3.475
Press height	8.00
Weight	14.663
Tool	25
	%
	Volume 1.0187 cm ³
	Volatile 2.01%

Powder details	
Date : 16.02.2006	Links
Item no : 4001019	Powder sheet
Batch no : 87701	RTP
Co % : 6.6	%
Grain size : 2 - 3	μ
PRZ reclaim? :	%
PEG content :	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	PI25-20	16.35	8.01	15.91	36.7	16.37	18.671	-0.300	OK	6.62	13.18	15.98	
20.5	PI25-205	16.19	8.00	15.91	32.1	16.21	18.511	-0.280	OK	6.605	13.13	15.82	
21.0	PI25-21	16.03	8.01	15.91	27.6	16.02	18.316	-0.250	OK	6.58	13.055	15.64	
21.5	PI25-215	15.86	8.00	15.91	24.7	15.86	18.122	-0.240	OK	6.55	13.03	15.49	
22.0	PI25-21	15.69	8.00	15.90	21.8	15.68	17.917	-0.230	OK	6.53	12.995	15.32	
22.5	PI25-225	15.53	8.00	15.90	19.1	15.52	17.722	-0.220	OK	6.51	12.94	15.15	
23.0	PI25-23	15.36	8.00	15.90	17.0	15.37	17.540	-0.210	OK	6.48	12.90	15.01	
23.5	PI25-235	15.18	8.00	15.90	14.0	15.16	17.300	-0.200	OK	6.47	12.845	14.81	
24.0	PI25-24	15.01	7.99	15.90	12.7	14.99	17.083	-0.200	OK	6.43	12.81	14.64	

A1.20.2 Grade PI-25 – Powder physical properties, SG and porosity reports

Table A1.96 – Historical screen fraction report for grade PI25 (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
197.0 g	0.1 g	4.8 g	89.7 g	51.0 g	31.7 g	19.7 g

Table A1.97 – Historical screen fraction report for grade PI25 (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.1 %	2.4 %	45.6 %	25.9 %	16.1 %	10.0 %

Table A1.98 – Apparent density report of grade PI25 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
84.3 g	25 cc	3.37 g/cm ³	3.53 g/cm ³

Table A1.99 – Sintered density report for grade PI25 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.8247	14.7716	0.0686	0.9981	15.3	14.08	14.08
2	9.9022	7.4115	0.0686	0.9981	15.3	14.10	
3	3.9510	3.9393	0.0686	0.9981	15.3	14.07	

Table A1.100 – Porosity report for best and worst sintered test pieces (midsize) for PI25

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
PI25	21 %	24 %	A00 B00	A00 B00	Very good	Very good

A1.21 Experimental data sheets for grade 'PI-40'

A1.21.1 'PI-40' Grade pressing test sheets – negative inserts

Powder details	
Date :	Links
Item no : 4001020	Powder sheet
Batch no : 87702	RTP
Co % : 8.5	%
Grain size : 2 - 4	μ
PRZ reclaim? :	%
PEG content :	%

Powder	PI-40
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	13.80
Volume	0.2547 cm ³
App. Density	3.300
Volatile	2.48%
Press height	2.00
Weight	3.602
Tool	25
	%

Equivalent Tool %	Pressing (Green)								Sintered			
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
21.0	PI40 - 21	3.94	2.00	15.92	35.6	3.94	4.597	-0.150	OK	1.62	13.14	3.84
21.5	PI40 - 215	3.90	2.00	15.92	32.0	3.91	4.582	-0.140	OK	1.61	13.12	3.81
22.0	PI40 - 22	3.86	2.00	15.92	27.4	3.85	4.503	-0.130	B	1.61	13.07	3.76
22.5	PI40 - 225	3.81	2.00	15.92	24.1	3.82	4.456	-0.120	OK	1.60	13.05	3.71
23.0	PI40 - 23	3.77	2.00	15.92	20.2	3.77	4.416	-0.110	OK	1.60	12.98	3.67
23.5	PI40 - 235	3.73	1.99	15.92	17.9	3.73	4.363	-0.110	OK	1.59	12.94	3.63
24.0	PI40 - 24	3.69	2.00	15.92	16.2	3.71	4.339	-0.100	OK	1.58	12.90	3.61
24.5	PI40 - 245	3.65										
25.0	PI40 - 25	3.60										
20.8		3.98	2.00	15.92	40.0	3.98	4.663	-0.160	OK	1.63	13.20	3.88
20.0		4.03	2.00	15.92	45.4	4.03	4.702	-0.170	OK	1.64	13.21	3.96

Powder	PI-40
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	13.80
App. Density	3.300
Press height	4.00
Weight	7.203
Tool	25
	%
	Volume 0.5093 cm ³
	Volatile 2.48%

Powder details	Links
Date :	
Item no : 4001020	<u>Powder sheet</u>
Batch no : 87702	<u>RTP</u>
Co % : 8.5	<u>Spray drying</u>
Grain size : 2 - 4	μ
PRZ reclaim? :	
PEG content :	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
21.0	PI40 - 21	7.87	4.00	15.92	32.4	7.88	9.317	-0.200	OK	3.29	13.165	7.68	
21.5	PI40 - 215	7.79	4.00	15.92	28.0	7.77	9.199	-0.190	OK	3.26	13.09	7.59	
22.0	PI40 - 22	7.71	4.00	15.91	25.7	7.73	9.128	-0.180	B	3.25	13.07	7.54	
22.5	PI40 - 225	7.63	4.00	15.91	22.2	7.62	9.022	-0.170	OK	3.24	13.02	7.44	
23.0	PI40 - 23	7.54	4.00	15.91	19.0	7.54	8.915	-0.160	OK	3.23	12.98	7.35	
23.5	PI40 - 235	7.46	4.00	15.91	17.0	7.48	8.844	-0.150	OK	3.22	12.94	7.29	
24.0	PI40 - 24	7.37	4.00	15.91	13.5	7.36	8.726	-0.140	OK	3.21	12.865	7.18	
24.5	PI40 - 245	7.29	3.99	15.91	12.5	7.29	8.631	-0.140	SS	3.19	12.825	7.11	
25.0	PI40 - 25	7.20	4.00	15.90	10.7	7.20	8.524	-0.130	S	3.18	12.78	7.03	

A1.21.2 Grade PI-40 – Powder physical properties, SG and porosity reports

Table A1.101 – Historical screen fraction report for grade PI-40 (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
195.9 g	0.1 g	6.5 g	97.2 g	49.5 g	25.8 g	17.0 g

Table A1.102 – Historical screen fraction report for grade PI-40 (mass percent)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.04 %	3.3 %	49.6 %	25.2 %	13.2 %	8.7 %

Table A1.103 – Apparent density report of grade test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
81.7 g	25 cc	3.27 g/cm ³	3.40 g/cm ³

Table A1.104 – Sintered density report for grade PI-40 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	15.0413	14.0212	0.0685	0.9974	18.7	13.78	13.81
2	7.5870	7.1075	0.0685	0.9974	18.7	13.81	
3	3.7583	3.5557	0.0685	0.9974	18.7	13.83	

Table A1.105 – Porosity report for best and worst sintered test pieces (midsize) for PI-40

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
PI40	22 %	25 %	A00 B00	A00 B00	Very good	good

A1.22 Experimental data sheets for grade 'Q3'

A1.22.1 'Q3' Grade pressing test sheets – negative inserts

Powder details		Links
Date :		<u>Powder sheet</u>
Item no : 4001021		<u>RTP</u>
Batch no : 85857		<u>Spray drying</u>
Co % : 23.0	%	
Grain size : 3 - 6	μ	
PRZ reclaim? :		
PEG content :	%	

Powder Q3	
Tool set	SNUN120412 - Phase 2 (Midsized)
Area - pressed	250.1
S Height	3.20
SG	13.18
Volume	0.5093 cm ³
App. Density	3.035
Volatile	2.12%
Press height	4.00
Weight	6.855
Tool	25
	%

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	Q3 - 20	7.65	4.00	15.93	37.0	7.65	9.603	-0.240	A	3.31	13.15	7.48	
20.5	Q3 - 205	7.57	4.00	15.93	34.0	7.57	9.490	-0.230		3.30	13.12	7.41	
21.0	Q3 - 21	7.49	4.00	15.93	31.5	7.51	9.390	-0.220	OK	3.30	13.08	7.35	
21.5	Q3 - 215	7.41	4.00	15.93	28.0	7.40	9.277	-0.210	J	3.28	13.03	7.25	
22.0	Q3 - 22	7.34	4.00	15.93	25.9	7.34	9.189	-0.200		3.27	12.99	7.19	
22.5	Q3 - 225	7.26	4.00	15.93	23.4	7.26	9.077	-0.190	5	3.26	12.92	7.09	
23.0	Q3 - 23	7.18	4.00	15.93	21.5	7.20	8.977	-0.180	OK	3.24	12.89	7.05	
23.5	Q3 - 235	7.10	4.00	15.92	18.5	7.08	8.852	-0.170		3.23	12.81	6.93	
24.0	Q3 - 24	7.02	4.00	15.92	16.9	7.02	8.752	-0.170	J	3.21	12.77	6.87	

Powder Q3	
Tool set	SNUN120412 - Phase 3 (Largesize)
Area - pressed	250.1
S Height	6.40
SG	13.18
App. Density	3.035
Press height	8.00
Weight	13.711
Tool	25
	%
	Volume 1.0187 cm ³
	Volatile 2.12%

Powder details	
Date :	Links
Item no : 4001021	Powder sheet
Batch no : 85857	RTP
Co % : 23.0	%
Grain size : 3 - 6	μ
PRZ reclaim? :	
PEG content :	%

Equivalent Tool %	Pressing (Green)										Sintered	
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight
20.0	Q3 - 20	15.29	8.00	15.93	35.2	15.27	19.282	-0.330		6.63 6.68	13.13 13.27	14.95
20.5	Q3 - 205	15.14	8.00	15.93	32.6	15.15	19.105	-0.320		6.61 6.65	13.09 13.24	14.83
21.0	Q3 - 21	14.99	8.00	15.93	29.6	15.00	18.929	-0.310	ok	6.56 6.64	13.05 13.19	14.68
21.5	Q3 - 215	14.83	8.00	15.93	27.5	14.84	18.664	-0.300		6.55	13.06	14.52
22.0	Q3 - 22	14.67	8.00	15.92	24.7	14.67	18.513	-0.290		6.52	12.94	14.37
22.5	Q3 - 225	14.52	8.00	15.92	23.0	14.52	18.324	-0.280	(B)	6.52	12.91	14.22
23.0	Q3 - 23	14.36	8.00	15.92	20.6	14.35	18.072	-0.280		6.51	12.85	14.05
23.5	Q3 - 235	14.20	7.99	15.92	19.2	14.22	17.825	-0.270	ok	6.47	12.81	13.92
24.0	Q3 - 24	14.04	7.99	15.92	16.9	14.03	17.619	-0.260		6.44	12.75	13.74

A1.22.2 Grade Q3 – Powder physical properties, SG and porosity reports

Table A1.106 – Screen fraction report for grade Q3 test sample (granule sizes)

Total	>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
196.7 g	0.2 g	16.0 g	98.8 g	41.6 g	22.5 g	17.6 g

Table A1.107 – Screen fraction report for grade Q3 (mass percent of previous table)

>300 µm	>212 µm	>125 µm	>90 µm	>63 µm	>0 µm
0.1 %	8.1 %	50.2 %	21.1 %	11.5 %	9.0 %

Table A1.108 – Apparent density report of grade Q3 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
78.2 g	25 cc	3.13 g/cm ³	3.10 g/cm ³

Table A1.109 – Sintered density report for grade Q3 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	14.3796	13.3659	0.0681	0.9974	19.2	13.26	13.26
2	7.1962	6.7228	0.0681	0.9974	19.2	13.25	
3	7.2521	6.7748	0.0681	0.9974	19.2	13.26	

Table A1.110 – Porosity report for best and worst sintered test pieces (midsize) for Q3

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
Q3	A00 B00	A00 B00	22.5 %	24 %	Very good	Good

A1.23.1 'S9' Grade pressing test sheets – negative inserts

A1.23.1 'S9' Grade pressing test sheets – negative inserts

Powder	S9
Tool set	SNUN120412 - Phase 1 (Small size)
Area - pressed	250
S Height	1.60
SG	12.38
Volume	0.2547 cm ³
App. Density	
Press height	2.00
Weight	3.225
Volatile	2.29%
Tool	25
	%

[illegible]

Powder S9	
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	12.38
App. Density	Volume 0.5093 cm ³
Press height	4.00
Weight	6.450
Tool	25 %
	Volatile 2.29%

Powder details	
Date :	Links
Item no : 4001026	Powder sheet
Batch no : 89311	RTP
Co % : 10.0 %	Spray drying
Grain size : 2 - 3 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
19.0	S9 - 20	7.33	4.01	15.91	31.8	7.33	8.727	-0.190	B	3.33	13.24	7.16	
19.5	S9 - 205	7.26	4.00	15.91	28.9	7.25	8.622	-0.190	OK	3.31	13.22	7.08	
20.0	S9 - 21	7.19	4.00	15.91	26.2	7.18	8.537	-0.180	OK	3.30	13.17	7.01	
20.5	S9 - 215	7.12	4.00	15.91	23.0	7.11	8.442	-0.170	OK	3.29	13.13	6.94	
21.0	S9 - 22	7.05	3.99	15.91	20.2	7.05	8.359	-0.170	OK	3.27	13.09	6.87	
21.5	S9 - 225	6.98	3.99	15.91	19.7	6.99	8.288	-0.170	OK	3.27	13.05	6.82	
22.0	S9 - 23	6.90	4.00	15.91	16.8	6.90	8.193	-0.160	S	3.25	13.01	6.74	
22.5	S9 - 235	6.83	4.00	15.91	14.5	6.82	8.098	-0.150	S	3.24	12.92	6.65	
23.0	S9 - 24	6.75	4.00	15.91	12.2	6.74	7.991	-0.120	S	3.23	12.87	6.57	

4.02

Powder S9	
Tool set SNUN120412 - Phase 3 (Largesize)	
Area - pressed	250.1
S Height	6.40
SG	12.38
App. Density	Volume 1.0187 cm ³
Press height	8.00
Weight	12.900
Tool	25 %
	%

Powder details	
Date :	Links
Item no : 4001026	Powder sheet
Batch no : 89311	RTP
Co % : 10.0 %	Spray drying
Grain size : 2 - 3 μ	
PRZ reclaim? :	
PEG content : %	

Equivalent Tool %	Pressing (Green)										Sintered		
	Marking	Th. Mass	Height	IC	Force	Weight	Fill height	Press O.S	Rating	Height	IC	weight	
20.0	S9 - 20	14.39	8.01	15.91	25.7	14.41	16.978	-0.250	SS/bk	6.64	13.13	14.09	
20.5	S9 - 205	14.24	8.00	15.91	23.1	14.25	16.813	-0.240	SS	6.65	13.09	13.92	
21.0	S9 - 21	14.10	8.00	15.91	20.5	14.09	16.636	-0.230	SS	6.60	13.04	13.77	
21.5	S9 - 215	13.95	8.00	15.91	17.9	13.93	16.436	-0.220	SS	6.58	12.99	13.60	
22.0	S9 - 22	13.81	8.00	15.91	16.4	13.82	16.236	-0.220	S	6.56	12.95	13.50	
22.5	S9 - 225	13.66	8.00	15.91	14.2	13.64	16.036	-0.210	S	6.52	12.89	13.33	
23.0	S9 - 23	13.51	8.00	15.90	12.4	13.52	15.871	-0.200	S	6.51	12.86	13.20	
23.5	S9 - 235	13.36	8.00	15.90	11.0	13.35	15.684	-0.190	S	6.50	12.81	13.04	
24.0	S9 - 24	13.21	7.99	15.90	9.8	13.21	15.600	-0.190	S	6.47	12.78	12.91	

A1.23.2 Grade S9 – Powder physical properties, SG and porosity reports

Table A1.111 – Screen fraction report for grade S9 test sample (granule sizes)

Total	>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
197.2 g	0.4 g	17.9 g	101.0 g	43.1 g	21.6 g	13.2 g

Table A1.112 – Screen fraction report for grade S9 (mass percent of previous table)

>300 μm	>212 μm	>125 μm	>90 μm	>63 μm	>0 μm
0.2 %	9.1 %	51.2 %	21.9 %	11.0 %	6.7 %

Table A1.113 – Apparent density report of grade S9 test sample

Sample weight	Container volume	Measured density	Historical density (1 yr)
80.1 g	25 cc	3.20 g/cm ³	3.25 g/cm ³

Table A1.114 – Sintered density report for grade S9 test pieces

Test piece	Dry mass	Wet mass	Wire mass	Water density	Water temperature	Calculated SG	Average SG
1	14.0845	13.0222	0.0676	0.9974	18.8	12.43	12.43
2	7.0929	6.5920	0.0676	0.9974	18.8	12.44	
3	3.5716	3.3526	0.0676	0.9974	18.8	12.43	

Table A1.115 – Porosity report for best and worst sintered test pieces (midsize) for S9

Grade	Best shrinkage marking	Worst shrinkage marking	Porosity best shrinkage	Porosity worst shrinkage	Commentary best	Commentary worst
S9	19 %	23 %	A00 B00	A00 B00	Very good	OK (Some A&B type)

Appendix 2

A2.1 – Negative and positive insert tests using ‘CR10’ grade powder

Powder CR10				Powder details			
Tool set SNUN120412 - Phase 2 (Midsize)				Date :	Links		
Area - pressed	248			Item no : 4001004	Powder sheet		
S Height	3.20		Green Volume 0.9947 cm ³	Batch no :	RTP		
SG	14.41		Volume 0.5093 cm ³	Co % : 9.5 %	Spray drying		
App. Density	3.12		Volatile 2.80%	Grain size : < 1 μ			
Press height	4.02			PRZ reclaim? :			
Weight	7.545			PEG content : %			
Tool	25.5	%					

Equivalent Tool %	Pressing (Green)										Sintered				Green density
	Marking	Th. Mass	Height	IC1	IC2	Force	Weight	Fill height	Press O.S	Rating	Height	IC1	IC2	weight	
24.0	CR10-24	7.81													
24.5	CR10-245	7.72	4.03	15.91		40.5	7.74	9.815	-0.200	SH	3.25	12.86			7.68
25.0	CR10-25	7.63	4.02	15.91		35.3	7.64	9.701	-0.190	G	3.24	12.81			7.60
25.5	CR10-255	7.54	4.01	15.91		30.9	7.52	9.535	-0.180	G	3.22	12.75			7.50
26.0	CR10-26	7.45	4.00	15.91		28.4	7.43	9.396	-0.180	B	3.19	12.71			7.43
26.5	CR10-265	7.36	4.01	15.91		25.0	7.35	9.270	-0.170	G	3.19	12.65			7.33
27.0	CR10-27	7.27	4.00	15.91		23.3	7.28	9.169	-0.170	G	3.17	12.62			7.28

A2.2 - Negative and positive insert tests using 'PI-25' grade powder

Powder	PI25
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	14.15
App. Density	3.64
Press height	4.00
Weight	7.359
Tool	25
	%
	8.32 to 8.15
	GD
	20.2 to 21.0
	Expected
	2.11%
	Volatile
	Volume
	0.5093 cm ³
	Green Volume
	0.9947 cm ³

Powder details		Links
Date :		
Item no :	4001019	Powder sheet
Batch no :	107342	RTP
Co % :	%	Spray drying
Grain size :	μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent Tool %	Pressing (Green)											Sintered			Green density
	Marking	Th. Mass	Height	IC1	IC2	Force	Weight	Fill height	Press O.S	Rating	Height	IC1	IC2		
19.5		8.36	4.01	15.92	-	36.1	8.36	9.332	-0.200	G				8.34	
20.0		8.28	4.01	15.92	-	32.1	8.27	9.231	-0.190	G				8.25	
20.5		8.20	4.00	15.91	-	27.5	8.18	9.120	-0.180	G				8.18	
21.0		8.12	4.00	15.91	-	25.2	8.11	9.020	-0.170	G				8.11	
21.5		8.04	4.00	15.91	-	22.7	8.05	8.942	-0.160	B				8.05	
22.0		7.96	4.00	15.91	-	20.3	7.96	8.853	-0.150	G				7.96	
22.5		7.87	4.00	15.91	-	17.6	7.87	8.665	-0.140	G				7.87	

Powder	PI25		
Tool set	SEHT		
Area - pressed			
S Height	4.98	Green Volume	0.8282 cm ³
SG	14.15	Volume	0.4140 cm ³
App. Density	3.64	Volatiles	
Press height	6.27		
Weight	5.858	Expected	20.2 to 21.0
Tool	26	% GD	8.32 to 8.15

Powder details		Links
Date :		
Item no :	4001019	Powder sheet
Batch no :	107342	RTP
Co % :	%	Spray drying
Grain size :	μ	
PRZ reclaim? :		
PEG content :	%	

Equivalent Tool %	Pressing (Green)										Sintered			Green density
	Marking	Th. Mass	Height	IC1	IC2	Force	Weight	Fill height	Press O.S	Rating	Height	IC1	weight	
19.5		6.78	6.28	-	-	19.0	6.79	11.950	0.100	G				8.20
20.0		6.72	6.28	-	-	16.5	6.72	11.690	0.110	G				8.11
20.5		6.65	6.28	-	-	13.8	6.63	11.390	0.110	G				8.01
21.0		6.59	6.28	-	-	12.3	6.57	11.126	0.120	B				7.93
21.5		6.53	6.28	-	-	11.4	6.52	10.991	0.120	B				7.87
22.0		6.46	6.27	-	-	10.7	6.47	10.790	0.120	G				7.81
22.5		6.40												0.00

A2.3 - Negative and positive insert tests using 'P40M' grade powder

Powder details		Links
Date :	Item no : 4001012	Powder sheet
	Batch no : 72086	RTP
	Co % :	Spray drying
	Grain size :	μ
	PRZ reclaim? :	
	PEG content :	%

Powder	P40M
Tool set	SNUN120412 - Phase 2 (Midsize)
Area - pressed	250.1
S Height	3.20
SG	13.17
App. Density	3.29
Press height	4.00
Weight	6.863
Tool	25
%	
Expected	20.2 to 20.7
GD	7.72 to 7.76
Green Volume	0.9947 cm ³
Volume	0.5093 cm ³
Volatile	2.32%

Equivalent Tool %	Pressing (Green)										Sintered				Green density
	Marking	Th. Mass	Height	IC1	IC2	Force	Weight	Fill height	Press O.S	Rating	Height	IC1	IC2	weight	
19.5		7.80	4.00	15.93	-	55.6	7.79	9.338	-0.280	G					7.79
20.0		7.73	4.01	15.92	-	50.3	7.73	9.254	-0.260	G					7.71
20.5		7.65	4.01	15.92	-	44.9	7.66	9.171	-0.240	G					7.64
21.0		7.58	4.01	15.92	-	40.3	7.57	9.075	-0.220	G					7.55
21.5		7.50	4.02	15.92	-	34.9	7.50	8.979	-0.190	G					7.46
22.0		7.42	4.02	15.92	-	30.0	7.42	8.883	-0.170	G					7.38
22.5		7.34	4.01	15.92	-	27.5	7.35	8.799	-0.170	B					7.33

A2.4 - Negative and positive insert tests using 'K15M' grade powder

Powder details									
Date :					Links				
Item no : 4001009					Powder sheet				
Batch no : 114825					RTP				
Co % : %					Spray drying				
Grain size : μ									
PRZ reclaim? : %									
PEG content : %									

K15M									
Tool set SNUN120412 - Phase 2 (Midsize)									
Area - pressed		250.1	Green Volume		0.9947	cm ³			
S Height		3.20	Volume		0.5093	cm ³			
SG		12.86	Volatile		2.50%				
App. Density		3.03 (measured)							
Press height		4.00	Expected		21.5				
Weight		6.713	GD		7.35				
Tool		25	%		7.35				

Equivalent Tool %	Pressing (Green)										Sintered				Green density
	Marking	Th. Mass	Height	IC1	IC2	Force	Weight	Fill height	Press O.S	Rating	Height	IC1	IC2	weight	
20.5		7.48	4.02	15.93	-	70.6	7.48	9.568	-0.320	H					7.44
21.0		7.41	4.01	15.93	-	61.9	7.39	9.453	-0.300	H					7.37
21.5		7.34	4.02	15.93	-	54.8	7.34	9.364	-0.270	H					7.30
22.0		7.26	4.03	15.92	-	46.8	7.25	9.249	-0.230	G					7.19
22.5		7.18	4.03	15.92	-	40.3	7.17	9.134	-0.210	G					7.11
23.0		7.11	4.00	15.92	-	38.7	7.07	9.057	-0.240	G					7.07
23.5		7.03	4.00	15.92	-	35.6	7.03	8.993	-0.230	G					7.03
24.0		6.95	3.99	15.92	-	30.6	6.93	8.878	-0.220	B					6.94

Powder	K15M
Tool set	SEHT
Area - pressed	
S Height	4.98
SG	12.86
App. Density	?
Press height	6.27
Weight	5.457
Tool	26
	%
	GD
	7.60
	20
	Expected
	Volatile
	Volume
	0.4140 cm ³
	Green Volume
	0.8282 cm ³

Powder details	Links
Date :	
Item no : 4001009	Powder sheet
Batch no : 114825	RTP
Co % :	Spray drying
Grain size :	μ
PRZ reclaim? :	
PEG content :	%

Equivalent Tool %	Pressing (Green)											Sintered				Green density
	Marking	Th. Mass	Height	IC1	IC2	Force	Weight	Fill height	Press O.S	Rating	Height	IC1	IC2	weight		
22.0		6.02	6.30			36.1	6.04	12.300	0.070	G					7.29	
22.5		5.96	6.30			29.6	5.96	12.039	0.080	G					7.20	
23.0		5.90	6.30			26.7	5.91	11.800	0.090	G					7.14	
23.5		5.84	6.29			23.0	5.82	11.509	0.100	G					7.03	
24.0		5.77	6.28			21.0	5.77	11.202	0.100	B					6.97	

Appendix 3

A3.1 Porosity reports for sintered test pieces

Powder	Porosity B	Porosity W
230	21%.	23%.
	V. Good	Good.
	Acc Bcc	Acc Bcc.
AP20	21.5 %.	24 %.
	Good Acc Bcc	Good Acc Bcc (general)
	instances of A & B	instances of A & B.
CN10	24%.	26%.
	Good Acc Bcc	Not bal. Acc Bcc
CR10	Shr. 24%.	Shr. 25
	Very Good.	Good Acc Bcc
	Acc Bcc	some instances of Acc Bcc
H1P	21	25.
	V. Good	Good.
	Acc Bcc	Acc Bcc
H6	23.5%.	25.5%.
	V. Good	V. Good.
	Acc Bcc	Acc Bcc
K15M	Acc Bcc	Acc Bcc.
	23%.	25%.
	OKish not gus	OKish not gus
MZ15	22% Shrink.	23%.
	Acc MAN Bcc	Acc MAN Bcc
	Scratches	very good.
N6/194	22.5	24
	Acc Bcc Acc	Acc Bcc Acc

Accepted
Excellent

Accepted
Excellent.

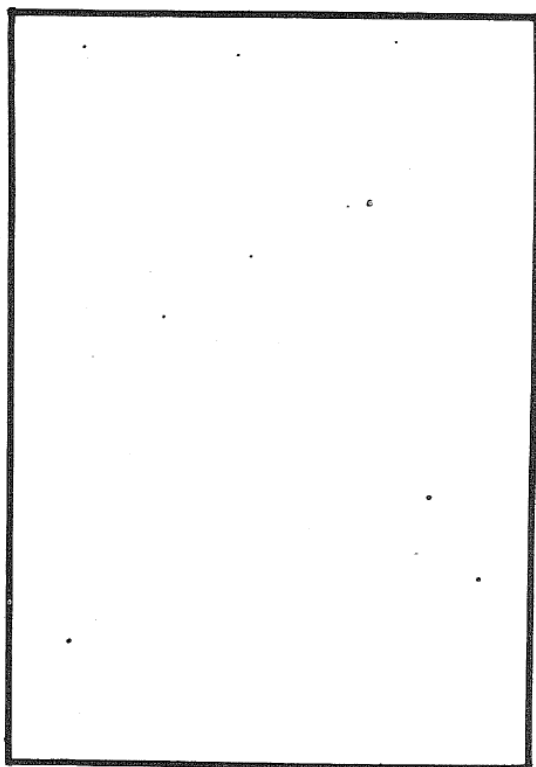
B

W

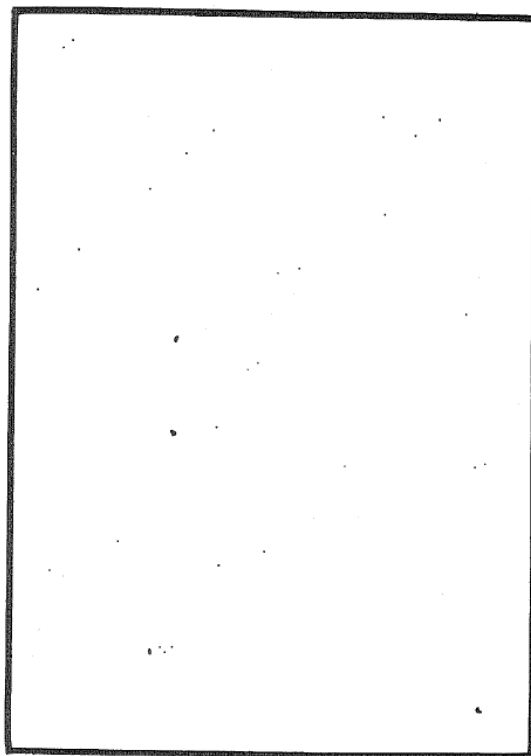
P25M	20% V.G.	24% ok.
	A00 B00	Mostly A00 B00
		instances of A & B.
P30M	20%.	24%.
	U.Good	Good.
	A00 B00	A00 B00
P40M	Shrink. 21%.	24%.
	A00 B00	A00 B00
	very Good.	Good → swatches
P511	22%.	24%.
	Excellent	Excellent
	A00 B00	A00 B00
P7	22%.	23%.
	Good (instances of A)	Good
	A00 B00	A00 B00
PBF	20.5%.	24%.
	U.Good. (Dirt.)	V.Good
	A00 B00	A00 B00
PBT	20%.	24%.
	ok instances of	ok.
	A02 B02.	A02 B02.
PC-20	21	23
	Good	Good
	A00 B00	A00 B00
PI-15	21 V.G.	24 V.G.
	A00 B00	A00 B00

PI-25	21%.	24%.
	V. Good	V. Good.
	Acc B00	Acc B00
PI-40	22%.	25%.
	V. Good	Good
	Acc B00	Acc B00
MT16	22%.	24%.
	V. Good.	Good.
	Acc B00	Acc B00
Q3	SN: 22.5%.	SN: 24%.
	Very Good.	Good
	Acc B00	Acc B00
S9	11%.	23%.
	V. Good.	Not Bad - ok.
	Acc B00	Acc B00, some B & A (very

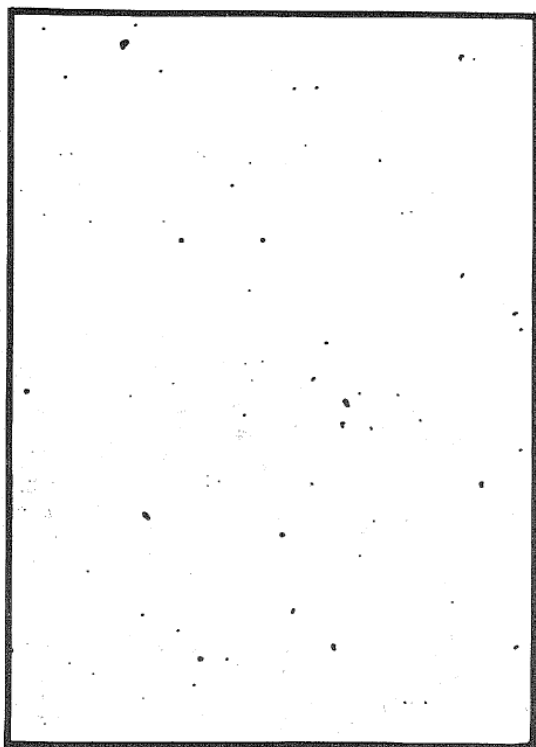
A3.2 Company micrographs to evaluate porosity



A02 0,02 Vol. %



A04 0,06 Vol. %

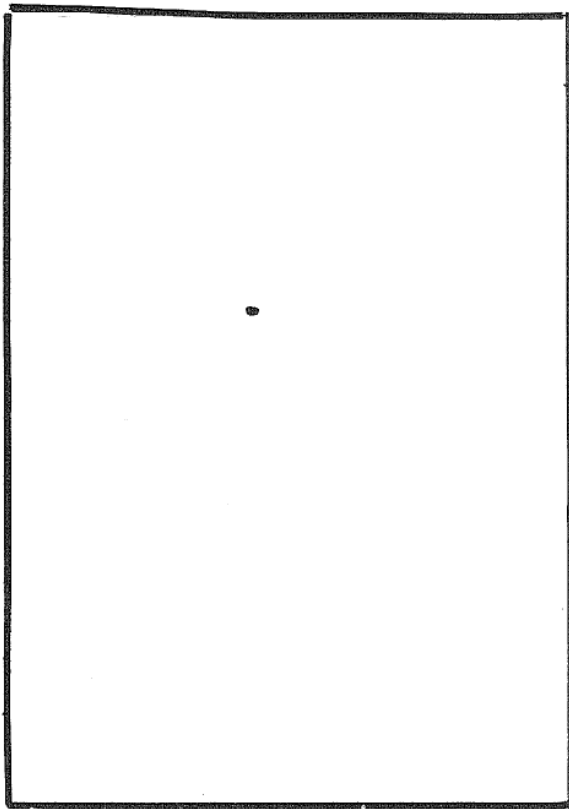


A06 0,2 Vol. %

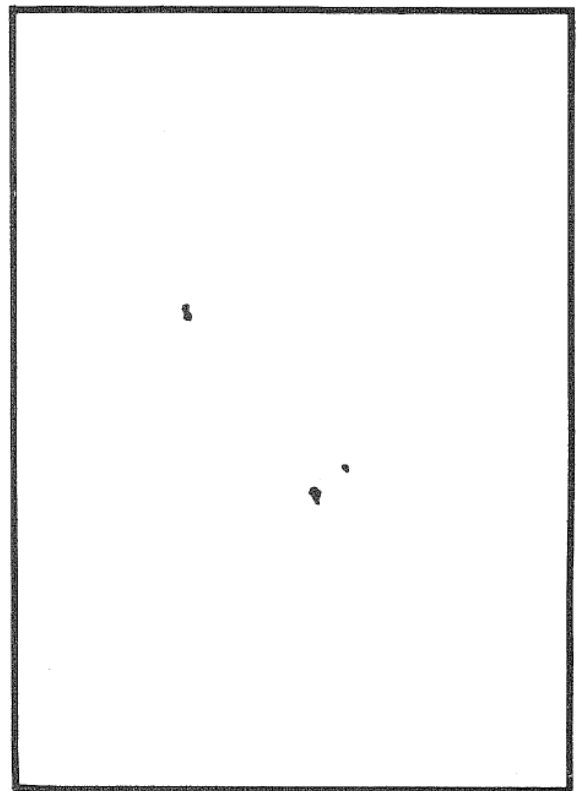


A08 0,6 Vol. %

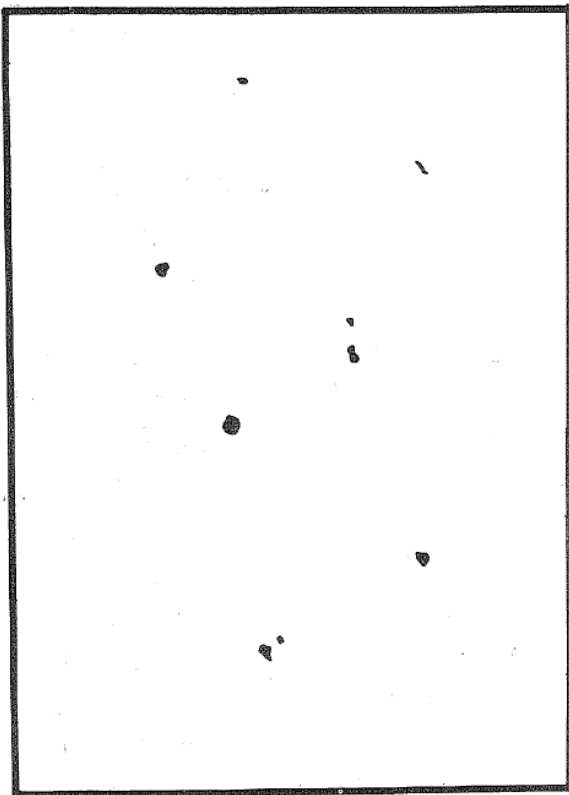
Bild 2 A-Porosität (x 200)



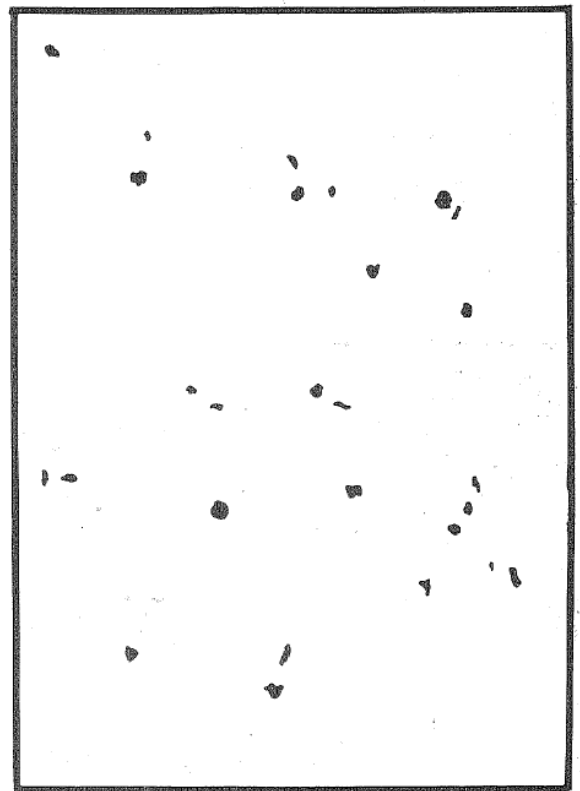
B02 0,02 Vol. %
(140 Poren/cm²)



B04 0,06 Vol. %
(430 Poren/cm²)

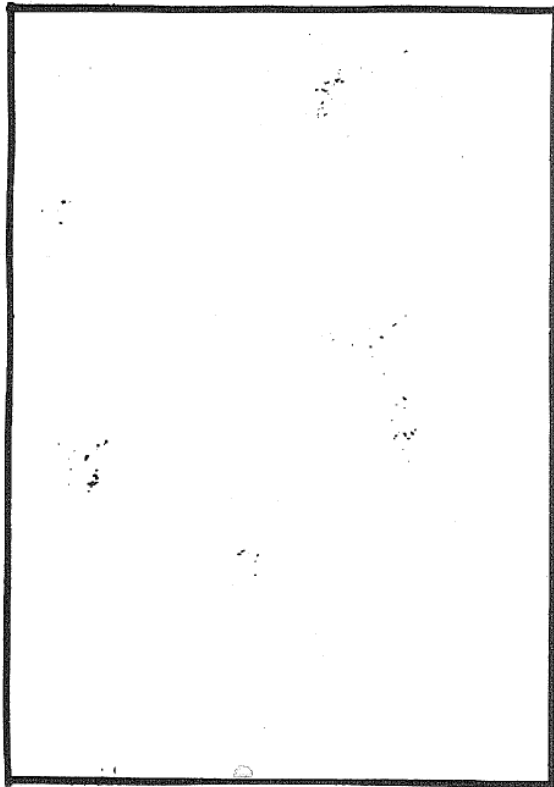


B06 0,2 Vol. %
(1300 Poren/cm²)



B08 0,6 Vol. %
(4000 Poren/cm²)

Bild 3 B-Porosität (x 100)



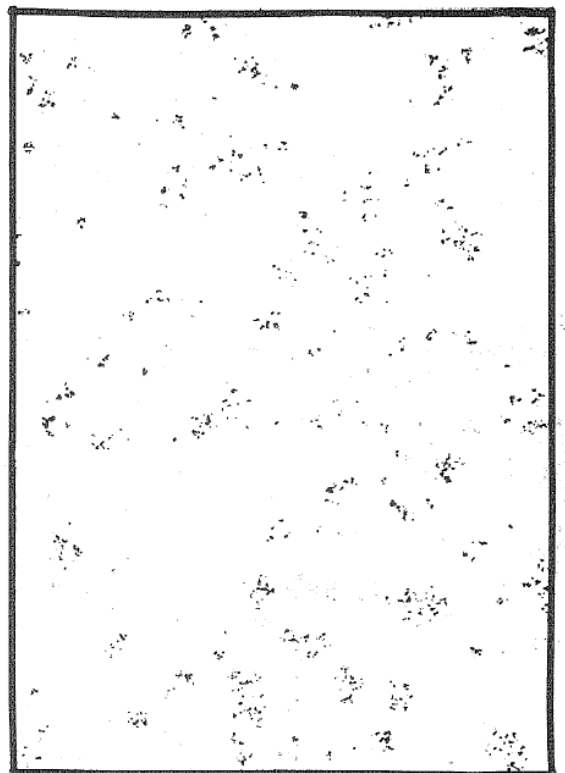
C02



C04



C06



C08

Bild 4 Freier Kohlenstoff (x 100)