AN INVESTIGATION INTO IMPROVING THE SPALL RESISTANCE OF
POLYCRYSTALLINE DIAMOND COMPACTS

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DECLARATION

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

(Signature of candidate)

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Abstract

An investigation of polycrystalline diamond compact (PDC) cutter failures, which are industrially known as spalling, was conducted by exploring changes in the diamond layer architecture and edge geometry of the cutter. Layer architecture was investigated through the use of layered functionally graded (FG) structures. Twenty one FG variations were prepared by the tape casting method and sintered using a high-pressure, high-temperature press.

The vertical borer test (VBX), a laboratory test method, was used to gauge the improvement in spall resistance of the FG specimens against the benchmarks. Due to cost constraints associated with VBX testing, of the 21 available specimens, only four variations were tested for spalling. Contrary to expectation, it was found that all four specimens spalled during VBX testing despite showing a slight improvement in the spall area. For this reason, this route was abandoned. It was concluded that the use of layered structures is not effective in resolving the spalling problem.

The use of novel edge geometry was investigated by taking three standard products and creating new geometric profiles on the specimens using a spark erosion machine. Each profile comprised a depression on the front face of the cutter. The specimens with novel geometry were also tested on the VBX. The spall was found to be confined between the chamfer breach and the depression feature. The depression appeared to have stopped the

spall from propagating beyond the allowable spall limit of 1.2mm. On the basis of this finding, it was concluded that spalling was successfully resolved. It is recommended that further optimization of this solution should be explored in field testing. In addition, a cost-effective way to fabricate the geometric profiles on the cutters should be further investigated because creating specimens using the spark erosion machine was quite expensive. Therefore, it is not viable for fabrication of large production volumes.

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DEDICATION

In memory of my father; Amos. You left fingerprints of grace to my life. You will not be forgotten.

ACKNOWLEDGEMENTS

CHAPTER 1

Introduction

It is well recognized that the diamond layer plays an important role in commercial polycrystalline diamond compacts (PDC). PDCs usually comprise the diamond layer, the working component, coated on a tungsten carbide (WC) substrate, the backing component, as illustrated in Figure 1.1. The typical shape of a PDC is a cylinder. Part of the reason why a cylindrical shape is preferred over other shapes on a PDC is that it is easy to achieve a large cutter density for a given drill bit profile [1]. The common size of a commercial PDC has a diameter of 16 mm, height of 12 mm and diamond layer thickness of 2 mm.

Diamond layer

WC substrate
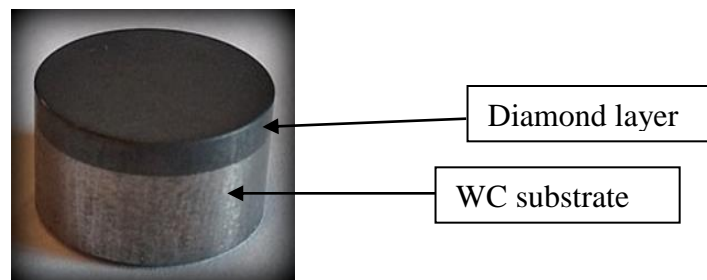
Figure 1.1 Commercial PDC product [1].

The diamond layer is usually made up of special diamond grit blends [1]. Research indicates that changes in the composition and geometry of the diamond layer influence the field performance in PDCs. The role of composition and geometry with respect to the performance of PDCs is central to this report. The predominant parameter that influences performance in this context is durability. Durability is defined by two main components, that is, resistance to wear and resistance to spalling. In this work, only the resistance to

spalling aspect is looked at because the challenges associated with this area have huge commercial impact.

Spalling is a failure mode defined as the mechanical breakage of the diamond layer on the PDC during use due to its inherently low fracture toughness. Typical examples of spalled PDCs are illustrated in Figure 1.2.

(a)   (b)
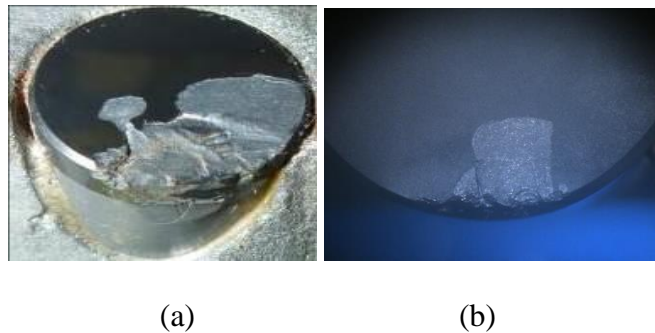
Figure 1.2 Spalled PDC cutters from (a) the field and (b) laboratory testing [2].

PDCs suffer from spalling problems during use in drill bits for oil and gas extraction. Commercially, spalling is extremely detrimental because it increases the overall drilling time due to the frequent stops required for replacement or repair of spalled PDC drill bits which causes drillers millions of dollars in drilling costs [2].

The spall resistance of PDC cutters is conventionally assessed using the laboratory test method called the Paarl granite test, also known as the VBX test. Testing of the PDC specimens involves cutting a large granite block on a vertical turret lathe (VTL).

Information from this test is used to gauge the spalling characteristics of the cutter by comparing the experimental cutters against their benchmark cutters. VBX is by far the most trusted test method that mimics actual conditions associated with drilling and the results are believed to be representative of how the PDC cutter will perform in the field [3].

There is an industrial imperative for spall resistant cutters. This need is conventionally addressed by the use of coarser PCD structures. Although resolving spalling through this route is believed to be feasible, the abrasion resistance is usually compromised which is equally important for achieving an adequate rate of penetration (ROP); i.e. drilling faster during application. This is because, fundamentally, abrasion resistance and fracture toughness are inversely related to each other – implying one property is improved at the expense of the other and vice versa [4].

This study explores two possible routes which are not presented in open literature. These routes are:

(a) The use of diamond layered structures

(b) The use of novel edge geometry

The purpose of this study is to assess the potential feasibility of these routes. Consequently, the hypothesis is that either a graded approach or geometric approach can lead to some improvement in spall resistance.

Diamond layered structures are gradient layered structures where each structure provides a small amount of functionality so that the overall properties of the multi-layered structure are superior to those of the individual layers. These structures are of interest because, unlike a conventional PDC cutter whose diamond feed layer is composed of a single layer, a gradient of mechanical properties can be created to achieve a combination of properties which might be the solution to the spalling problem.

Edge geometry PDCs are shaped cutters whose cutter faces are usually modified geometrically to improve their performance. This area is of interest because performance can be boosted by creating geometric shapes on the cutter, in contrast to the conventional flat round face on a standard PDC. Shaped PDC cutters have become an encouraging area of research which offers avenues for making better cutters. It is a new evolution with potential yet to be fully explored [5].

While the development of a spall resistant PDC has been under study for several years, finding an effective solution to the spalling problem has become increasingly more important. The purpose of this research, therefore, is to investigate how changes in diamond layer architecture and edge geometry affect the spall resistance of PDCs so that it can be determined whether a diamond layer architecture and/or novel edge geometry could be a solution to the spalling problem. It is hoped that the results of this investigation will show an improvement in spall resistance over a benchmark cutter on VBX results. If this is the case, then this cutter could be developed further with a view to field testing and possibly even eventual introduction into the market.

CHAPTER 2

Literature review

A drill bit is a mechanical tool designed to cut through rock formations and generate wellbores on onshore and offshore fields for oil and gas extraction [6]. There are two main types of drill bits in the market, that is; fixed cutter bits which are well known as PDC cutter bits and roller cone bits [7]. For cutting action, fixed cutter bits use PDC cutters while roller cone bits use tungsten carbide inserts [8].

In 2015, the global oil and gas drill bit market slumped to about $2 billion after reaching $5 billion in 2014 [9]. The challenge in industry growth is due to the declining oil price caused by a systemic demand-supply imbalance in the global crude oil market [9]. The imbalance is caused by crude oil oversupply triggered by prosperous US shale oilfields, OPEC overproduction and sluggish demand in Europe and Asia. The global forecast predicts that the global oil and gas drill bits market will be negatively impacted by the prevailing decline in prices for the next two or three years, which could result in reduced investments in drilling activities [10].

The market share in the drill bit market is dominated by five key players, namely Baker Hughes, Varel International, Smith International, National Oilwell Varco and Halliburton. They account for over 70% of the market share [11]. Smith International continues to lead the industry with 22.5% market share by total market revenue. To sustain industry dominance, Smith International has engaged in innovation initiatives that support a wide range of application-specific bit activities [11].

Geographically, the global oil and gas drill bits market is segmented into seven continents of the world [12]. In terms of the total share by volume, North America dominates the industry with 35%. The reason for the dominance is thought to be the shale gas boom in the region. Production and exploration activities in this region are expected to grow in the coming years [13].

The drill bit market had been dominated by roller cone bits, until PDCs for fixed cutter bits were introduced by General Electric (GE) in 1971 [14]. Soon after this introduction, PDC bits demonstrated possibilities to revolutionize the drill bit industry. With the expansion of the oil fields from North American basins to the Persian Gulf countries and North Africa, there was a need during this time for an innovation that would cope with the new reserves particularly those that had hard rock formations. This was because oil and gas wells with hard rock formations required equipment with an enhanced rate of penetration and durability. The invention could not have come at any time better than 1971 [15].

Due to higher efficiency and durability possibilities with PDC cutters, fixed cutter bits began gaining popularity in the market. Drill bit vendors, PDC cutter manufactures and individuals were interested in the technology and executed small scale drilling and exploration experiments with this new PDC cutter. From 1972 through to 1974, many problems associated with this technology were being identified and solved.

In 1974, although PDCs were still in early development, initial successes began being reported at the technical drilling conferences. In the middle of 1974, Diamant Boart, now part of Baker Hughes, reported some degree of success in drilling salt rocks in the Persian Gulf basins. In early 1976, Drilling & Service (D&S), which later became part of Hycalog, reported some success in the North Sea [16]. These successes included many improvements in drilling practices, bit designs and hydraulics. The improvements assisted in providing some positive signals for the commercial possibility of the prototype PDC [16].

Development of the PDC cutter continued until GE commercially launched their PDC production line in 1976. Soon after that, competition among established vendors began. New entrants entered the market to compete [17]. The global oil and gas drill bit market started growing and became the driving force for moving the technology forward. Cutter improvement in the midst of competition added value to the performance front, preventing the PDC cutter from becoming a commodity product [18].

In the early 80s, there was growing concern with the performance of PDC bits in many drilling applications. The cutters delaminated from the backing component too frequently. The cutters spalled, broke and chipped too often. The cutters suffered from thermal degradation. The identified issues needed to be resolved to exploit the full merits of PDC technology. With the significant improvement in drill bit design at the time, PDC cutter development initiatives among competitors began [18].

PDC cutter vendors such as Valdiamant, DeBeers Industrial Diamond and US Synthetic (USS) began initiatives on the improvement of the mechanical fracture of PDC cutters. In the mid-80s, Valdiamant entered the market by supplying prototype PDCs to drill bit vendors. Their major contribution was the commercialization of the non-planar interface PDC to manage thermal residual stresses at the interface between the diamond layer and the substrate. These arose in manufacture and were a problem on the early cutters and caused failures such as delamination and cracking [18].

Soon after that, non-planar interface cutters for better management of the residual stress became the standard in the market. A lot of interface designs have been patented and commercialized by the PDC cutter makers and individuals. Examples of non-planar interface designs are shown in Figure 2.1[19].
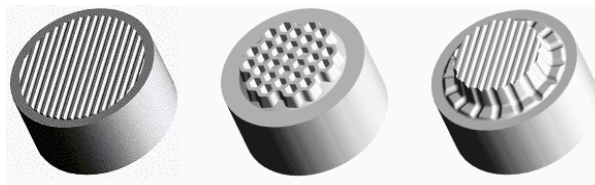
Figure 2.1. US Synthetic's patented non planar interface designs [20].

Finite Element Analysis (FEA) became popular as a cutter development tool for managing stresses at the interface [20]. FEA analysis allows the sizes and shapes of the interface features on the substrate to be optimized to give the most favorable residual stress states in the PCD layer at room temperature, which implies engineering more

compression in the working region of the PCD layer and / or reducing tensile "hot spots" in critical areas of the PCD layer. This practice became a step change at the time in reducing the risk of delamination of the cutting layer from the substrate and thus, extended the operating life of the cutter [21].

In 1981, DeBeers Industrial Diamond, now known as Element Six, entered the market. Their contribution was the introduction of the thicker diamond table which was beneficial in tough applications. This success helped in making major industry penetrations in the 1980s [22].

In 1983, US Synthetic (USS) entered the market. They became the first to commercialize tough diamond tables in the industry at the time. Early cutters used a coarse unimodal diamond grit mix and USS pioneered the development and introduction of bi-modal diamond and multimodal mixes which became an industry standard in the 1990s. USS became the market leader in 1997 [28].

In the 1980s, GE and Sumitomo studied the removal of cobalt from the diamond layer using leaching and did not patent the work [9]. Later, Hycalog, a division of National Oilwell Varco (NOV), snatched the idea and patented a leached diamond cutter technology [29]. The wear resistance benefit with a leached cutter can be seen in Figure 2.2.
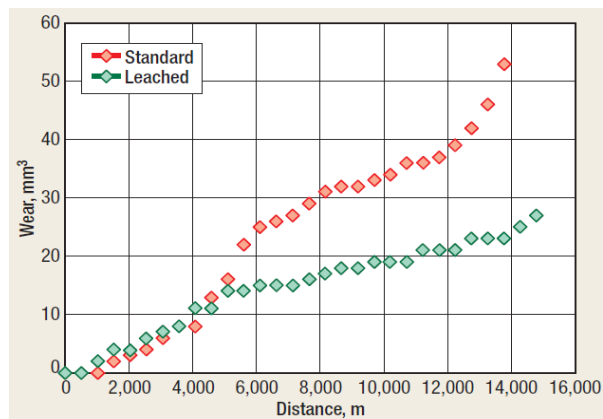
Figure 2.2. Wear rate for leached vs. non-leached cutters on the VBX test [30].

In Figure 2.2, two identical PDC cutters — one leached and the other non-leached were subjected to VBX testing. At the end of test, after cutting about 15,000 m of rock, the wear rate of the leached cutter is superior to that of the non-leached cutter. The poor wear resistance of non-leached cutters is thought to be caused by the presence of cobalt in the diamond layer, an indispensable catalyst during manufacturing, but deleterious during application [31]. Today, the whole industry uses this method and NOV collects royalties on this invention [31].

Sintered PCD usually comprises a network of inter-grown diamond particles in a cobalt matrix. In the non-leached state, wear performance is found to be inferior due to accelerated wear. This is because cobalt is softer and wears quicker than diamond. In addition, cobalt converts diamond into graphite at temperatures in excess of 700^0C. This problem is usually addressed by partial or complete leaching of the cobalt from the PCD with mineral acids. Although the leaching treatment significantly improves wear performance, the spalling problem is introduced [31].

To avoid paying royalties on leached cutters, competitors have tried alternative routes with the hope of creating a PDC cutter with properties nearly as good as a leached cutter. In this effort, Barker Hughes have patented a cutter made using nanotechnology. In the invention it is claimed that by incorporating ceramic nanoparticles into the PCD layer, during manufacturing with heat and pressure, the nanoparticles fill the spaces around the big diamond grains and thus restrict cobalt infiltration from the substrate into the diamond layer. The cutter has not yet hit the market. It is said this approach has some manufacturing challenges which need to be overcome before the benefits can be realized [32].

In another effort, Element Six filed a patent for a cutter made from alternative catalysts. In the invention, it is claimed that different catalyst materials other than cobalt such as iron, nickel, manganese, aluminum, calcium carbonate and combinations of these can reduce the problems caused by cobalt during application. There is no cutter in the market that is made from this approach yet. It is said there are many manufacturing challenges associated with these alternative catalysts. The quality of sintering is not as good as that obtained using cobalt. Cobalt remains the best catalyst for the manufacture of PDC cutters [33].

In the mid-1990s, an innovation that became widespread was the introduction of chamfer technology. The chamfered cutter dealt with chipping along the edge of the working surface, thereby increasing the fracture resistance of the cutter during service. Since then,

the use of chamfered cutters has become a standard in the industry and different chamfer designs have been patented and commercialized by the PDC cutter makers and individuals. Improvements in chamfer technology have evolved to become an area with potential benefits. Recently, Baker Hughes has successfully introduced a patented dual chamfer design for regions with hard rock formations [33].

In 1995, another innovation was seen with the introduction by Baker Hughes of a patented polished cutter [34]. A polished cutter is thought to reduce frictional heat in specific rock formations. It is said that the cutter was successful in full-scale drilling tests and field trials. [34]

In 1999, a step change was seen when Element Six pioneered the introduction of a cutter with a layered diamond structure. The cutter comprised a fine layer backed up by a coarser layer on a non-planar interface substrate. The fine layer was intended for abrasion resistance while the coarser layer was intended for impact resistance. This approach was possible because abrasion resistance decreases with diamond grain size, while fracture toughness increases with grain size [32]. This signature cutter became the foundation for abrasive and high-impact applications. Today, Element Six remains the leading supplier of this product line [23].

This idea of layered structures was discovered in 1989 by the researchers at NKK Corporation in Japan. They were confronted with a problem when working on a project

for thermal-resistant structures of space shuttles. They could not find a uniform material with the right properties. Therefore, to solve the problem, they came up with a concept of fabricating a material by gradually changing or grading the material composition through the thickness. They then called this innovative material a Functionally Graded Material (FGM) [24].

Today, functionally graded materials have found a wide range of applications in aerospace, medicine, defense, energy and optoelectronics [25]. Several commercial products are produced with functionally graded structures. For example, Mitsubishi Materials introduced their functionally graded Miracle Coated Indexable Inserts, which are made of a carbide substrate coated with a graded structure on the surface. Functionally graded materials also occur in nature as bio-tissues of animals, such as bones and teeth, and plants [25].

There are two categories of fabrication processes for functionally graded materials (FGM) namely: thin and bulk FGMs. In thin FGMs, thin graded sections are coated on the surface of the substrate using techniques such as electro-deposition, chemical vapour deposition (CVD), plasma spraying, etc. Meanwhile, in bulk FGMs, mass production of materials is realizable using techniques such as powder metallurgy, tape casting, centrifugal force and solid free form casting [26].

Recently, a step change in cutter design was seen with Smith International developing the rotating cutter. An example of a rotating cutter is shown by the red dot in Figure 2.3.

Figure 2.3. The rotating cutter is marked by the red dot [27].

During drilling, the patented cutter is thought to continually present a new cutting surface to the rock face, allowing the entire circumference of the cutting edge to be used and thus extending the sharpness of the cutting edge without compromising bit durability [28]. Success with this bit technology has been reported.

Another recent step change is seen with the introduction of the indented cutter by Baker Hughes. The patented cutter design is shown in Figure 2.4.

Figure 2.4. Baker Hughes's proprietary cutter design with a depression in the center [28].

This indented design is thought to deal with the frictional heat generated during service by reducing the contact area of the cutter against the rock, keeping the cutting edge cooler for longer. The cutter is reported to lower the temperature by 40% [29]. It is reported that the field test showed an indented cutter drilling 12% farther than the conventional cutter. In addition, the penetration rate increased by 15% over that of the standard cutter. This performance benefit was attributed to the low frictional heat on the cutting edge compared to a standard cutter [29]. It is clear from this cutter that the use of geometric shapes on PDCs is a promising area of activity for making better cutters.

Due to the fall in crude oil price, there has been mounting pressure for optimized drilling performance. PDC manufactures in collaboration with drill bit vendors have invested a lot in innovation programs for reducing the operational costs of drilling. They have engaged in different research fronts to develop better cutters which can reduce the overall

drilling time by minimizing the stops required for replacement or repair of PDC drill bits [30].

However, the competition in the global drill bits industry is very high [31]. The competitive advantage is based on product differentiation and ground breaking solutions [31]. The life time of a newly commercialized PDC cutter is about 6 months before it is outdated. After this time, the customer requires a new cutter version. In this regard, innovation becomes a vital tool for drill bit vendors in the competitive race.

Today, among several research areas for PDC development, there are two that stand out. They have become so popular lately because they possess the potential for ground breaking solutions. A drill bit vendor or a PDC cutter manufacture or an individual that can provide a solution to either of the areas could gain the competitive advantage. The first area is the development of a spall resistant cutter, while the second area is the development of a thermally stable cutter. A spall resistant cutter seeks to reduce cost associated with removing the drill string to replace spalled cutters and to present the possibility of rotating the cutter after use in the field by containing the extent of cutter damage. A thermally stable cutter seeks to outperform or match the performance of a leached cutter. A lot of different ideas are being tried – but no breakthrough has been reported yet.

CHAPTER 3

Experimental investigation

This chapter details the experimental context of the investigation of diamond layered structures and edge geometry approaches. The experimental design of the investigation involved a series of experiments on both approaches. The effect of changes in diamond layer architecture was investigated first because this approach had the prospect of creating a new platform for diamond layered PDCs and the facilities for fabricating specimens were readily available. Changes in the PDC edge geometry do not require sourcing of raw materials, and it is also easy to create a geometric feature on the cutter but the benefits were considered to be fewer compared to changes in the architecture of layered PDC and so this approach was investigated second.

3.1 Layered structures approach

3.1.1 Fabrication of specimens

Description of different cutter designs

Three different basic structures were considered. In all cases the layered structure had a thickness of 3mm. The first configuration, known as the "type 1" design had each layer arranged circumferentially around the cutter. The second configuration, known as the "type 2" cutter had the layered structure placed on the cutting face of the cutter. The third configuration was more complicated in its internal design. It was also arranged on the

face of the cutter. The control cutter is a benchmark cutter whose VBX performance is outstanding. In this case, the control has a fine grit blend, and its diamond layer is not graded. The results of the control are taken from the database records. Cutter development activities compare themselves against this control. For example, in this study, designs type 1, 2 and 3 are compared against the control in performance in the results section. If any of the designs outperforms the control, that design will be considered to be a step change in performance and a better cutter is developed.

Type 1 design:

Figure 3.1 shows the diagrammatic sketch of the cutter of the set of variations of type 1. Type 1 design involves the arrangement of layers with varying fracture toughness in the radial direction. Layer 1, the surface layer, has high wear resistance but poor fracture toughness. Layer 4, the tough core layer, possesses high fracture toughness but poor wear resistance. Fracture toughness is configured such that it increases from layer 1 towards layer 4. The thinking is that as the spall crack propagates through the layers with different fracture toughness, it will lose energy and stop, thus containing the extent of the spall.
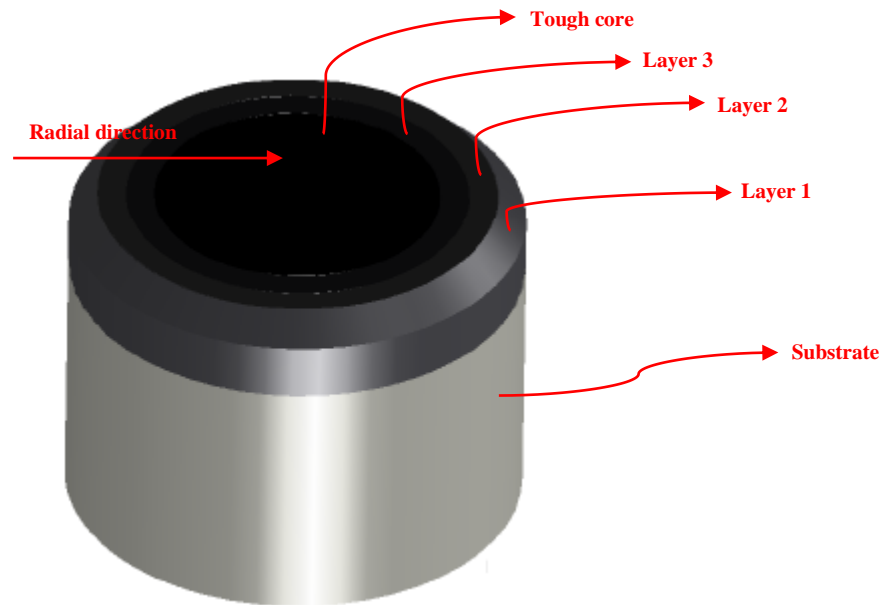
Figure 3.1 Diagrammatic sketch of type 1.

The parameters that can be varied in this approach include grading type (e.g. continuous or stepwise) and material type (e.g. cobalt or WC). Grading type defines the compositional gradient of the material being stacked through the thickness of the diamond layer. Figure 3.2 shows the common grading types that are considered in this work.
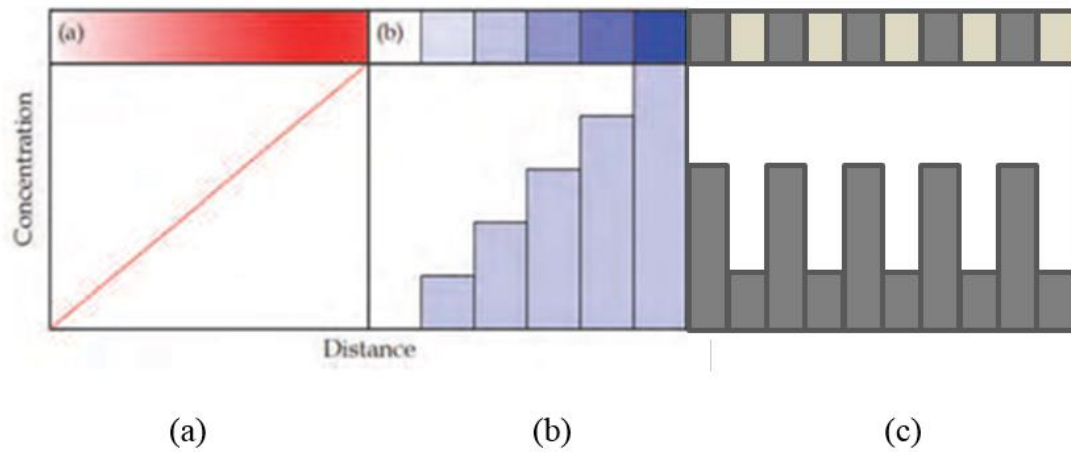
Figure 3.2 Gradation types (a) continuous, (b) stepwise and (c) alternating [41].

As an example, if the material type that is selected is cobalt, this would mean that 3 variations can be generated from cobalt material. The first variation could have interlayers stacked in a continuous manner, the second variation could have interlayers stacked in a stepwise fashion and the last variation could have interlayers stacked in an alternating manner.

Material type defines the selected materials that are used to create specific structures. In this work, WC, cobalt and size of the average grain size of the diamond grit are used. Figure 3.3 shows the different common structures that can be created.
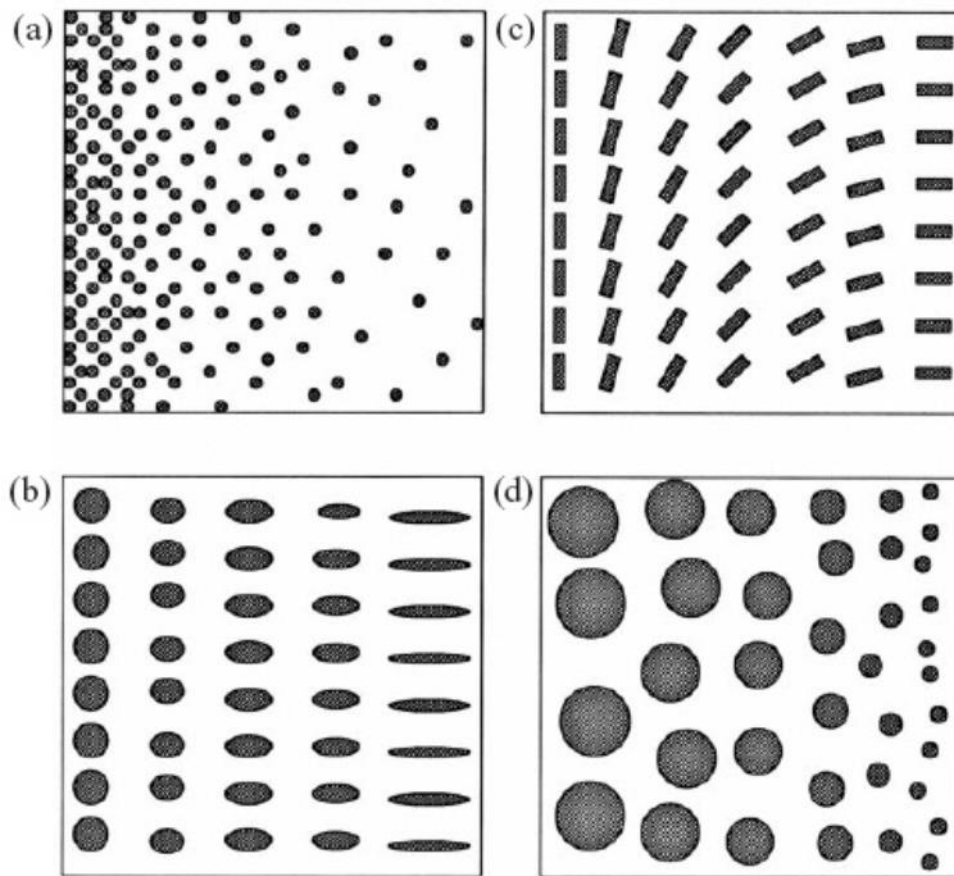
Figure 3.3 Different types of structures (a) volume fraction, (b) shape, (c) orientation and

(d) size [42].

In this work, only varying volume fraction and size structures are possible to create using

cobalt, WC and diamond grit materials. Structures with changes in shape and orientation

are difficult to create due to the lack of fabrication facilities.

The parameters that could be fixed in this approach include grading orientation (radial or

axial). Grading orientation defines the direction at which the interlayers are positioned

within the diamond layer. Interlayers are the individual layers with different compositions that are stacked together to form the diamond layer thickness of the cutter. For the Type 1 design variations, the grading orientation parameter varied in the radial direction only.

Based on these parameters, nine experimental combinations were generated as shown in Table 3.1 It can be seen in Figure 3.1 that all type 1 design variations are radially graded. "Material type" refers to the material parameter which is being varied radially across the structure. Under material type, for the WC material, the cobalt level was fixed at 6% to achieve adequate sintering. For the cobalt material, the WC level was fixed at 0% because when the cobalt level is high, the WC becomes a cobalt infiltration inhibitor resulting in sintering challenges. For the diamond grit material, when the diamond grit is the bulk matrix, a small amount of cobalt is needed to facilitate sintering. WC is not required in the diamond layer as it is considered a contaminant. For this reason, the WC level was fixed at 0% and the cobalt level was fixed at 1%. Each variation could then be tested with a minimum of 3 specimens as required for VBX testing.

Table 3.1 Type 1 design variations.

Number of variants	Grading type	Material type	Grading orientation
Variation 1	Continuous	Volume fraction (WC) Co=6%	Radial
Variation 2	Continuous	Volume fraction (Co) WC=0	Radial
Variation 3	Continuous	Size (Diamond grain size) WC=0 Co=1%	Radial
Variation 4	Stepwise	Volume fraction (WC) Co=6%	Radial
Variation 5	Stepwise	Volume fraction (Co) WC=0	Radial
Variation 6	Stepwise	Size (Diamond grain size) WC=0 Co=1%	Radial
Variation 7	Alternative	Volume fraction (WC) Co=6%	Radial
Variation 8	Alternative	Volume fraction (Co) WC=0	Radial
Variation 9	Alternative	Size (Diamond grain size) WC=0 Co=1%	Radial

Type 2 design:

Type 2 design involves the arrangement of layers with varying fracture toughness in the axial direction as shown in Figure 3.4. Unlike the Type 1 design, this design does not have a tough core. Interlayers are stacked strategically onto the substrate with increasing fracture toughness towards the substrate. In this design, the fracture toughness is configured such that it is lower on the surface layer (layer 1) and highest on layer 4. The thinking is that as the spall crack propagates deeper it will penetrate into the underlying layers with higher fracture toughness and therefore be retarded.
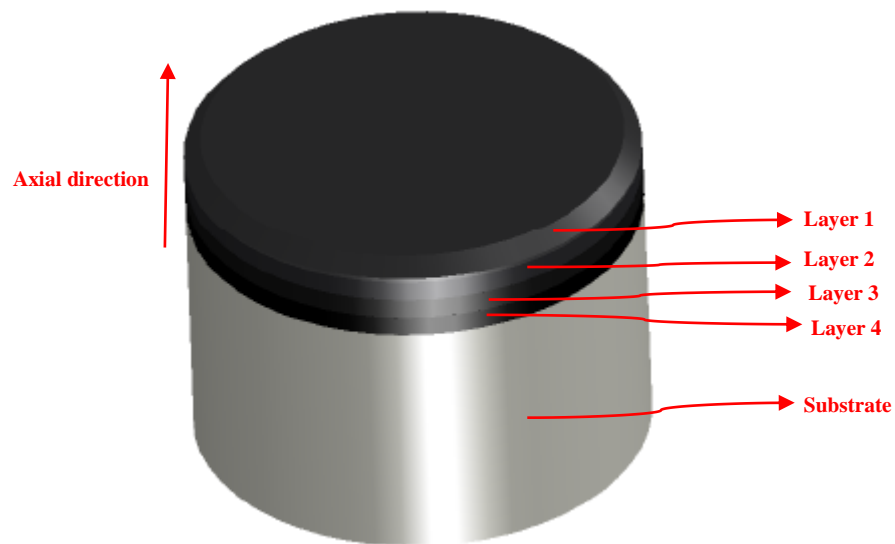
Figure 3.4 Diagrammatic sketch of type 2 design.

The parameters that could be fixed and varied in this design are listed on Table 3.2.

Table 3.2 Type 2 design variations.

Number of variants	Grading type	Material type	Grading orientation
Variation 1	Continuous	Volume fraction (WC) Co=6%	Axial
Variation 2	Continuous	Volume fraction (Co) WC=0	Axial
Variation 3	Continuous	Size (Diamond grain size) WC=0 Co=1%	Axial
Variation 4	Stepwise	Volume fraction (WC) Co=6%	Axial
Variation 5	Stepwise	Volume fraction (Co) WC=0	Axial
Variation 6	Stepwise	Size (Diamond grain size) WC=0 Co=1%	Axial
Variation 7	Alternative	Volume fraction (WC) Co=6%	Axial
Variation 8	Alternative	Volume fraction (Co) WC=0	Axial
Variation 9	Alternative	Size (Diamond grain size) WC=0 Co=1%	Axial

Once again, "material type" refers to the material parameter which is being varied, but in this case the variation is in the axial direction. The table of type 2 variations is similar to the table of type 1 variations. The only difference is that type 1 variations are graded in the radial direction while type 2 variations are graded in the axial direction. Each type 2 design variation could then be tested with a minimum of 3 specimens as required for VBX testing.

Type 3 design:

The Type 3 design involves the use of two distinct materials stacked in a chequered pattern. The distinct material types could be made from variations in cobalt or tungsten carbide composition or size in the average grain of the diamond grit. It is thought that a cutter with two wear fronts built up using two distinct materials stacked in a chequered pattern might be able to block spalling by disrupting the crack propagation through the two distinct material types. The diagrammatic sketch of the cutter to illustrate this hypothesis is shown in Figure 3.5.
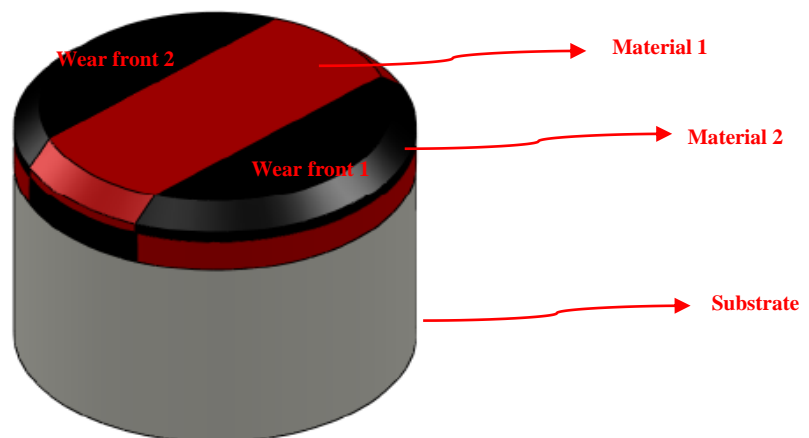
Figure 3.5 Diagrammatic sketch of type 3 design.

Due to the complexity associated with fabrication of this design, only two experimental combinations were generated. These variations are shown in Table 3.3. The two variations were created using firstly, variations in tungsten carbide composition and secondly, the average grain size of the diamond grit.

Table 3.3 Type 3 design variations.

Number of variants	Grading type	Material type
Variation 1	Chequered pattern	Volume fraction (WC) Co=6%
Variation 2	Chequered pattern	Size (Diamond grain size) WC=0 Co=1%

Preparation

Materials:

Cobalt, tungsten carbide and diamond grit powders were used in this research. Type 1 design specimens were graded in the radial direction while type 2 design specimens were stacked in the axial direction. Type 3 design specimens were stacked in a chequered pattern. Tape casting was the method chosen for producing diamond films for the grading operation.

Tape casting:

A total of 22 powder mixes were selected for the tape casting of the films for cobalt, WC and diamond grit material types.

Table 3.4 Powder mixes for diamond grit material.

Name	Composition
Mix 1	15G2:85G6 +1%Co
Mix 2	15G2:85G12 +1%Co
Mix 3	15G2:85G22 +1%Co
Mix 4	15G2:85G30 +1%Co
Mix 5	15G2:85G45 +1%Co
Mix 6	15G2:85G75 +1%Co

Table 3.4 shows the powder mixes for the diamond grit material. These mixes were graded by grain size. Therefore, a series of diamond mixes that varied gradually in grain size were created and were termed as mixes 1 to 6. Using mix 1 as an example; the designation 15G2:85G6+1%SP denotes the composition 15% by mass of grade 2 of

diamond powder and 85% by mass of grade 6 of diamond powder with 1% by mass spherical cobalt. In addition, it is worth noting that the WC level was fixed at 0% and the cobalt level was fixed at 1%.

The powder mixes for WC material were graded by creating a gradient in the composition of the WC material using a volume fraction method as shown in Table 3.5.

Table 3.5 Powder mixes for tungsten carbide (WC) material.

Name	Composition
Mix 7	15G2:85G22 +1%Co
Mix 8	85G22:15WC +6%Co
Mix 9	70G22:30WC +6%Co
Mix 10	55G22:45WC +6%Co
Mix 11	40G22:60WC +6%Co
Mix 12	25G22:75WC +6%Co
Mix 13	10G22:90WC +6%Co
Mix 14	75G22:25WC +6%Co
Mix 15	50G22:50WC +6%Co

Powder mixes graded by volume fraction of WC were termed as mixes 7 to 15. Mix 7 is the working layer and accordingly has no WC in it and has a reduced cobalt concentration of only 1%. The cobalt level on the remaining interlayers is fixed at 6%.

Table 3.6 shows the powder mixes for the cobalt material which were graded by the composition of the cobalt material.

Table 3.6 Powder mixes for cobalt material.

Name	Composition
Mix 16	15G2:85G22 +1%Co
Mix 17	97.5G22:2.5Co
Mix 18	96G22:4Co
Mix 19	94.5G22:5.5Co
Mix 20	93G22:7Co
Mix 21	91.5G22:8.5Co
Mix 22	90G22:10Co
Mix 23	88.5G22:11.5Co

The powder mixes the for cobalt material were termed as mixes 16 to 23. Mix 16 is the working layer and is actually identical to mix 7. It can be seen that the gradient of cobalt starts at 1% on the working layer and then increases gradually to 11.5% at mix 23.

All mixes were prepared by the wet milling process using a Retsch PM400 industrial ball mill. The ratio of the WC balls to diamond powder was 3:1. The rotational speed was set to 130 rpm. The milling cycle time was 50 minutes. After milling, the mixed powders were sieved using a 125 micron mesh. For quality analysis, a sample of each diamond mix was taken for SEM examination and particle size analysis. The results are attached in Appendices A and B. The mixes were then converted into diamond films using the tape casting method.

Assembly:

Type 1 design variations as illustrated in Figure 3.13 were assembled by rolling the diamond films around a steel rod to create a radial configuration. Cylinders of the diamond films were then cut off the steel rod using a Stanley knife and placed onto the substrate. The tough core was created by placing a diamond webbing at the centre of the substrate. A webbing is a crushed diamond film to form fine granules. Type 2 design variations as illustrated in Figure 3.15 were made by punching 16mm diameter discs from a diamond film and stacking them onto the substrate. The Type 3 design variations presented in Figure 3.17 were made by cutting out pieces of the diamond films using a Stanley knife and stacking them onto the substrate in the chequered pattern.

Sintering:

Assembled specimens were put through a vacuum furnace heat treatment process set to 1100°C to remove volatile impurities. Another reason for heat treating the specimens was to partially graphitise the diamond surfaces. At the sintering temperature, it is believed that graphitised diamond particles dissolve in the cobalt solution and re-precipitate out as diamond, which facilitates diamond to diamond bonding [31].

The specimens were sintered using the Mk9+ press, a high-pressure, high-temperature synthesis system with an estimated pressure of 6.8 GPa. The sintered specimens were then ground to a nominal 12mm diameter. The finished specimens were partially leached to about 600μm depth in a proprietary mixture of boiling acids.

Scanning electron microscope (SEM) examination

It was important to verify the layered structures of the different cutter designs after sintering. To observe the layers of Type 1, 2 and 3 designs, the SEM technique was chosen.
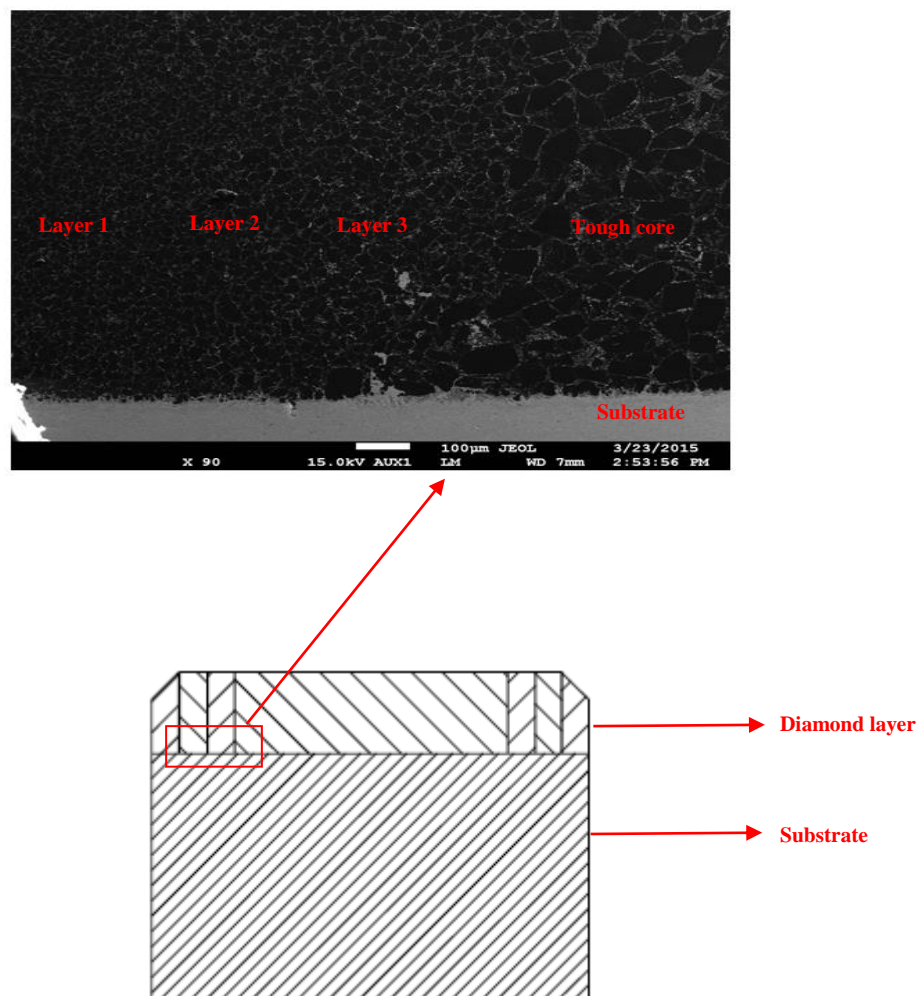
Type 1 design:

Figure 3.6 SEM microstructure of type 1 design at 90X magnification.

Figure 3.6 allows a radial increase in fracture toughness to be inferred due to the change in microstructure from layer 1 – 4 (tough core). In addition, it can be said that fabrication of type 1 specimens was successfully achieved because all the layers are visible in the microstructure and there is clear evidence of good integration and continuous transition between the layers.
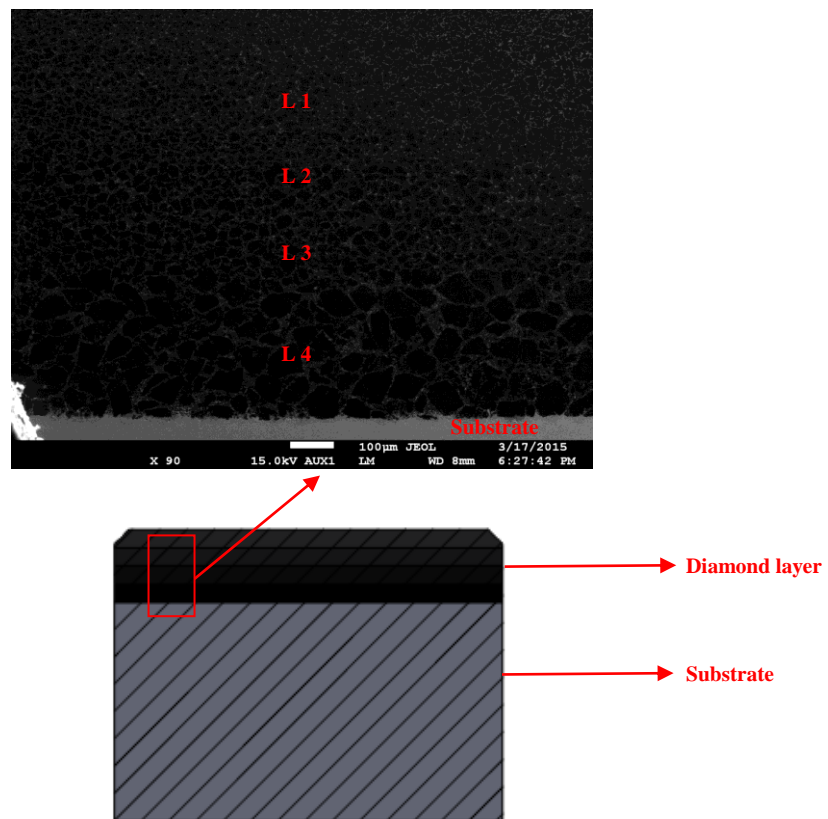
Type 2 design:

Figure 3.7 SEM microstructure of type 2 design at 90X magnification.

Type 2 variants were graded by size of the average grain size of the diamond grit in the axial direction as shown in figure 3.7. It is interesting to see that all the layers are visible

and well integrated in the microstructure. It might be inferred that fracture toughness increases axially from the surface layer (L1) to layer 4. Therefore, it can be said that type 2 variants were successfully made.
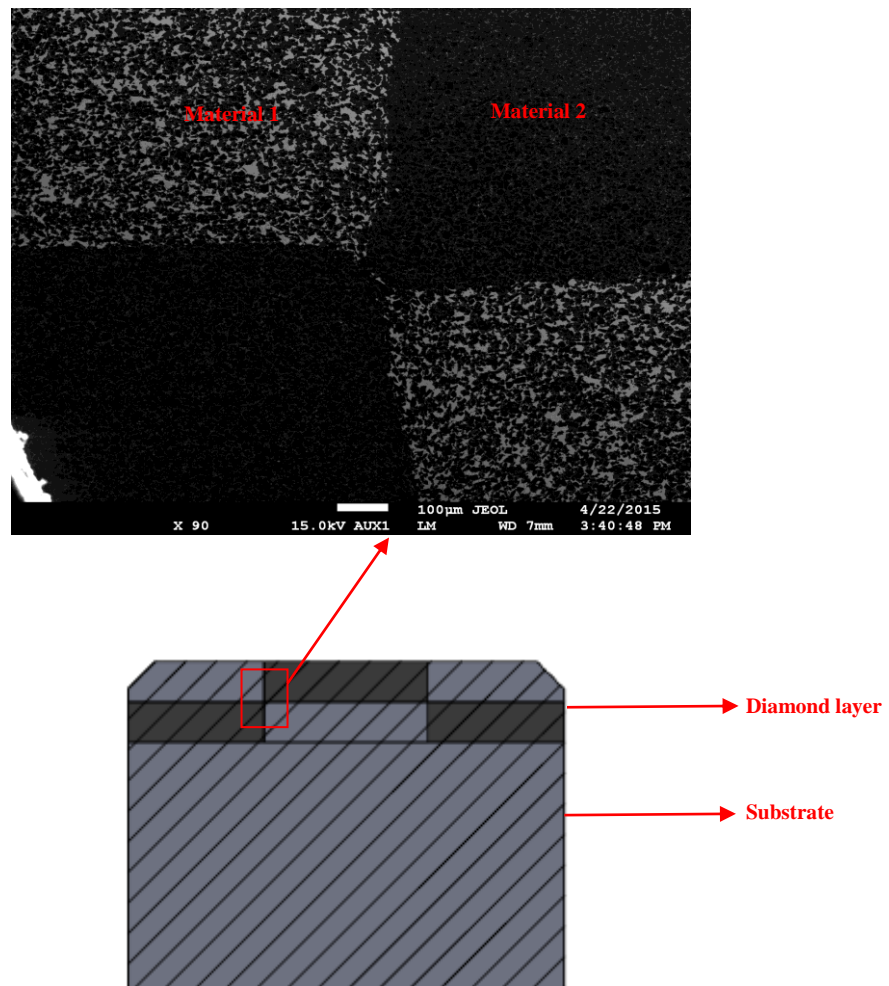
Type 3 design:

Figure 3.8 SEM microstructure of type 3 design at 90X magnification.

Type 3 variants were graded by using two distinct materials stacked in a chequered pattern as shown in figure 3.8. It was encouraging to see that the chequered pattern was

visible in the microstructure. It was also interesting to see that the layer pockets were not deformed. As a result, it can be said that type 3 variants were successfully made.

3.1.2 Testing of specimens

The rock cutting experiments were carried out using the VBX test as shown in Figure 3.9. Testing of the PDC specimens involved cutting a large granite block on a vertical turret lathe (VTL) machine.

Figure 3.9. VBX test set up.

Due to the cost constraints associated with leaching and VBX testing, of the 21 manufactured specimen types, only 4 variations could be tested. With overheads and depreciation inclusive, it costs about R11 000 to leach and test one specimen on the VBX. Each VBX test requires 3 specimens, so 63 specimens would need to be tested at

R693 000. Testing was therefore limited to what was realistically affordable. Variations 3 and 9 (type 1 design), variation 6 (a type 2 design) and variation 2 (a type 3 design) were selected and designated as variants A, B, C and D respectively. Each was then leached to 600µm depth. These configurations were chosen so that all 3 cutter design types could be tested. The other reason for choosing these configurations is that it was felt that these variants in particular stand a better chance of blocking the spall crack than their counterparts.

Prior to testing, each specimen was brazed into a tool holder at 10 degrees rake angle. The tool holder was then slid into the bracket of the lathe and screwed in. The bracket was lowered and positioned until the chamfer edge of the specimen touched the periphery of the rock. The orthogonal granite rock was rotated around its axis at 100 rpm. Cutting proceeded from the outside inwards at a depth of 0.5mm. Each such pass took approximately 25-30 minutes. Each progression was measured as a single run which is industrially known as a "pass". At the end of the pass, the specimen was washed and another progression was made. After every 30 passes, a specimen was removed from the machine and a picture of the wear scar was taken. The wear scar area was measured using the software of the Hawk measuring stereo microscope.The test was stopped immediately when the cutter failed prematurely by catastrophic fracture. The test was also ended when the wear scar length was equal to or greater than 5 mm.
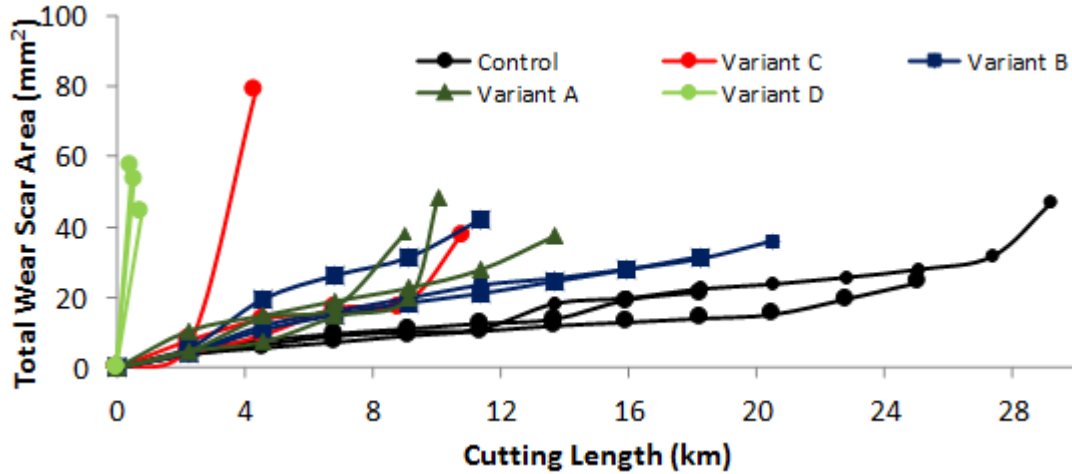
3.1.3 Results

Figure 3.10 Results of VBX test.

This section outlines the results of the VBX test. The full set of wear progression images for all samples are attached in Appendix C. These results are presented graphically in Figure 3.10. In this Figure, the layered variants are compared against control samples. The control in this context is a benchmark cutter whose VBX performance is outstanding. The results of the control shown in Figure 3.10 were taken from the database records.

Figure 3.10 plots the wear scar area against cutting length which was obtained from the number of passes experienced in the VBX test. One pass is equivalent to 0.076 km in cutting length or distance.

Figure 3.10 provides a graphical view of the cutter behaviour in relation to durability. In other words, it provides an indication of how long the cutter can cut through rock. This

information is important for developing new cutters by evaluating cutter performance relative to the benchmark.

Apart from the component of durability as being the resistance to cutter wear, resistance to spalling is another important component of durability. Spalling is seen by a sudden change in wear scar area which can be identified by a step increase in slope. For example the change in wear scar area of the 2 specimens of the control occurs at around 12km on average which means that at this point the benchmark can be said to begin to spall. After this point, the spall grows and the wear area becomes unsteady and unpredictable.

Table 3.7 Summary of VBX results.

	Test	Distance to spall (km)	Maximum cutting distance (km)	Test stopped due to:
Variant A	1	4.3	4.3	Catastrophic failure
	2	4.6	8.8	Allowable spall limit of 5mm exceeded
	3	9.0	10.8	Allowable spall limit of 5mm exceeded
	Average	6.0	8.0	
Variant B	1	4.6	20.5	Catastrophic failure
	2	4.6	11.0	Allowable spall limit of 5mm exceeded
	3	4.6	18.0	Catastrophic failure
	Average	4.6	16.5	
Variant C	1	6.8	10.0	Catastrophic failure
	2	2.3	13.7	Allowable spall limit of 5mm exceeded
	3	4.6	8.9	Allowable spall limit of 5mm exceeded
	Average	4.6	10.9	
Variant D	1	0.7	0.7	Catastrophic failure
	2	0.5	0.5	Catastrophic failure
	3	0.5	0.5	Catastrophic failure
	Average	0.6	0.6	

On average, spalling of variant A occurred at 6km and the maximum cutting length was 8km. These values are both less than the corresponding values of 12km and 20km in the control. So, it is clear that variant 6 does not show much promise.

A similar situation occurred with variant B which spalled at 4.6km and cut until 16.5km. Again, these parameters are both less than those of the control. As a result, variant B is also not a good cutter.

The distance at which spalling of Variant C occurred was consistent. All the cutters spalled at 4.6km. The maximum cutting length was 16.5km. Neither of these values match the performance of the control, however, So, Variant C is not a breakthrough cutter either.

The result of variant D was disappointing. All the specimens spalled prematurely and could not cut to even a kilometer. Variant D is the worst performer of the specimens tested.

3.1.4 Discussion

Based on the VBX test results, it is clear that none of the variants came close to matching the control. The results are disappointing and do not promise much in terms of improving on the control. This suggests that the approach of using layered structures to stop spalling did not work. There are four possibilities why this approach did not succeed in providing a solution to the spalling problem.

The first possible reason is that the fracture toughness of the interlayers might be too low to contain the spall crack. Miess et al. [32] studied the relationship between the fracture toughness of PCD and the starting grain size of diamond particles. They found that this relationship is exponential and is significant up to diameters of 30µm, beyond which the influence of grain size on the fracture toughness becomes insignificant. Therefore, in this work, it was expected that creating a gradual change in the microstructure by increasing the average grain size of the interlayers from layer 1 – 4 would affect the fracture toughness of the diamond layer. It was hoped that zones of fine diamond particles would have high wear resistance and very coarse diamond particles would possess high fracture toughness for blocking the spalling crack. However, the findings were not consistent with this hypothesis.
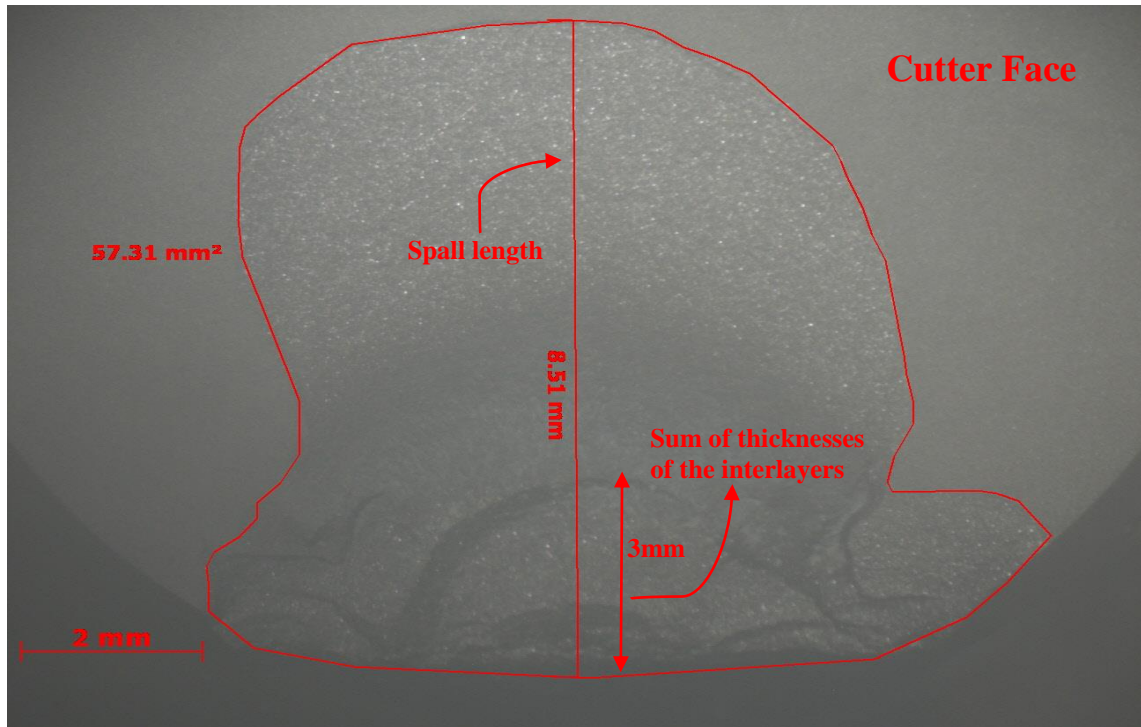
Figure 3.11 VBX test image of variant A at end of test (EOT).

For example, Figure 3.11 shows an image of variant A with the interlayer thickness of 3 mm. It can be seen in the image that the spall crack propagated past the region of the interlayers and was measured to be 8.5 mm in length. This indicates that the tough interlayers did not dampen the spall crack. The reason for this might be that although the fracture toughness of the interlayers on the cutter face is higher than that at the cutting surface, it is too low to contain spalling. Presumably an alternative material with even higher fracture toughness is required.

The second possible reason why the use of interlayers does not work is that the tough core is not used as a supporting layer despite having the potential to contain the spalling

crack. A supporting layer is a layer located adjacent to the cutting layer on the periphery of the cutter face. When the cutting layer wears out, the supporting layer usually becomes part of the wear scar depending on the thickness of the layers. In this work, the tough core is designed as a central core in the centre of the cutter face to optimize the volume of interlayers next to the cutting layer. It is not intended to block the spalling crack. Use of a tough core layer as a supporting layer adjacent to the cutting layer was not considered due to significant differences in grain size compositions between these two materials. In other words, it was thought that if a tough core layer was placed adjacent to the cutting layer, the large discrepancy in average grain size would result in a big step change of properties between the cutting layer and the interlayer. This could be problematic because these layers would take up different levels of cobalt binder from the substrate during the infiltration process which might result in sintering problems. However, the images from variant B show that the tough core might contain the spalling crack propagation.
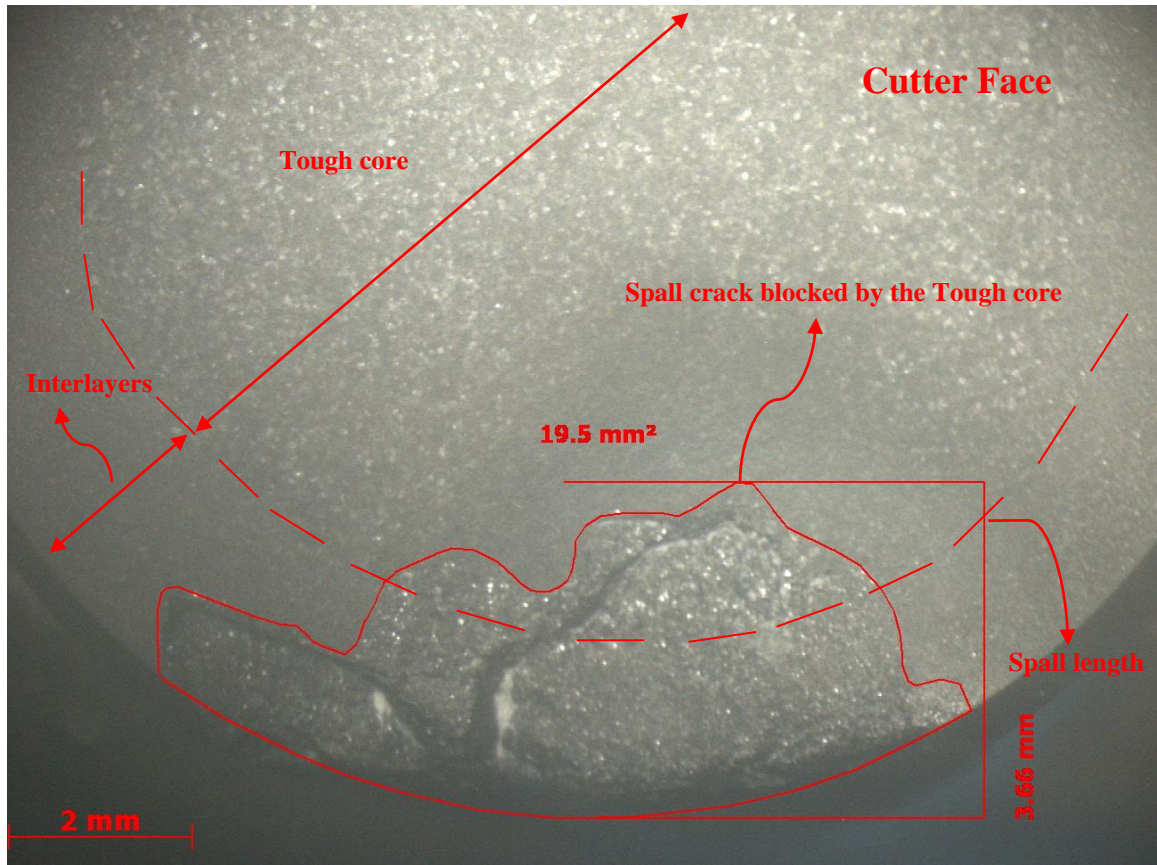
Figure 3.12 VBX test image of variant B at end of test (EOT).

For example, in Figure 3.12, the distinction between the working interlayers and the tough core can be seen clearly on the cutter face as indicated by the dashed line. The interlayers appear as a fine dark grey texture of 3 mm thickness. Meanwhile, the tough core appears as a coarse bright texture and is located at the centre of the cutter face. The tough core is composed of coarse diamond grit with the average grain size of 75 μm. The spalling crack was measured to be 3.7 mm which indicates that although the interlayers did not stop spalling, the tough core succeeded in blocking it. In other words, the crack had only travelled 0.7 mm through the tough core layer when it was dampened.

This result might suggest that the fracture toughness of the core might be high enough to stop the spalling crack. If this assumption is true, to stop a spalling crack, based on the composition of the tough core, a diamond layer of 75 µm average grain size might be suggested. Presumably, if the tough core was used as the supporting interlayer to the cutting layer, spalling might have been contained at the tough core layer. Despite this benefit, having a supporting layer of 75 µm average grain size might reduce the overall abrasion resistance on the wear front which is equally important for achieving good penetration rate during application. Therefore, although the use of tough core layer to stop spalling could be a potential solution, the consequential drawback is a serious concern. This is because PDC cutters with a diamond layer with an average grain size above 25 µm are known to be inferior products particularly in applications with harder rock formations [33].

The third possible reason why the use of interlayers does not work is that the stacking of the interlayers in the axial direction might be ineffective in reducing spalling. The direction in which the interlayers are stacked on the substrate seems to affect the ability of the diamond layer to stop spall cracks. The effectiveness of the diamond layer seems to depend on the thickness of the interlayers through which a crack propagates.
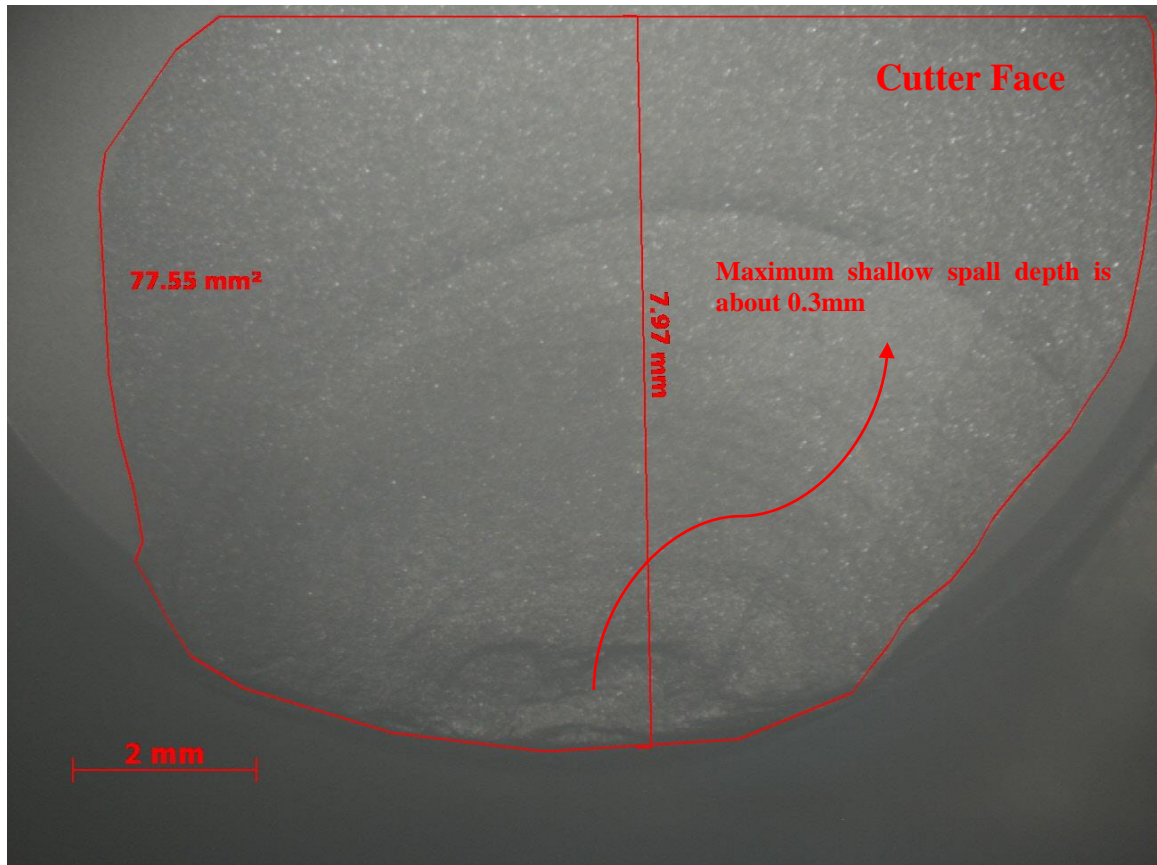
Figure 3.13 VBX test image of variant C at end of test (EOT).

For example, Figure 3.13 shows a cutter face image of variant C which was graded continuously by the average grain size of the diamond grit in the axial direction with the cutting layer located on the cutter face and fracture toughness increasing towards the substrate interface. From the image, it can be seen that the spall depth is shallow and was measured to be only 0.3 mm in depth which implies that the spall did not get a chance to reach the subsequent interlayers of higher fracture toughness because each stacked interlayer is only 1 mm in thickness. In addition, it also implies that the spalling crack only penetrated 0.3 mm into the cutter – meaning that the crack would have needed to

penetrate an additional 0.7 mm into the cutter depth before it ran into any material of higher fracture toughness in the other interlayers. This suggests that spalling occurred as normal, and the presence of underlying layers with higher fracture toughness played no role in the propagation of the crack. As a consequence, the presence of the additional interlayers became irrelevant.

The reason for this is likely to be that the thickness of each interlayer in this case seems to be too thick for the targeted shallow spalls. To engage the rest of the layers during a spall event, the thickness of each interlayer needs to be very thin. Presumably 100 µm would be a good starting point. However, thin interlayers could present problems during fabrication. During fabrication of cutters for sintering, very thin layers tend to crack during tape casting. In addition, thin layers are difficult to handle when stacking onto the substrates. So, although the use of very thin interlayers could work, this approach might pose manufacturing problems when it is implemented in practice.

The other possible reason why the use of interlayers does not work is that the catastrophic failures observed on the VBX might be caused by the new interfaces created in the diamond layer. The incorporation of tough material in the diamond layer resulted in the creation of new interfaces between the interlayers. Under loading, it is possible that these new interfaces have become weak areas for the separation of layers.
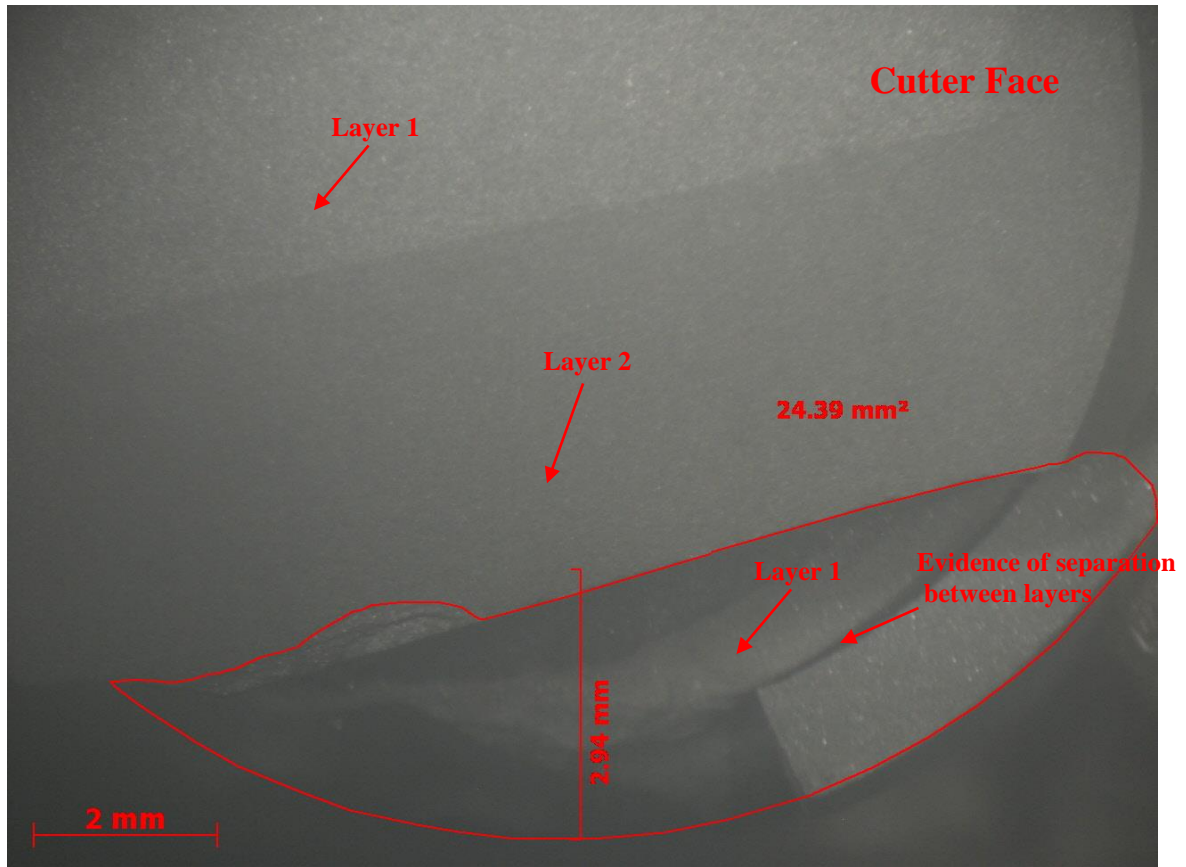
Figure 3.14 VBX test image of variant D at end of test (EOT).

For instance, Figure 3.14 shows an image of variant D which was designed with two wear fronts using two distinct layers stacked in a chequered pattern. It can be seen that layer 2 succeeded in stopping the spall propagation. But, although it has successfully stopped spalling, the mode of failure is undesirable. The manner in which Layer 1, which is adjacent to layer 2, failed is problematic. The entire piece of layer 1 separated from layer 2 and completely delaminated. In application, this type of failure mechanism is categorized as catastrophic failure. Catastrophic failures are not preferred wear

mechanisms because when delamination occurs, the cutting edge is compromised which results in bit failure [18].

This result suggests that the material of layer 2, which is composed of high levels of WC, has high enough fracture toughness to stop the spall crack. However, the drawback is that this layer appears not to sinter well with the working layer, i.e. layer 1, resulting in delamination problems.

Delamination might have been caused by a WC gradient in the layers which resulted in stress intensification along the layer interfaces [34]. Layer 2 contains 25% of WC and the working layer contains zero. Therefore, due to a possible sharp discontinuity in thermal properties, this WC gradient might be high enough to cause high stress concentrations between layers 1 and 2 resulting in separation problems. But, if this gradient is reduced by increasing the WC level in the working layer, this will reduce the potential chance of the design working because a high WC level in the working layer would make the overall diamond layer softer and so reduce its durability [34].

The use of WC layers to stop spalling seems to be working, but appears to have some fundamental problems that would prevent the implementation of this approach in practice. Presumably, an alternative material with higher fracture toughness similar to layer 2 is required. On the other hand, if the delamination problem could be resolved by other means, then this approach might become viable.

3.1.5 Conclusion

On the basis of the discussion points of this experimental investigation, the following conclusions were made:

• The selected tough interlayers do not have enough fracture toughness to stop spalling.

• The tough core layer might have the potential to stop spalling, but at the cost of reduced abrasion resistance.

• Axial stacking prevents the underlying layers from influencing the spall crack propagation.

• The incorporation of tough WC interlayers in the diamond layer can alter the failure mode of the PDC.

Overall, none of the variants tested performed as well as the control variant and so it appears that the use of layering has limited potential to eliminate the spalling problem.

3.2   Geometry approach

It was found that none of the materials used in this work have enough fracture toughness to stop the spalling cracks. Although the use of layering could still be pursued by the incorporation of a very high fracture toughness interlayer into the cutter, it was realized that other ways to stop the spalling were possible. One of the ways was to approach the problem from the shaped cutter perspective. This section explores the use of a depression on the cutter face to stop spalling as shown in Figure 3.15. It was thought that the spalling problem could be addressed by the removal of material through which a spalling crack could propagate. This could be achieved by incorporating a depression on the front face of the cutter. Using this approach, the crack could only propagate over a short distance before stopping when it reached the depression.
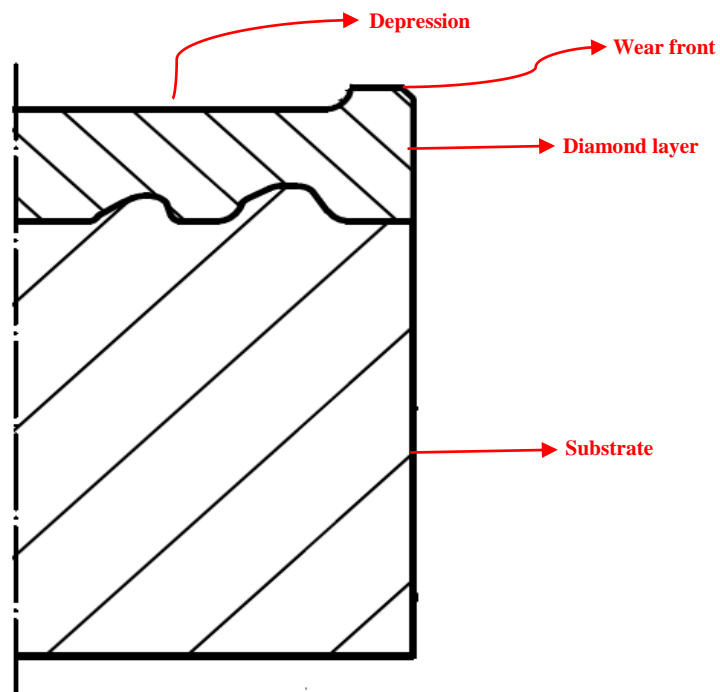
Figure 3.15 Schematic of the PDC cutter.

3.2.1 Fabrication of specimens

A spark erosion machine was used to create depression features on three standard cutters. Two variations were created. The first variation was created by taking all three cutters and created a concentric hollow which was 13.4mm in diameter and 0.5mm in depth. This resulted in a depression that was located 1mm from the chamfer edge. The second variation was then created by taking one cutter and added an extra small concentric hollow at the centre of the depression. The extra small hollow was 8.0mm in diameter and 0.5mm in depth. The reason for incorporating the extra feature was to increase the barrier to the spall crack. It was thought that if the spall crack penetrated into the first feature, it would be blocked by the second feature. The sketches below show the pictorial view of the concepts.
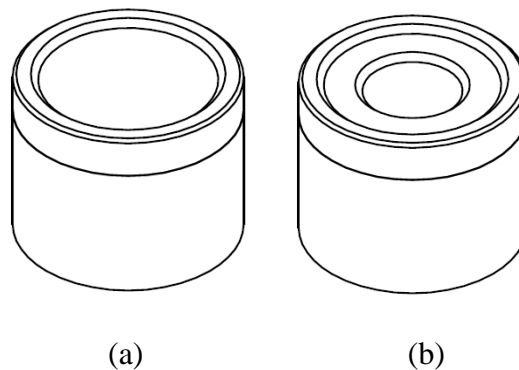

(a)   (b)

Figure 3.16 Geometric concepts fabricated using a spark erosion machine, (a) first variation and (b) second variation.

3.2.2 Testing of specimens

All 3 specimens with geometric surface features which were fabricated using a spark erosion machine were tested on the VBX. Each was then leached to 600μm depth as shown in Figure 3.17. The background about VXB test is covered in 3.1.2.
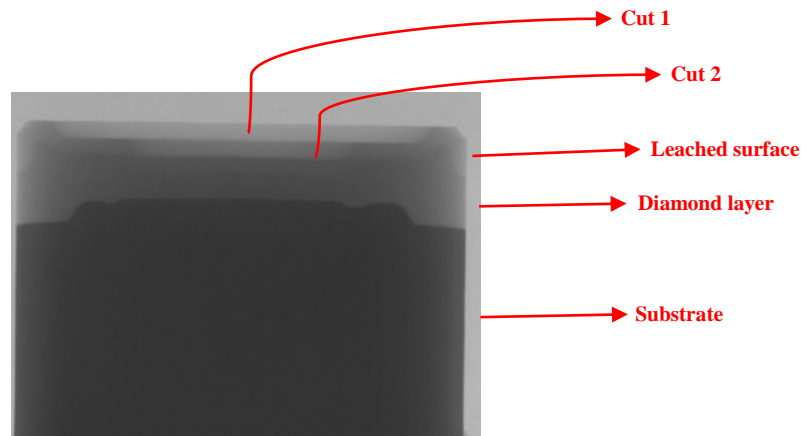

Figure 3.17 X-ray image of a leached specimen of the second variation.
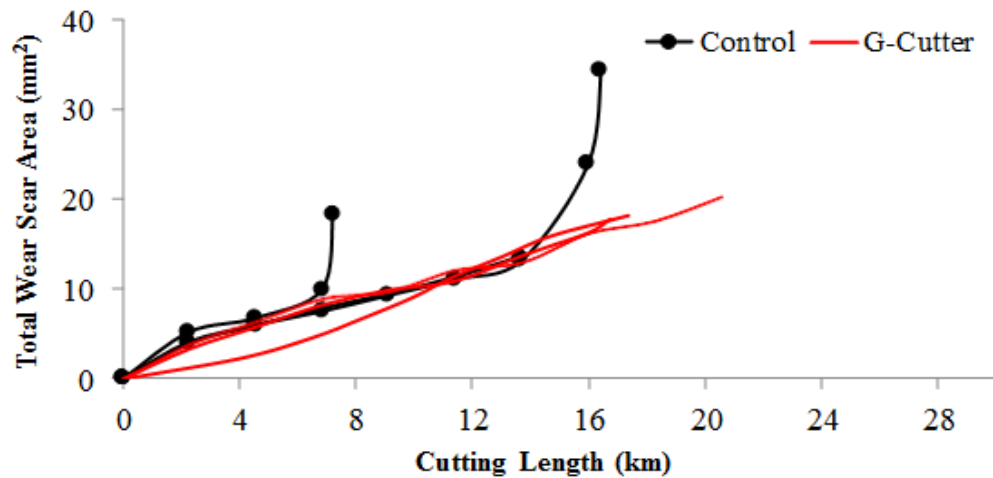
3.2.3 Results

Figure 3.18 Results of VBX test.

This section outlines the results of the geometric variant (G-cutter). These results are presented graphically in Figure 3.18 where the G-cutter is compared against control samples. The control is a benchmark cutter whose composition is identical to the G-cutter. The depression is the only feature that distinguishes the control from the G-cutter, the rest is identical. The results of the control cutter shown in Figure 3.18 were taken from the database records.

On average, the G-cutter seems to be more consistent than the control. This suggests that spalling in G-cutter specimens was stopped at a length of 1 mm which implies a successful blocking of the spall crack by the depression feature on the cutter face.

Table 3.8 Summary of VBX results.

	Test	Distance to spall (km)	Maximum cutting distance (km)	Test stopped due to:
G-cutter	1	6.8	20.5	Allowable spall limit of 1mm
	2	6.8	16.7	Allowable spall limit of 1mm
	3	4.6	18.2	Allowable spall limit of 1mm
	Average	**6.1**	**18.5**	

On average, the maximum cutting length of the G-cutter was 18.5km. This value is more than the corresponding value of 12.3km in the control. It is therefore clear that the G-cutter show much promise. The boost in overall durability on the G-cutter over the control appears to come from the consistency of the results. This consistency could be attributed to the successful spall crack confinement by the depression.

3.2.4 Discussion

Based on the VBX test results, it is clear that the G-cutter has less scatter and cuts longer than the control and so the results are promising in terms of developing a new cutter. This suggests that the approach of using a depression on the front face of the cutter to stop spalling is effective.
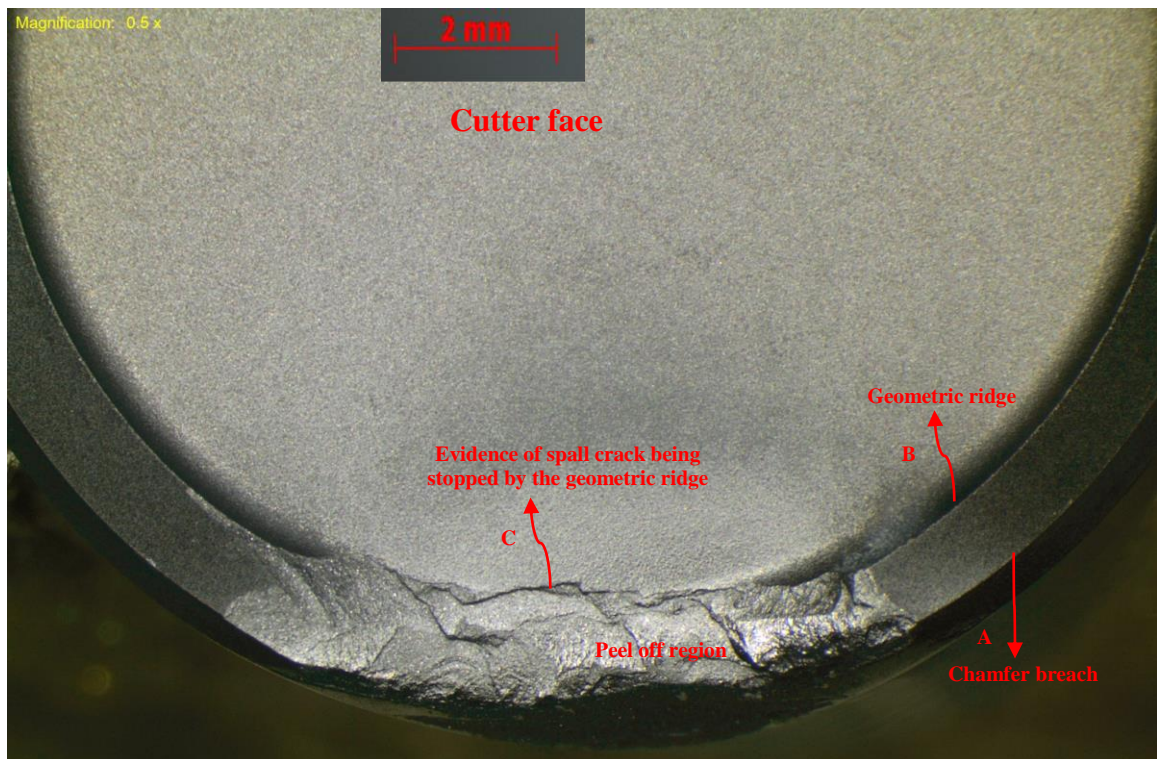

Figure 3.19 VBX test image of the G-cutter first variation at end of test (EOT).

As an illustration of this, Figure 3.19 shows the face of the G-cutter after VBX testing. It can be seen that spalling started at the chamfer breach (A). It propagated until point (B), where it was contained by the depression. The evidence of blocking is indicated by point C which suggests the effectiveness of the depression in blocking spalling. The allowable

spall limit, which is the distance between the chamfer breach and the depression, was selected to be 1 mm. It appears that limiting the spall crack to 1 mm is a reasonable approach because putting a depression much closer to the chamfer breach could potentially cause cutting to occur inside the depression in addition to the usual cutting at the chamfer breach. This might interfere with the integrity of the cutting front. A shorter distance between the chamfer breach and the depression would presumably also reduce the structural integrity of the remaining rim around the cutter.

The assumed reason that this approach works is the existence of the depression on the front face which was selected to be 0.5 mm deep as the starting point. So, under loading, when the spall crack reaches the edge of the depression, it gets blocked because there is no material to propagate through.

This result suggests that whilst this approach does not completely prevent spalling from occurring, it has two useful benefits. The first benefit is that the cutter damage is contained, and so the cutter can be rotated and used more than once during application which would enhance the cutter value.

The second benefit is that by confining spalling, the cutter can cut further in VBX testing than the benchmark. Spalling makes the cutter blunt which renders it unusable for further cutting. So, the delayed spalling through the use of a depression means that the cutter remains sharp, thereby allowing it to cut for a longer distance. In this instance, the G-cutter was able to cut for a distance of 34% greater than the control cutter.

Although this approach seems to be a solution to the spalling problem, it only demonstrates the prospect associated with this concept. Further optimization of this

solution should be explored in field testing. Therefore, the shaped PDC design in this investigation provides a useful basis for the future development. In addition, the fabrication method which was used to create the depression on the cutter is not cost-effective for implementation. Alternative methods such as near-net size should be investigated.

3.2.5   Conclusion

On the basis of the discussion of this experimental investigation, the following conclusion can be drawn:

- The use of a depression on the front face of a cutter has potential to reduce problems associated with spalling.

CHAPTER 4

Overall Discussion

On the basis of the findings of the two approaches, the following discussion points were covered:

The use of layered structures and a novel geometry were the two approaches used in the attempt to resolve the spalling problem. It was found that the new geometry worked, but the layered approach did not succeed. This finding suggests that the dominating factor to spalling behaviour seems to be geometric rather than compositional within the limited range of material tested on the VBX.

The fundamental issue associated with the composition of the material tested might be a lack of sufficient fracture toughness. Although other factors such as stacking orientation and grading type could affect spalling, fracture toughness is thought to be the primary factor.

Better materials in terms of fracture toughness over what was tested may exist, but they must still meet the wear and thermal properties which might not be possible to find. For example, a tough core layer is found to be effective in containing spalling. Its potential is associated with its average grain size of 75µm which is very coarse for a typical PDC cutter. A PDC cutter with this material will wear quickly on the VBX and thus be deemed as inferior. So, although the tough core layer is a potential material for stopping spalling, it does not meet the wear requirements.

Alternative materials to achieve good fracture toughness other than increasing the average grain size of the diamond grit might be possible to find. The challenge though, might be the difficulty in sintering these materials which would prevent easy implementation of the concepts. For example, high WC content material was found to be successful in blocking the spall propagation, but the material did not sinter possibly due to residual stresses which resulted in delamination problems during testing on the VBX.

On the other hand, the geometry approach seems to be favoured due the nature of the depression design. The depression was conceptualized under the assumption that the spalling crack can only propagate through the PCD cutter if material exists for it to propagate through. It was found that the depression was effective in this regard in that in the VBX tests, it blocked the spalling crack from propagating. This finding suggests that a shaped PDC is a likely solution to the spalling problem. The simplicity of the depression design makes it easy to implement in practice. Near-net size might be the favourable approach for mass production. If this concept can be manufactured at a reasonable cost, the concept could be commercially viable because tests results indicate that the concept cutter can cut for a greater distance with reduced variability than the standard cutter. In addition, the concept cutter can also be reused.

CHAPTER 5

Overall conclusions

On the basis of the discussion points of the two approaches, the following overall conclusions were made:

- The use of layered structures was not effective in resolving the spalling problem. As a result, a layered PDC is not a solution to the spalling problem.

- The use of a geometric depression on the front face of the cutter successfully stopped spalling. Therefore, a shaped PDC is a likely solution to the spalling problem.

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APPENDIX A

Particle size analysis results of diamond mixes

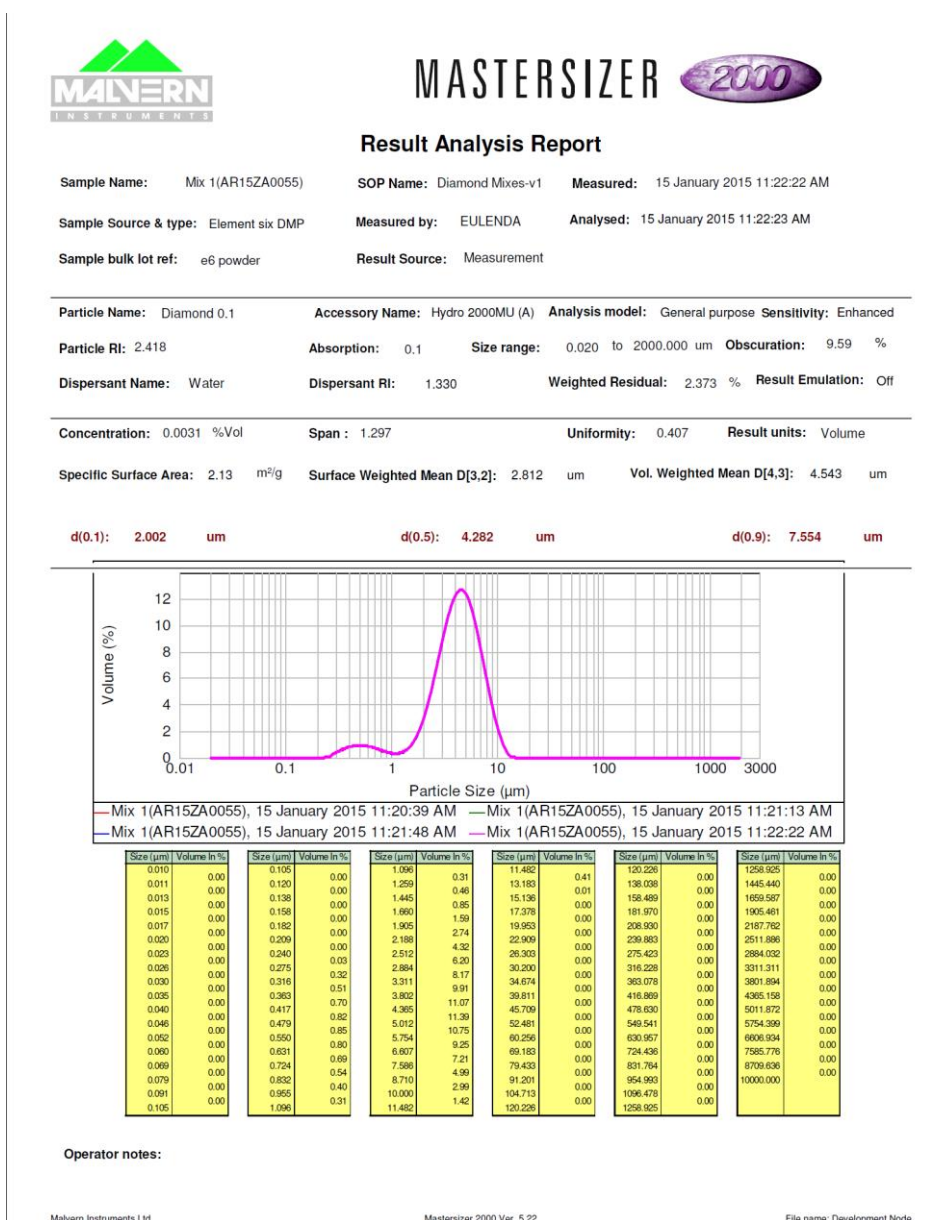This appendix contains particle size analysis results of mix 1 to mix 23.

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 1(AR15ZA0055)	**SOP Name:** Diamond Mixes-v1	**Measured:** 15 January 2015 11:22:22 AM	
Sample Source & type: Element six DMP	**Measured by:** EULENDA	**Analysed:** 15 January 2015 11:22:23 AM	
Sample bulk lot ref: e6 powder	**Result Source:** Measurement		

Particle Name: Diamond 0.1	**Accessory Name:** Hydro 2000MU (A)	**Analysis model:** General purpose	**Sensitivity:** Enhanced
Particle RI: 2.418	**Absorption:** 0.1	**Size range:** 0.020 to 2000.000 um	**Obscuration:** 9.59 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 2.373 %	**Result Emulation:** Off

Concentration: 0.0031 %Vol	**Span:** 1.297	**Uniformity:** 0.407	**Result units:** Volume
Specific Surface Area: 2.13 m²/g	**Surface Weighted Mean D[3,2]:** 2.812 um	**Vol. Weighted Mean D[4,3]:** 4.543 um	

d(0.1): 2.002 um   **d(0.5):** 4.282 um   **d(0.9):** 7.554 um

Mix 1(AR15ZA0055), 15 January 2015 11:20:39 AM — Mix 1(AR15ZA0055), 15 January 2015 11:21:13 AM
Mix 1(AR15ZA0055), 15 January 2015 11:21:48 AM — Mix 1(AR15ZA0055), 15 January 2015 11:22:22 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.31	11.482	0.41	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.46	13.183	0.01	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	0.85	15.136	0.00	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.59	17.378	0.00	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	2.74	19.953	0.00	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	4.32	22.909	0.00	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.03	2.512	6.20	26.303	0.00	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.32	2.884	8.17	30.200	0.00	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.51	3.311	9.91	34.674	0.00	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.70	3.802	11.07	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.82	4.365	11.39	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.85	5.012	10.75	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.80	5.754	9.25	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.69	6.607	7.21	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.54	7.586	4.99	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.40	8.710	2.99	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	0.31	10.000	1.42	104.713	0.00	1096.478	0.00		
0.105	0.00	1.096	0.31	11.482		120.226	0.00	1258.925	0.00		

Operator notes:

Malvern Instruments Ltd.   Mastersizer 2000 Ver. 5.22   File name: Development Node

Figure A.1 Particle size analysis of mix 1

MASTERSIZER 2000

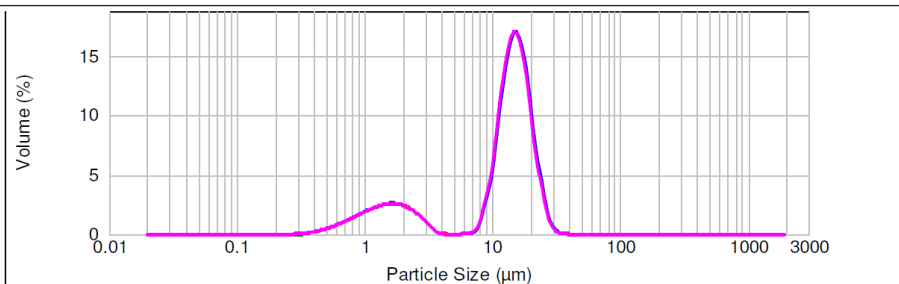### Result Analysis Report

Sample Name: Mix 2(AR15ZA0055)   **SOP Name:** Diamond Mixes-v1   **Measured:** 15 January 2015 11:30:37 AM

Sample Source & type: Element six DMP   **Measured by:** EULENDA   **Analysed:** 15 January 2015 11:30:38 AM

Sample bulk lot ref: e6 powders   **Result Source:** Measurement

---

Particle Name: Diamond 0.1   **Accessory Name:** Hydro 2000MU (A)   **Analysis model:** General purpose   **Sensitivity:** Enhanced

Particle RI: 2.418   **Absorption:** 0.1   **Size range:** 0.020 to 2000.000 um   **Obscuration:** 9.32 %

Dispersant Name: Water   **Dispersant RI:** 1.330   **Weighted Residual:** 1.052 %   **Result Emulation:** Off

---

Concentration: 0.0047 %Vol   **Span:** 1.337   **Uniformity:** 0.354   **Result units:** Volume

Specific Surface Area: 1.48 m²/g   **Surface Weighted Mean D[3,2]:** 4.056 um   **Vol. Weighted Mean D[4,3]:** 8.575 um

d(0.1): 1.326 um   **d(0.5):** 9.052 um   **d(0.9):** 13.427 um
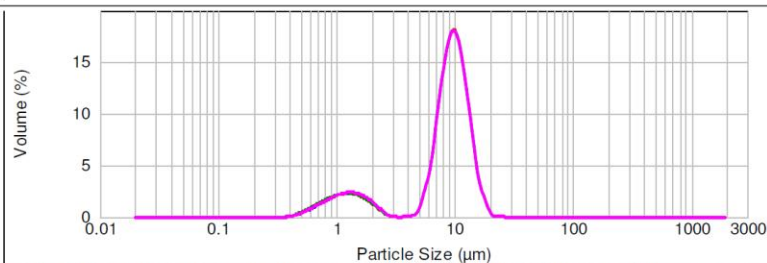
— Mix 2(AR15ZA0055), 15 January 2015 11:28:53 AM   — Mix 2(AR15ZA0055), 15 January 2015 11:29:28 AM
— Mix 2(AR15ZA0055), 15 January 2015 11:30:02 AM   — Mix 2(AR15ZA0055), 15 January 2015 11:30:37 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	2.11	11.482	11.72	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	2.17	13.183	7.13	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.05	15.136	2.88	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.75	17.378	1.09	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	1.29	19.953	0.10	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.73	22.909	0.00	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.22	26.303	0.00	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.01	30.200	0.00	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.00	34.674	0.00	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.00	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.05	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.17	5.012	0.29	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.38	5.754	1.77	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.67	6.607	4.08	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.98	7.586	8.85	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.32	8.710	13.28	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	1.64	10.000	16.01	104.713	0.00	1096.478	0.00		
0.105	0.00	1.096	1.92	11.482	15.37	120.226	0.00	1258.925	0.00		

Operator notes:

Figure A.2 Particle size analysis of mix 2

Figure A.3 Particle size analysis of mix 3

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 4(AR15ZA0055) **SOP Name:** Diamond Mixes-v1 **Measured:** 15 January 2015 11:47:46 AM

Sample Source & type: Element six DMP **Measured by:** EULENDA **Analysed:** 15 January 2015 11:47:47 AM

Sample bulk lot ref: e6 powders **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 9.38 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 0.911 % **Result Emulation:** Off

Concentration: 0.0054 %Vol **Span :** 1.396 **Uniformity:** 0.384 **Result units:** Volume

Specific Surface Area: 1.23 m²/g **Surface Weighted Mean D[3,2]:** 4.888 um **Vol. Weighted Mean D[4,3]:** 19.620 um

d(0.1): 1.433 um **d(0.5):** 21.550 um **d(0.9):** 31.513 um
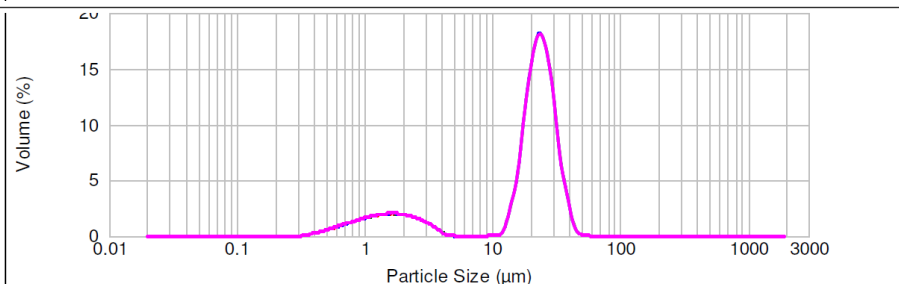
Mix 4(AR15ZA0055), 15 January 2015 11:46:02 AM — Mix 4(AR15ZA0055), 15 January 2015 11:46:36 AM
Mix 4(AR15ZA0055), 15 January 2015 11:47:11 AM — Mix 4(AR15ZA0055), 15 January 2015 11:47:46 AM

Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.010	0.00	0.105	0.00	1.096	1.66	11.482	0.53	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	1.77	13.183	2.83	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	1.83	15.136	6.10	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.82	17.378	11.66	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	1.75	19.953	15.45	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	1.60	22.909	16.20	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.38	26.303	13.44	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.08	30.200	7.95	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.01	3.311	0.72	34.674	4.03	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.21	3.802	0.27	39.811	1.05	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.36	4.365	0.01	45.709	0.14	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.52	5.012	0.00	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.72	5.754	0.00	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.92	6.607	0.00	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	1.13	7.586	0.00	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.32	8.710	0.00	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	1.50	10.000	0.00	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482	0.04	120.226	0.00	1258.925			

Operator notes:

Malvern Instruments Ltd.
Malvern, UK

Mastersizer 2000 Ver. 5.22
Serial Number : 34264-20

File name: Development Node
Record Number: 3000

Figure A.4 Particle size analysis of mix 4

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 5(AR15ZA0055) **SOP Name:** Diamond Mixes-v1 **Measured:** 15 January 2015 11:56:11 AM

Sample Source & type: Element six DMP **Measured by:** EULENDA **Analysed:** 15 January 2015 11:56:12 AM

Sample bulk lot ref: e6 powder **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 9.67 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 0.817 % **Result Emulation:** Off

Concentration: 0.0050 %Vol **Span :** 1.441 **Uniformity:** 0.426 **Result units:** Volume

Specific Surface Area: 1.37 m²/g **Surface Weighted Mean D[3,2]:** 4.376 um **Vol. Weighted Mean D[4,3]:** 26.006 um

d(0.1): 1.214 um **d(0.5):** 29.550 um **d(0.9):** 43.783 um
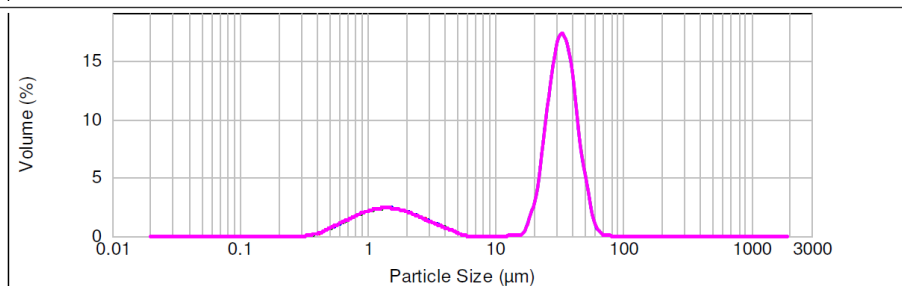
Mix 5(AR15ZA0055), 15 January 2015 11:54:27 AM — Mix 5(AR15ZA0055), 15 January 2015 11:55:02 AM
Mix 5(AR15ZA0055), 15 January 2015 11:55:36 AM — Mix 5(AR15ZA0055), 15 January 2015 11:56:11 AM

Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.010	0.00	0.105	0.00	1.096	2.11	11.482	0.00	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	2.18	13.183	0.00	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.16	15.136	0.01	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.05	17.378	0.12	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	1.87	19.953	1.53	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	1.64	22.909	3.74	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.38	26.303	8.77	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.12	30.200	13.20	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.01	3.311	0.87	34.674	15.63	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.15	3.802	0.63	39.811	14.42	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.37	4.365	0.40	45.709	10.01	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.62	5.012	0.12	52.481	5.31	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.91	5.754	0.00	60.256	1.94	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	1.20	6.607	0.00	69.183	0.34	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	1.49	7.586	0.00	79.433	0.01	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.75	8.710	0.00	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	1.96	10.000	0.00	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Operator notes:

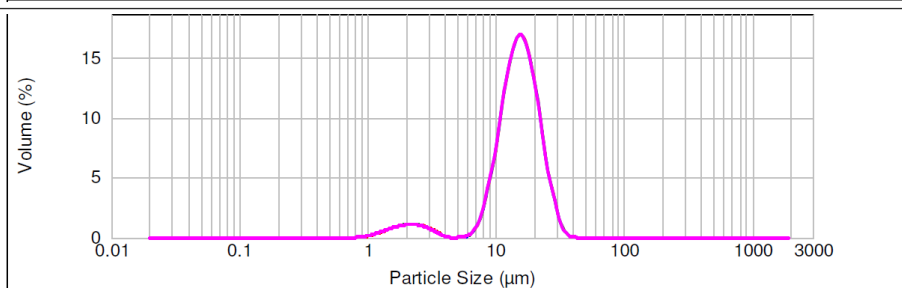Figure A.5 Particle size analysis of mix 5

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 6(AR15ZA0160) **SOP Name:** Diamond Mixes-v1 **Measured:** 27 January 2015 10:14:51 AM

Sample Source & type: Element six DMP **Measured by:** Khosi **Analysed:** 27 January 2015 10:14:52 AM

Sample bulk lot ref: Mix 6 **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 8.49 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 0.894 % **Result Emulation:** Off

---

Concentration: 0.0055 %Vol **Span :** 1.515 **Uniformity:** 0.442 **Result units:** Volume

Specific Surface Area: 1.09 m²/g **Surface Weighted Mean D[3,2]:** 5.508 um **Vol. Weighted Mean D[4,3]:** 72.458 um

d(0.1): 1.442 um **d(0.5):** 80.963 um **d(0.9):** 124.087 um

---
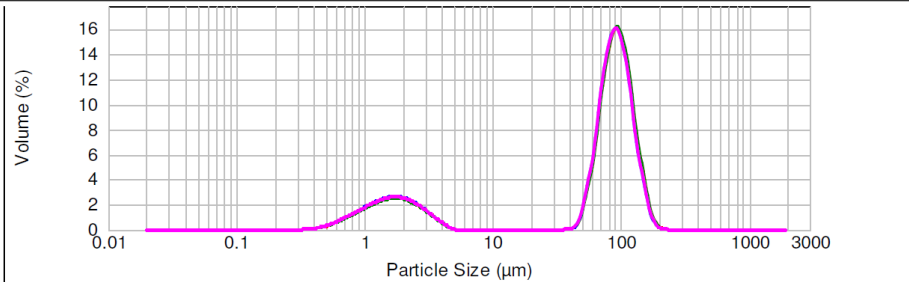
Mix 6(AR15ZA0160), 27 January 2015 10:13:08 AM — Mix 6(AR15ZA0160), 27 January 2015 10:13:42 AM
Mix 6(AR15ZA0160), 27 January 2015 10:14:17 AM — Mix 6(AR15ZA0160), 27 January 2015 10:14:51 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	1.94	11.482	0.00	120.226	6.89	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	2.17	13.183	0.00	138.038	3.81	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.32	15.136	0.00	158.489	1.15	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.37	17.378	0.00	181.970	0.21	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	2.28	19.953	0.00	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	2.06	22.909	0.00	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.73	26.303	0.00	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.32	30.200	0.00	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.88	34.674	0.00	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.49	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.06	4.365	0.12	45.709	0.16	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.17	5.012	0.00	52.481	1.23	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.33	5.754	0.00	60.256	3.90	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.53	6.607	0.00	69.183	7.43	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.78	7.586	0.00	79.433	11.80	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.05	8.710	0.00	91.201	14.26	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	1.35	10.000	0.00	104.713	14.07	1096.478	0.00		
0.105		1.096	1.66	11.482		120.226	11.47	1258.925			

Operator notes:

Malvern Instruments Ltd.
Malvern, UK

Mastersizer 2000 Ver. 5.22
Serial Number : 34264-20

File name: Development Node
Record Number: 3040

Figure A.6 Particle size analysis of mix 6

Figure A.8 Particle size analysis of mix 8

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 9(AR15ZA0160)	**SOP Name:** Diamond Mixes-v1	**Measured:** 27 January 2015 10:30:55 AM
Sample Source & type: Element six DMP	**Measured by:** Khosi	**Analysed:** 27 January 2015 10:30:56 AM
Sample bulk lot ref: Mix 9	**Result Source:** Measurement	

Particle Name: Diamond 0.1   **Accessory Name:** Hydro 2000MU (A)   **Analysis model:** General purpose   **Sensitivity:** Enhanced

Particle RI: 2.418   **Absorption:** 0.1   **Size range:** 0.020 to 2000.000 um   **Obscuration:** 9.37 %

Dispersant Name: Water   **Dispersant RI:** 1.330   **Weighted Residual:** 0.963 %   **Result Emulation:** Off

Concentration: 0.0100 %Vol   **Span :** 1.524   **Uniformity:** 0.451   **Result units:** Volume

Specific Surface Area: 0.75 m²/g   **Surface Weighted Mean D[3,2]:** 7.999 um   **Vol. Weighted Mean D[4,3]:** 13.719 um

d(0.1): 3.895 um   **d(0.5):** 13.047 um   **d(0.9):** 23.779 um
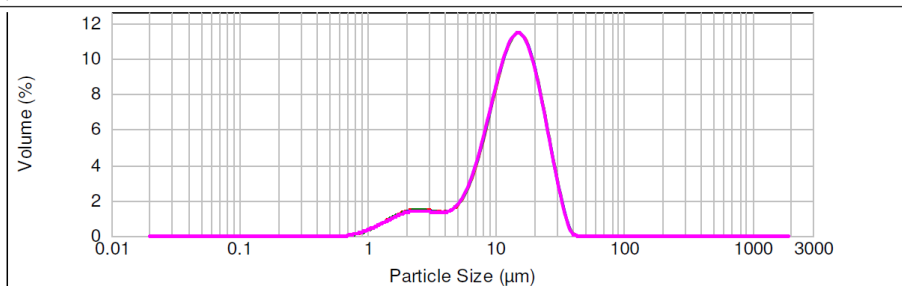
Mix 9(AR15ZA0160), 27 January 2015 10:29:12 AM — Mix 9(AR15ZA0160), 27 January 2015 10:29:46 AM
Mix 9(AR15ZA0160), 27 January 2015 10:30:21 AM — Mix 9(AR15ZA0160), 27 January 2015 10:30:55 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.49	11.482	9.61	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.69	13.183	10.27	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	0.89	15.136	10.18	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.06	17.378	9.29	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	1.19	19.953	7.75	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	1.26	22.909	5.78	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.26	26.303	3.72	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.22	30.200	1.84	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	1.17	34.674	0.42	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	1.20	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	1.40	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	1.85	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	2.64	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	3.78	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	5.24	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.07	8.710	6.83	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	0.17	10.000	8.40	104.713	0.00	1096.478	0.00		
0.105		1.096	0.32	11.482		120.226	0.00	1258.925			

Operator notes:

Malvern Instruments Ltd.
Malvern, UK

Mastersizer 2000 Ver. 5.22
Serial Number : 34264-20

File name: Development Node
Record Number: 3048

Figure A.9 Particle size analysis of mix 9

Figure A.10 Particle size analysis of mix 10

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 11(AR154ZA0160)	**SOP Name:** Diamond Mixes-v1	**Measured:** 27 January 2015 10:46:03 AM
Sample Source & type: Element six DMP	**Measured by:** Khosi	**Analysed:** 27 January 2015 10:46:04 AM
Sample bulk lot ref: Mix 11	**Result Source:** Measurement	

Particle Name: Diamond 0.1	**Accessory Name:** Hydro 2000MU (A)	**Analysis model:** General purpose **Sensitivity:** Enhanced
Particle RI: 2.418	**Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um	**Obscuration:** 9.13 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 1.477 % **Result Emulation:** Off

Concentration: 0.0075 %Vol	**Span :** 1.721	**Uniformity:** 0.52 **Result units:** Volume
Specific Surface Area: 0.964 m²/g	**Surface Weighted Mean D[3,2]:** 6.226 um	**Vol. Weighted Mean D[4,3]:** 11.214 um

d(0.1): 2.729 um   **d(0.5):** 10.388 um   **d(0.9):** 20.604 um

— Mix 11(AR154ZA0160), 27 January 2015 10:44:19 AM
— Mix 11(AR154ZA0160), 27 January 2015 10:44:54 AM
— Mix 11(AR154ZA0160), 27 January 2015 10:45:28 AM
— Mix 11(AR154ZA0160), 27 January 2015 10:46:03 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010		0.105	0.00	1.096	0.74	11.482	8.91	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.99	13.183	8.74	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	1.23	15.136	7.99	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.46	17.378	6.75	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	1.64	19.953	5.20	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	1.77	22.909	3.51	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.87	26.303	2.04	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.97	30.200	0.61	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	2.11	34.674	0.00	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	2.37	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	2.81	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	3.48	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	4.39	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	5.47	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	6.65	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.17	8.710	7.72	91.201	0.00	954.993	0.00	10000.000	
0.091	0.00	0.955	0.32	10.000	8.54	104.713	0.00	1096.478	0.00		
0.105		1.096	0.51	11.482		120.226	0.00	1258.925	0.00		

Operator notes:

Figure A.11 Particle size analysis of mix 11
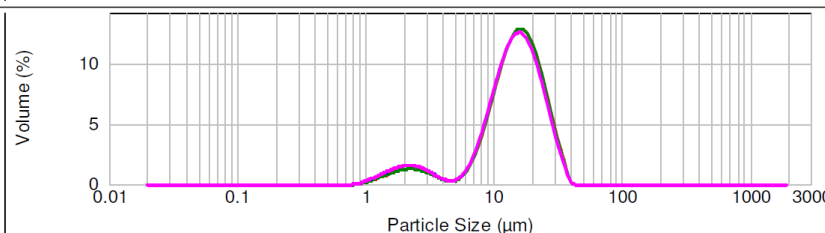
MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 12 (AR15ZA0253) **SOP Name:** Diamond Mixes-v1 **Measured:** 10 February 2015 02:58:50 PM

Sample Source & type: Element six DMP **Measured by:** Khosi **Analysed:** 10 February 2015 02:58:51 PM

Sample bulk lot ref: Mix 12 **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 9.09 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 2.198 % **Result Emulation:** Off

Concentration: 0.0037 %Vol **Span :** 2.161 **Uniformity:** 0.67 **Result units:** Volume

Specific Surface Area: 1.77 m²/g **Surface Weighted Mean D[3,2]:** 3.392 um **Vol. Weighted Mean D[4,3]:** 7.142 um

d(0.1): 1.684 um   **d(0.5):** 5.925 um   **d(0.9):** 14.487 um
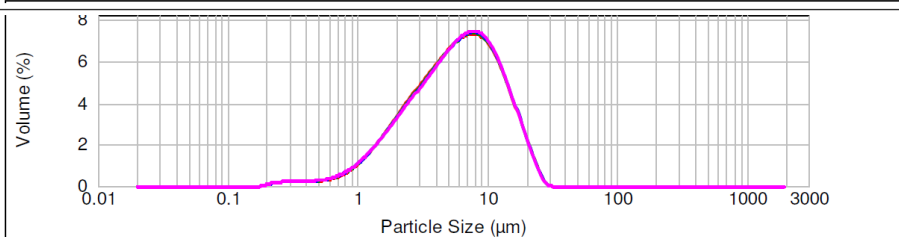
—Mix 12 (AR15ZA0253), 10 February 2015 02:57:07 PM
—Mix 12 (AR15ZA0253), 10 February 2015 02:57:41 PM
—Mix 12 (AR15ZA0253), 10 February 2015 02:58:16 PM
—Mix 12 (AR15ZA0253), 10 February 2015 02:58:50 PM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	1.34	11.482	5.50	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	1.71	13.183	4.63	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.10	15.136	3.63	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.53	17.378	2.58	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.05	1.905	2.97	19.953	1.59	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.13	2.188	3.42	22.909	0.72	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.18	2.512	3.87	26.303	0.11	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.20	2.884	4.32	30.200	0.00	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.22	3.311	4.78	34.674	0.00	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.22	3.802	5.23	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.23	4.365	5.68	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.26	5.012	6.10	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.32	5.754	6.44	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.42	6.607	6.67	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.57	7.586	6.73	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.77	8.710	6.57	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	1.03	10.000	6.15	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482		120.226	0.00	1258.925	0.00		

Operator notes:

Figure A.12 Particle size analysis of mix 12

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 13 (AR15ZA0253) **SOP Name:** Diamond Mixes-v1 **Measured:** 10 February 2015 03:08:42 PM

Sample Source & type: Element six DMP **Measured by:** Khosi **Analysed:** 10 February 2015 03:08:43 PM

Sample bulk lot ref: Mix 13 **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 8.59 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 1.258 % **Result Emulation:** Off

---

Concentration: 0.0043 %Vol **Span :** 1.849 **Uniformity:** 0.574 **Result units:** Volume

Specific Surface Area: 1.48 m²/g **Surface Weighted Mean D[3,2]:** 4.047 um **Vol. Weighted Mean D[4,3]:** 7.326 um

d(0.1): 2.169 um   **d(0.5):** 6.349 um   **d(0.9):** 13.912 um
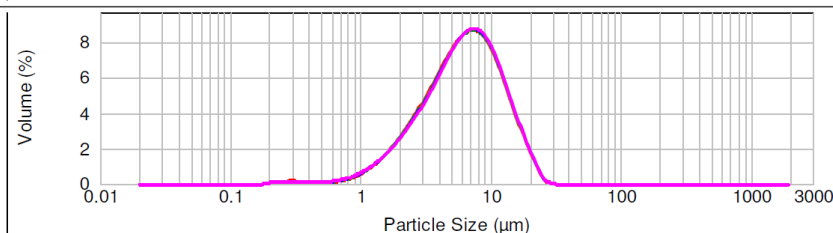
— Mix 13 (AR15ZA0253), 10 February 2015 03:06:59 PM
— Mix 13 (AR15ZA0253), 10 February 2015 03:07:33 PM
— Mix 13 (AR15ZA0253), 10 February 2015 03:08:08 PM
— Mix 13 (AR15ZA0253), 10 February 2015 03:08:42 PM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.83	11.482	5.75	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	1.13	13.183	4.56	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	1.47	15.136	3.35	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.87	17.378	2.20	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.04	1.905	2.33	19.953	1.29	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.11	2.188	2.86	22.909	0.43	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.13	2.512	3.46	26.303	0.09	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.14	2.884	4.13	30.200	0.00	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.13	3.311	4.86	34.674	0.00	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.12	3.802	5.63	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.11	4.365	6.40	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.11	5.012	7.10	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.14	5.754	7.64	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.19	6.607	7.93	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.28	7.586	7.89	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.41	8.710	7.50	91.201	0.00	954.993	0.00	10000.000	
0.091	0.00	0.955	0.60	10.000	6.76	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Operator notes:

Figure A.13 Particle size analysis of mix 13

Figure A.14 Particle size analysis of mix 14

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 15 (AR15ZA0253) **SOP Name:** Diamond Mixes-v1 **Measured:** 10 February 2015 03:25:58 PM

Sample Source & type: Element six DMP **Measured by:** Khosi **Analysed:** 10 February 2015 03:25:59 PM

Sample bulk lot ref: Mix15 **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 8.95 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 1.386 % **Result Emulation:** Off

---

Concentration: 0.0089 %Vol **Span :** 1.605 **Uniformity:** 0.472 **Result units:** Volume

Specific Surface Area: 0.804 m²/g **Surface Weighted Mean D[3,2]:** 7.460 um **Vol. Weighted Mean D[4,3]:** 13.210 um

d(0.1): 3.314 um **d(0.5):** 12.495 um **d(0.9):** 23.375 um
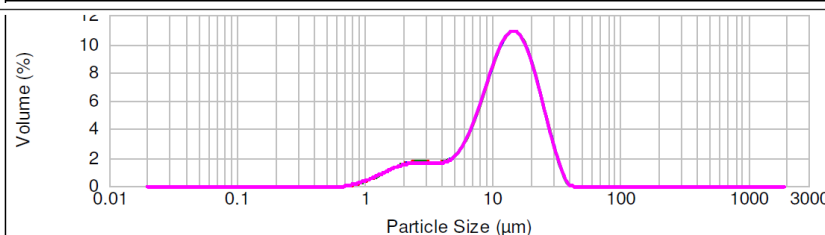
— Mix 15 (AR15ZA0253), 10 February 2015 03:24:15 PM
— Mix 15 (AR15ZA0253), 10 February 2015 03:24:49 PM
— Mix 15 (AR15ZA0253), 10 February 2015 03:25:24 PM
— Mix 15 (AR15ZA0253), 10 February 2015 03:25:58 PM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.56	11.482	9.37	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.78	13.183	9.86	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	1.01	15.136	9.64	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.22	17.378	8.70	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	1.37	19.953	7.19	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	1.46	22.909	5.33	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.49	26.303	3.42	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.46	30.200	1.72	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	1.43	34.674	0.42	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	1.47	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	1.68	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	2.14	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	2.93	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	4.04	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	5.43	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.08	8.710	6.91	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	0.20	10.000	8.33	104.713	0.00	1096.478	0.00		
0.105	0.00	1.096	0.37	11.482		120.226	0.00	1258.925	0.00		

Operator notes:

Figure A.15 Particle size analysis of mix 15
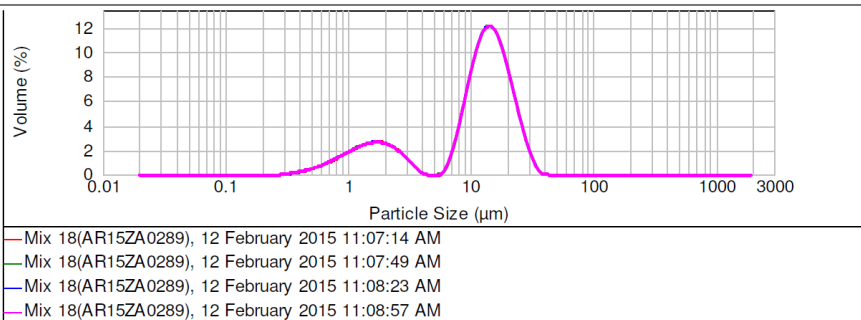
MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 17 (AR15ZA0253)   **SOP Name:** Diamond Mixes-v1   **Measured:** 10 February 2015 03:33:56

Sample Source & type: Element six DMP   **Measured by:** Khosi   **Analysed:** 10 February 2015 03:33:57 PM

Sample bulk lot ref: Mix 17   **Result Source:** Measurement

Particle Name: Diamond 0.1   **Accessory Name:** Hydro 2000MU (A)   **Analysis model:** General purpose   **Sensitivity:** Enhanced

Particle RI: 2.418   **Absorption:** 0.1   **Size range:** 0.020 to 2000.000 um   **Obscuration:** 9.11 %

Dispersant Name: Water   **Dispersant RI:** 1.330   **Weighted Residual:** 1.283 %   **Result Emulation:** Off

---

Concentration: 0.0051 %Vol   **Span :** 1.382   **Uniformity:** 0.388   **Result units:** Volume

Specific Surface Area: 1.31 m²/g   **Surface Weighted Mean D[3,2]:** 4.597 um   **Vol. Weighted Mean D[4,3]:** 13.842 um

d(0.1): 1.449 um   **d(0.5):** 15.142 um   **d(0.9):** 22.378 um
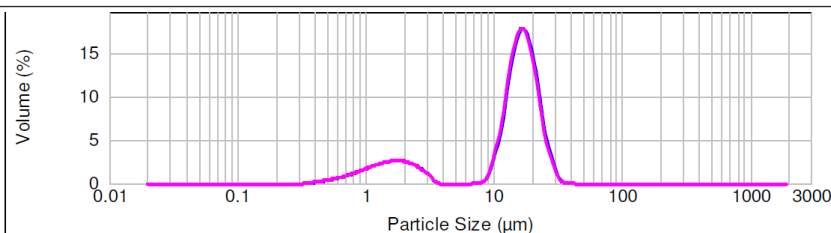
—Mix 17 (AR15ZA0253), 10 February 2015 03:32:13 PM
—Mix 17 (AR15ZA0253), 10 February 2015 03:32:47 PM
—Mix 17 (AR15ZA0253), 10 February 2015 03:33:22 PM
—Mix 17 (AR15ZA0253), 10 February 2015 03:33:56 PM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	1.88	11.482	8.72	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	2.14	13.183	13.70	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.31	15.136	15.98	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.37	17.378	14.71	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	2.27	19.953	10.85	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	2.00	22.909	5.43	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.54	26.303	2.57	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.97	30.200	0.46	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.01	3.311	0.16	34.674	0.05	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.14	3.802	0.00	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.24	4.365	0.00	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.37	5.012	0.00	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.54	5.754	0.00	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.76	6.607	0.01	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	1.01	7.586	0.18	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.29	8.710	1.36	91.201	0.00	954.993	0.00	10000.000	
0.091	0.00	0.955	1.59	10.000	4.41	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Operator notes:

Mastersizer 2000 Ver. 5.22
Serial Number : 34264-20

File name: Development Node
Record Number: 3216

Figure A.17 Particle size analysis of mix 17

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 18(AR15ZA0289)   **SOP Name:** Diamond Mixes-v1   **Measured:** 12 February 2015 11:08:57

Sample Source & type: Element six DMP   **Measured by:** Khosi   **Analysed:** 12 February 2015 11:08:59 AM

Sample bulk lot ref: Mix 18   **Result Source:** Measurement

Particle Name: Diamond 0.1   **Accessory Name:** Hydro 2000MU (A)   **Analysis model:** General purpose   **Sensitivity:** Enhanced

Particle RI: 2.418   **Absorption:** 0.1   **Size range:** 0.020 to 2000.000 um   **Obscuration:** 11.67 %

Dispersant Name: Water   **Dispersant RI:** 1.330   **Weighted Residual:** 5.502 %   **Result Emulation:** Off

---

Concentration: 0.0058 %Vol   **Span:** 1.664   **Uniformity:** 0.492   **Result units:** Volume

Specific Surface Area: 1.46 m²/g   **Surface Weighted Mean D[3,2]:** 4.096 um   **Vol. Weighted Mean D[4,3]:** 12.157 um

d(0.1): 1.359 um   **d(0.5):** 12.370 um   **d(0.9):** 21.947 um

— Mix 18(AR15ZA0289), 12 February 2015 11:07:14 AM
— Mix 18(AR15ZA0289), 12 February 2015 11:07:49 AM
— Mix 18(AR15ZA0289), 12 February 2015 11:08:23 AM
— Mix 18(AR15ZA0289), 12 February 2015 11:08:57 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	1.98	11.482	10.34	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	2.21	13.183	10.95	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.36	15.136	10.39	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.39	17.378	8.86	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	2.28	19.953	6.75	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	2.02	22.909	4.50	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.61	26.303	2.53	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.01	2.884	1.10	30.200	1.00	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.10	3.311	0.56	34.674	0.12	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.19	3.802	0.09	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.30	4.365	0.00	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.45	5.012	-0.01	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.64	5.754	0.39	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.87	6.607	1.79	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	1.13	7.586	3.91	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.41	8.710	6.36	91.201	0.00	954.993	0.00	10000.000	
0.091	0.00	0.955	1.71	10.000	8.70	104.713	0.00	1096.478	0.00		
0.105	0.00	1.096		11.482		120.226	0.00	1258.925	0.00		

Operator notes:

Figure A.18 Particle size analysis of mix 18

MALVERN INSTRUMENTS

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 19(AR15ZA0289) **SOP Name:** Diamond Mixes-v1 **Measured:** 12 February 2015 11:22:03

Sample Source & type: Element six DMP **Measured by:** Khosi **Analysed:** 12 February 2015 11:22:04 AM

Sample bulk lot ref: Mix 19 **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 9.65 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 0.766 % **Result Emulation:** Off

Concentration: 0.0051 %Vol **Span :** 1.403 **Uniformity:** 0.403 **Result units:** Volume

Specific Surface Area: 1.37 m²/g **Surface Weighted Mean D[3,2]:** 4.372 um **Vol. Weighted Mean D[4,3]:** 13.407 um

d(0.1): 1.410 um **d(0.5):** 14.757 um **d(0.9):** 22.113 um
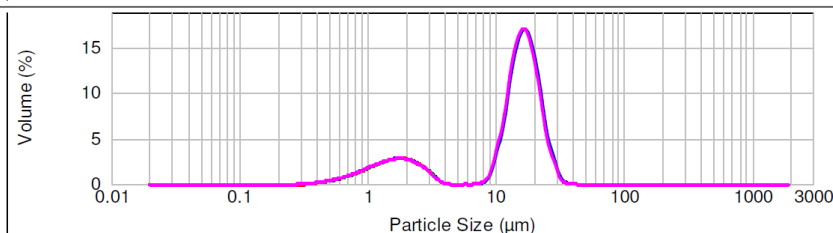
Mix 19(AR15ZA0289), 12 February 2015 11:20:19 AM
Mix 19(AR15ZA0289), 12 February 2015 11:20:54 AM
Mix 19(AR15ZA0289), 12 February 2015 11:21:28 AM
Mix 19(AR15ZA0289), 12 February 2015 11:22:03 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	1.98	11.482	8.99	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	2.26	13.183	13.60	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.46	15.136	15.44	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.54	17.378	13.92	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	2.45	19.953	10.10	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	2.19	22.909	5.01	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.73	26.303	2.36	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.01	2.884	1.15	30.200	0.43	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.07	3.311	0.43	34.674	0.05	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.14	3.802	0.03	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.24	4.365	0.00	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.37	5.012	0.00	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.55	5.754	0.00	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.77	6.607	0.01	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	1.04	7.586	0.29	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.34	8.710	1.63	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	1.66	10.000	4.78	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Operator notes:

Malvern Instruments Ltd.
Malvern, UK

Mastersizer 2000 Ver. 5.22
Serial Number : 34264-20

File name: Development Node
Record Number: 3224

Figure A.19 Particle size analysis of mix 19

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 20 (AR15ZA0289) **SOP Name:** Diamond Mixes-v1 **Measured:** 12 February 2015 11:29:25

Sample Source & type: Element six DMP **Measured by:** Khosi **Analysed:** 12 February 2015 11:29:26 AM

Sample bulk lot ref: Mix 20 **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 10.16 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 0.857 % **Result Emulation:** Off

---

Concentration: 0.0053 %Vol **Span :** 1.398 **Uniformity:** 0.4 **Result units:** Volume

Specific Surface Area: 1.37 m²/g **Surface Weighted Mean D[3,2]:** 4.384 um **Vol. Weighted Mean D[4,3]:** 14.040 um

d(0.1): 1.383 um **d(0.5):** 15.426 um **d(0.9):** 22.944 um

Volume (%) vs Particle Size (µm)
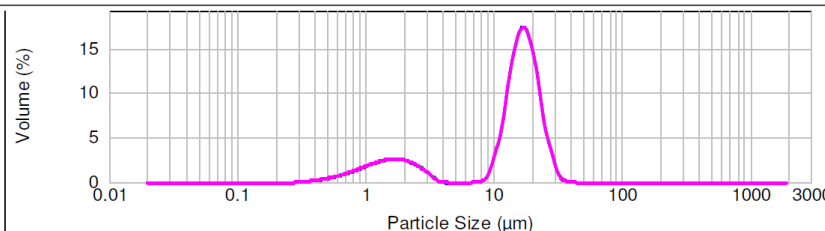
— Mix 20 (AR15ZA0289), 12 February 2015 11:27:41 AM
— Mix 20 (AR15ZA0289), 12 February 2015 11:28:16 AM
— Mix 20 (AR15ZA0289), 12 February 2015 11:28:50 AM
— Mix 20 (AR15ZA0289), 12 February 2015 11:29:25 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010		0.105		1.096		11.482		120.226		1258.925	
0.011	0.00	0.120	0.00	1.259	1.96	13.183	7.77	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.20	15.136	12.70	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.35	17.378	15.45	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	2.37	19.953	14.89	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	2.25	22.909	11.57	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.95	26.303	6.21	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.51	30.200	3.13	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.01	3.311	0.96	34.674	0.67	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.09	3.802	0.31	39.811	0.10	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.18	4.365	0.01	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.28	5.012	0.00	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.42	5.754	0.00	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.60	6.607	0.00	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.83	7.586	0.01	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.09	8.710	0.15	91.201	0.00	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	1.38	10.000	1.14	104.713	0.00	1096.478	0.00		
0.105	0.00	1.096	1.68	11.482	3.80	120.226	0.00	1258.925	0.00		

Operator notes:

Malvern Instruments Ltd.
Malvern, UK

Mastersizer 2000 Ver. 5.22
Serial Number : 34264-20

File name: Development Node
Record Number: 3228

Figure A.20 Particle size analysis of mix 20

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 22 (AR15ZA0289) **SOP Name:** Diamond Mixes-v1 **Measured:** 12 February 2015 11:45:34

Sample Source & type: Element six DMP **Measured by:** Khosi **Analysed:** 12 February 2015 11:45:35 AM

Sample bulk lot ref: Mix 22 **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 8.87 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 1.925 % **Result Emulation:** Off

Concentration: 0.0049 %Vol **Span :** 1.377 **Uniformity:** 0.388 **Result units:** Volume

Specific Surface Area: 1.3 m²/g **Surface Weighted Mean D[3,2]:** 4.619 um **Vol. Weighted Mean D[4,3]:** 13.462 um

d(0.1): 1.502 um   **d(0.5):** 14.773 um   **d(0.9):** 21.850 um
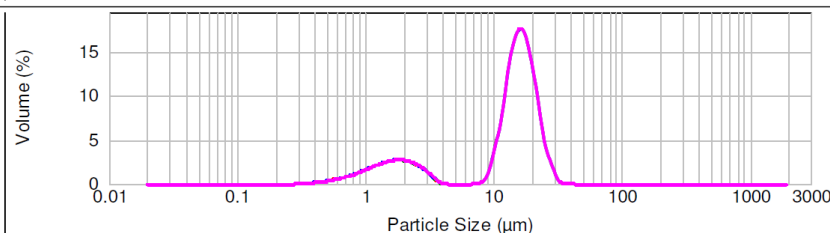
—Mix 22 (AR15ZA0289), 12 February 2015 11:47:17 AM
—Mix 22 (AR15ZA0289), 12 February 2015 11:46:43 AM
—Mix 22 (AR15ZA0289), 12 February 2015 11:46:08 AM
—Mix 22 (AR15ZA0289), 12 February 2015 11:45:34 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010		0.105	0.00	1.096		11.482		120.226		1258.925	
0.011	0.00	0.120	0.00	1.259	1.82	13.183	9.40	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.13	15.136	14.28	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.36	17.378	16.03	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	2.48	19.953	14.16	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	2.45	22.909	9.96	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	2.22	26.303	4.70	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.01	2.884	1.79	30.200	2.10	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.07	3.311	1.21	34.674	0.32	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.13	3.802	0.46	39.811	0.03	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.21	4.365	0.04	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.31	5.012	0.00	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.46	5.754	0.00	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.65	6.607	0.01	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.89	7.586	0.21	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.18	8.710	1.54	91.201	0.00	954.993	0.00	10000.000	
0.091	0.00	0.955	1.50	10.000	4.88	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Operator notes:

Malvern Instruments Ltd. Mastersizer 2000 Ver. 5.22 File name: Development Node
Malvern, UK Serial Number : 34264-20 Record Number: 3229

Figure A.22 Particle size analysis of mix 22

MASTERSIZER 2000

Result Analysis Report

Sample Name: Mix 23 (AR15ZA0289) **SOP Name:** Diamond Mixes-v1 **Measured:** 12 February 2015 12:01:27

Sample Source & type: Element six DMP **Measured by:** Khosi **Analysed:** 12 February 2015 12:01:28 PM

Sample bulk lot ref: Mix 23 **Result Source:** Measurement

Particle Name: Diamond 0.1 **Accessory Name:** Hydro 2000MU (A) **Analysis model:** General purpose **Sensitivity:** Enhanced

Particle RI: 2.418 **Absorption:** 0.1 **Size range:** 0.020 to 2000.000 um **Obscuration:** 9.54 %

Dispersant Name: Water **Dispersant RI:** 1.330 **Weighted Residual:** 1.573 % **Result Emulation:** Off

Concentration: 0.0050 %Vol **Span:** 1.397 **Uniformity:** 0.404 **Result units:** Volume

Specific Surface Area: 1.38 m²/g **Surface Weighted Mean D[3,2]:** 4.347 um **Vol. Weighted Mean D[4,3]:** 13.008 um

d(0.1): 1.425 um **d(0.5):** 14.354 um **d(0.9):** 21.483 um
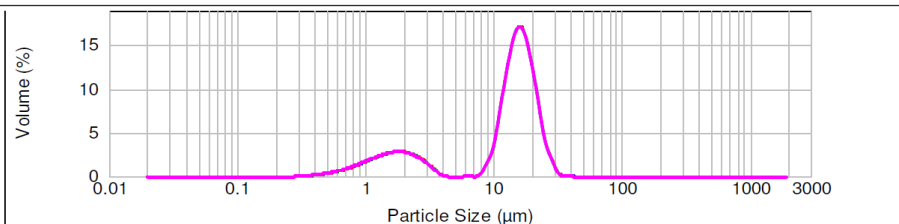
Mix 23 (AR15ZA0289), 12 February 2015 11:59:44 AM
Mix 23 (AR15ZA0289), 12 February 2015 12:00:18 PM
Mix 23 (AR15ZA0289), 12 February 2015 12:00:53 PM
Mix 23 (AR15ZA0289), 12 February 2015 12:01:27 PM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	1.95	11.482	10.07	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	2.24	13.183	14.12	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	2.46	15.136	15.50	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	2.56	17.378	13.27	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	2.51	19.953	9.13	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	2.26	22.909	4.20	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.84	26.303	1.84	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.01	2.884	1.26	30.200	0.26	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.08	3.311	0.57	34.674	0.02	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.15	3.802	0.08	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.24	4.365	0.00	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.37	5.012	0.00	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.53	5.754	0.00	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.74	6.607	0.00	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	1.00	7.586	-0.00	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.30	8.710	0.47	91.201	0.00	954.993	0.00	10000.000	
0.091	0.00	0.955	1.62	10.000	1.96	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482	5.37	120.226	0.00	1258.925	0.00		

Operator notes:

Malvern Instruments Ltd.
Malvern, UK

Mastersizer 2000 Ver. 5.22
Serial Number : 34264-20

File name: Development Node
Record Number: 3236

Figure A.23 Particle size analysis of mix

APPENDIX B

SEM results of the diamond powder mixes

This appendix contains the SEM results from mix 1 to mix 23.
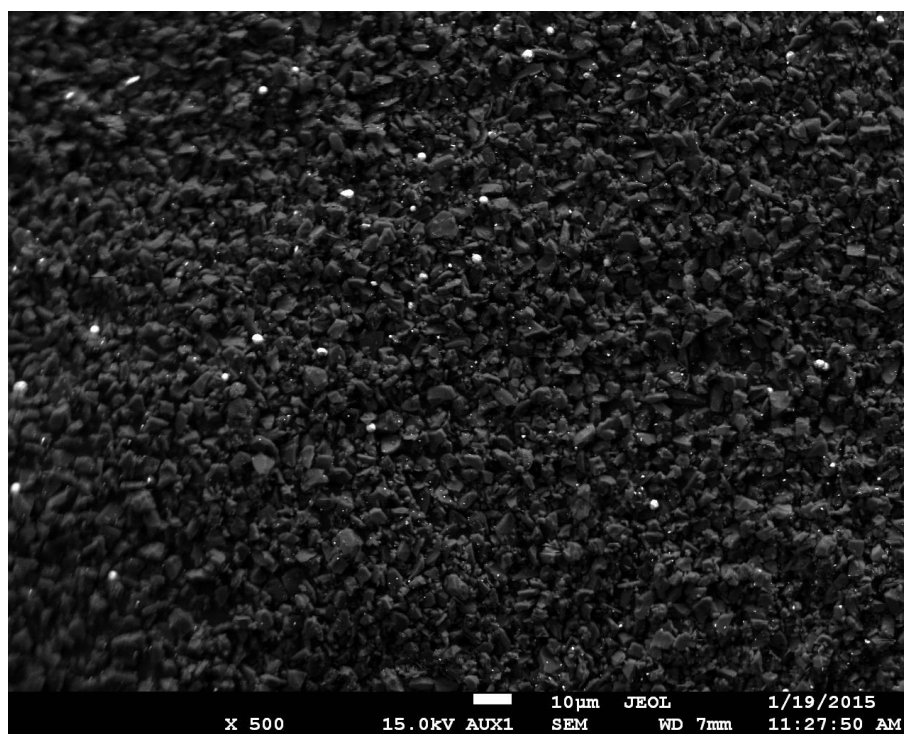
Figure B.1 SEM image of mix 1
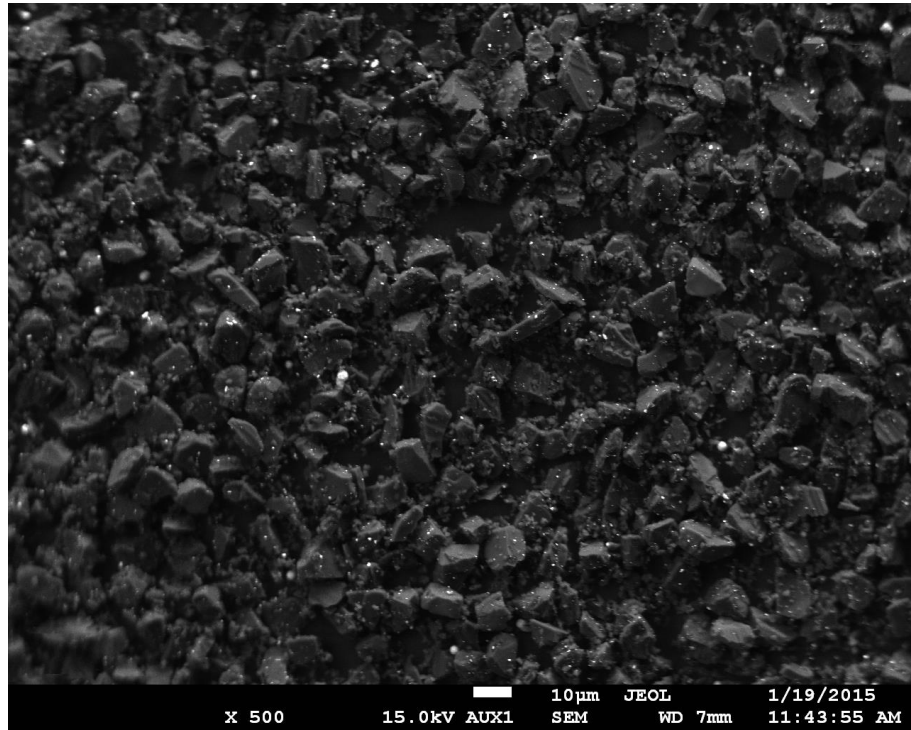
Figure B.2 SEM image of mix 2
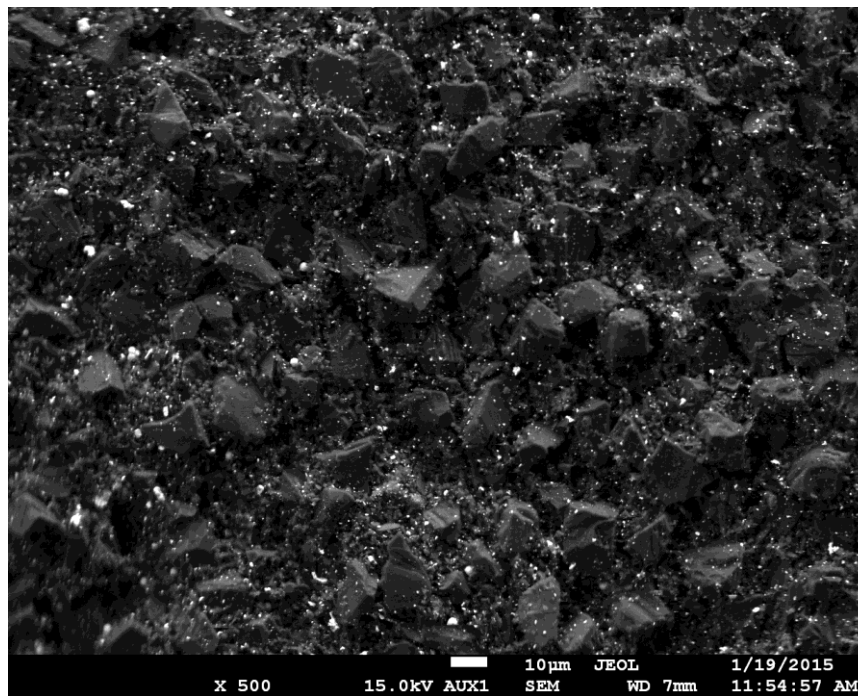
Figure B.3 SEM image of mix 3
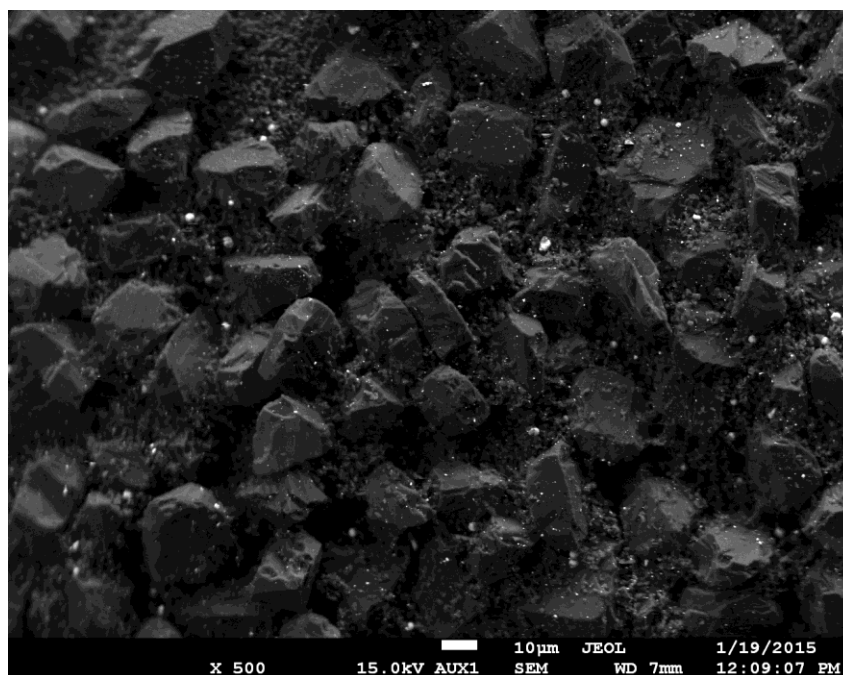
Figure B.4 SEM image of mix 4
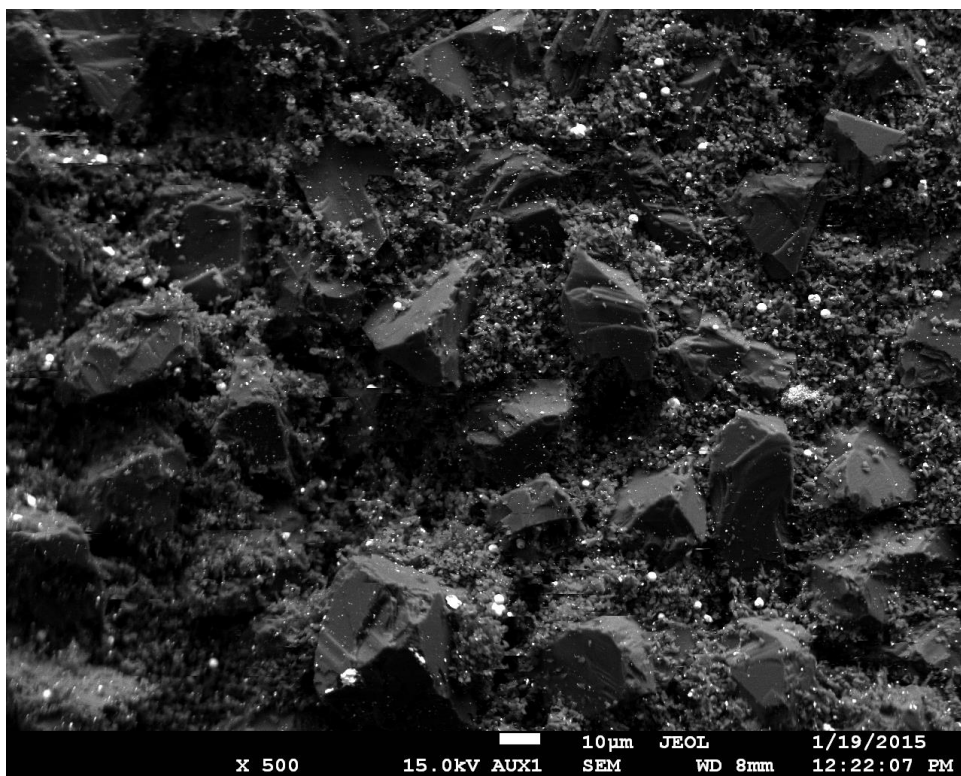
Figure B.5 SEM image of mix 5
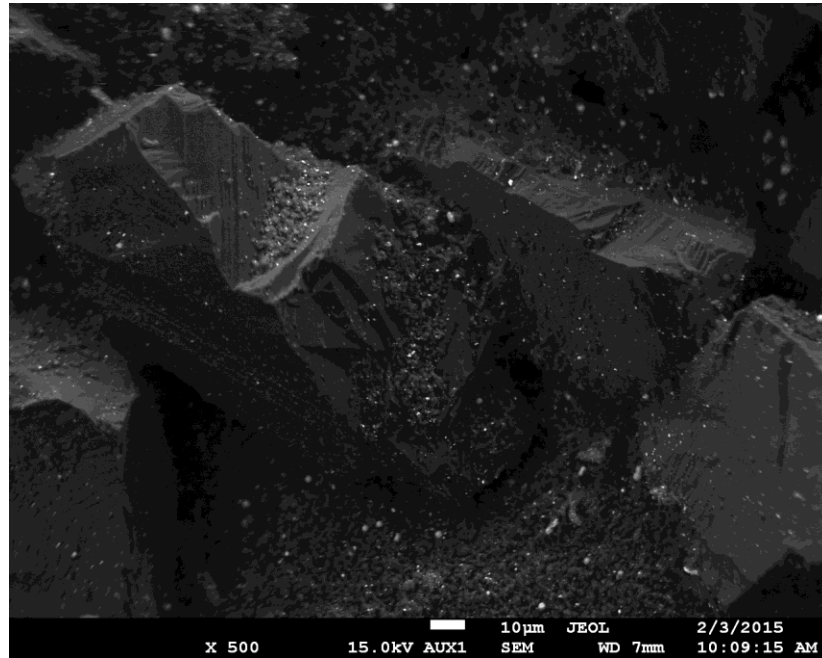
Figure B.6 SEM image of mix 6
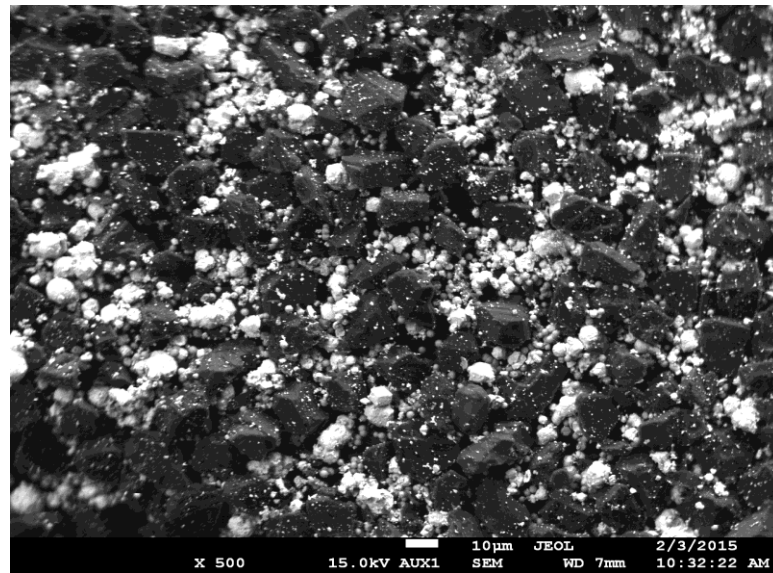
Figure B.8 SEM image of mix 8
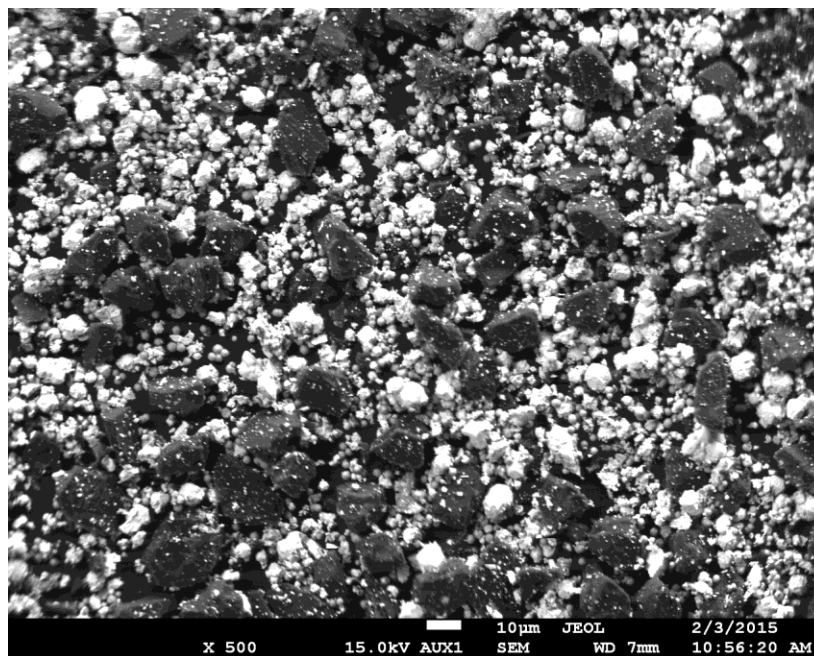
Figure B.9 SEM image of mix 9
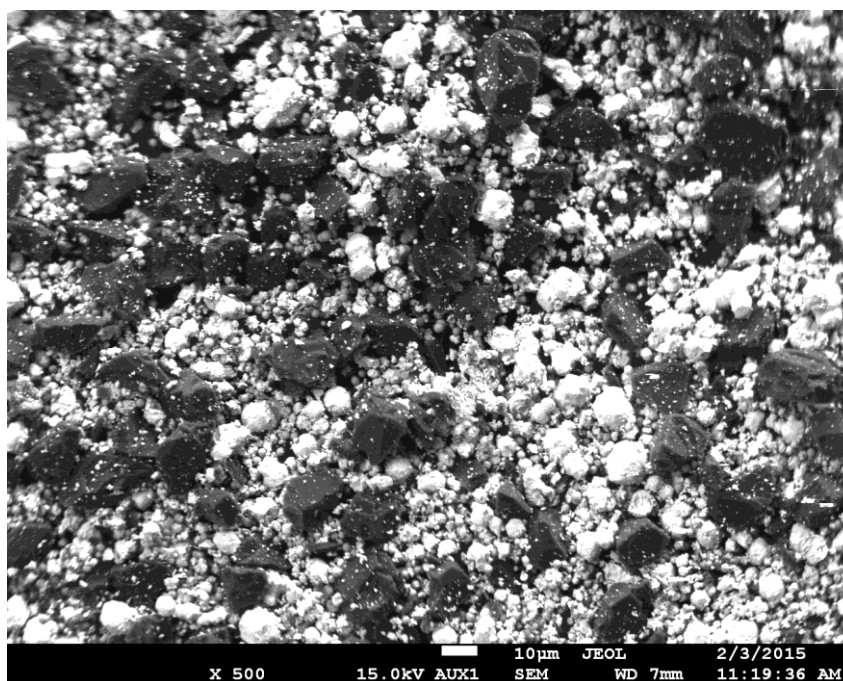
Figure B.10 SEM image of mix 10
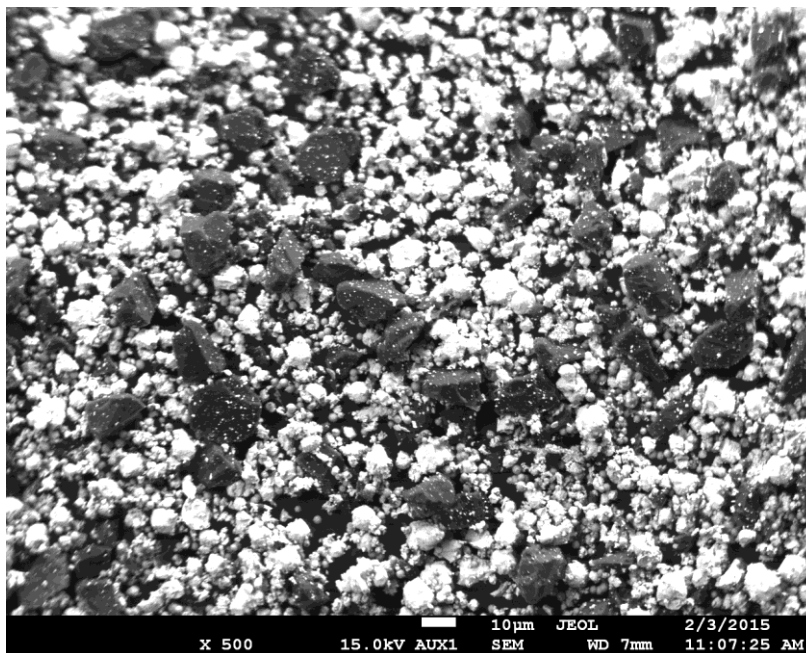
Figure B.11 SEM image of mix 11
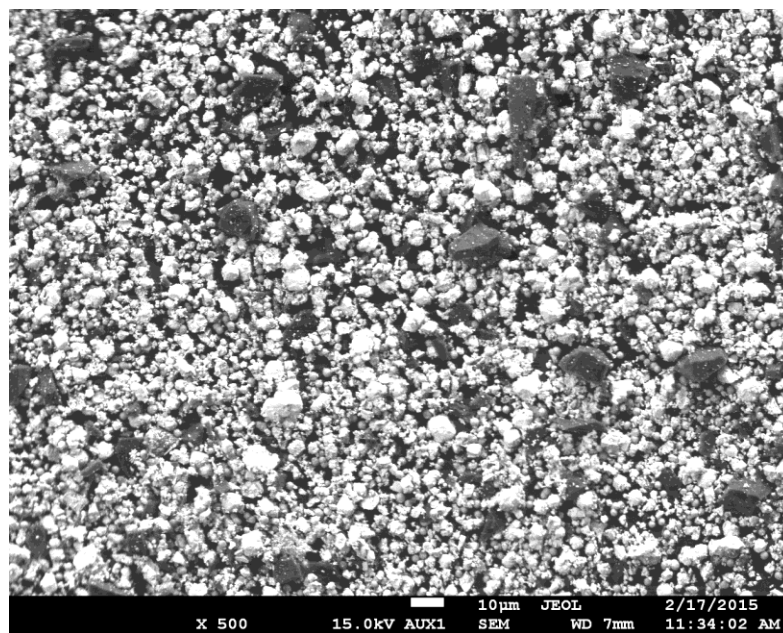
Figure B.12 SEM image of mix 12
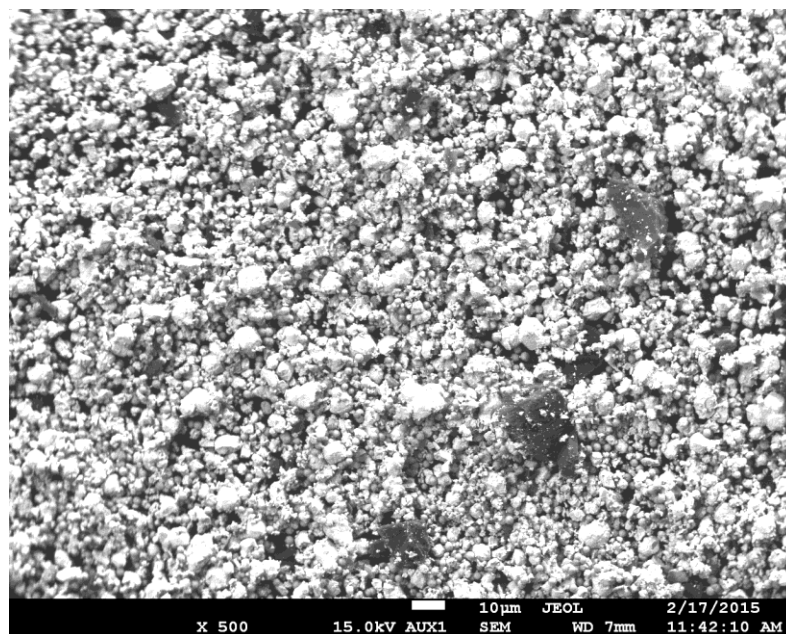
Figure B.13 SEM image of mix 13
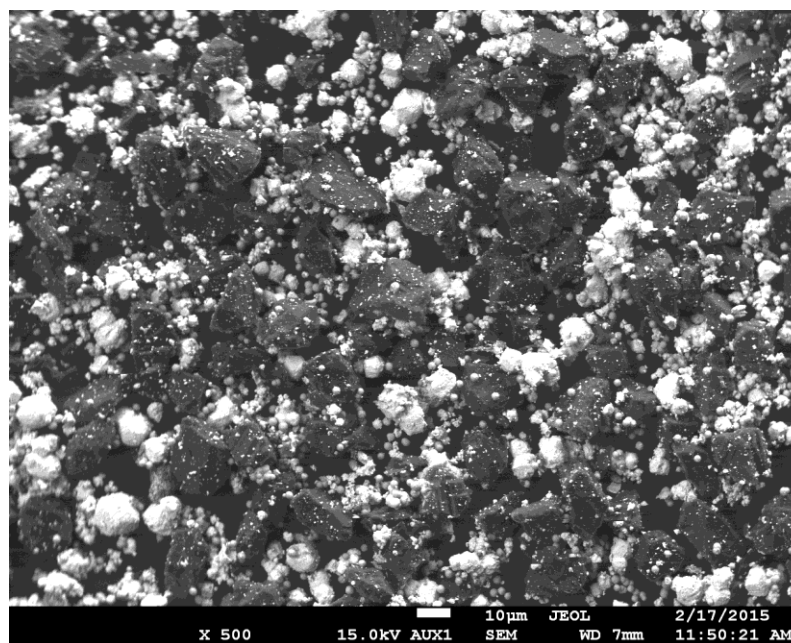
Figure B.14 SEM image of mix 14
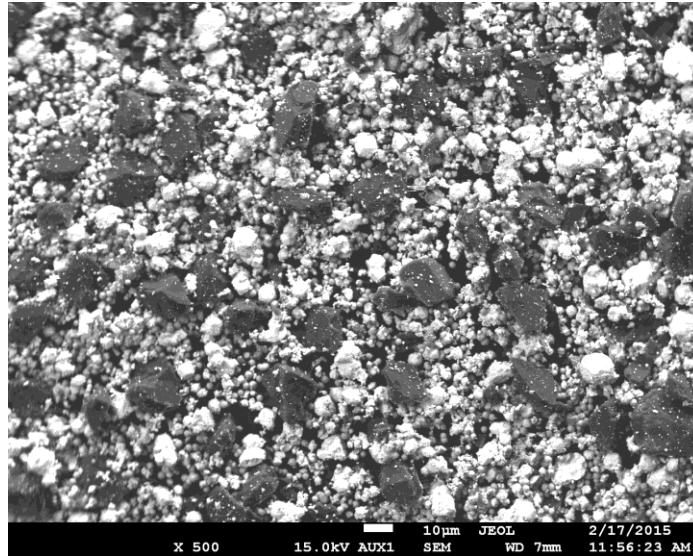
Figure B.15 SEM image of mix 15
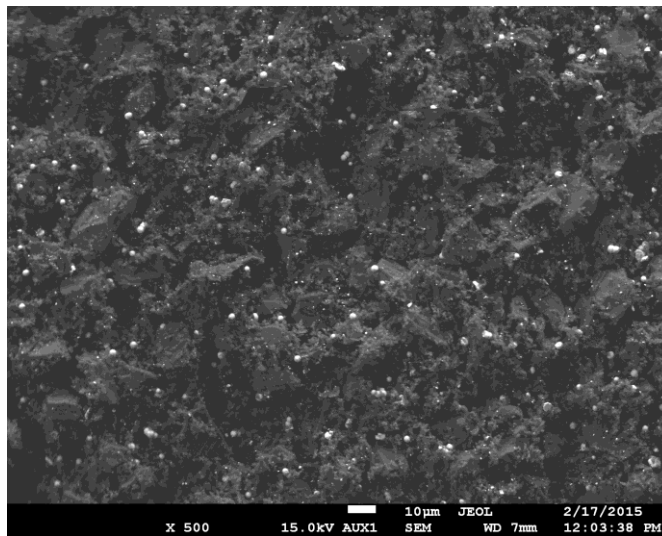
Figure B.17 SEM image of mix 17
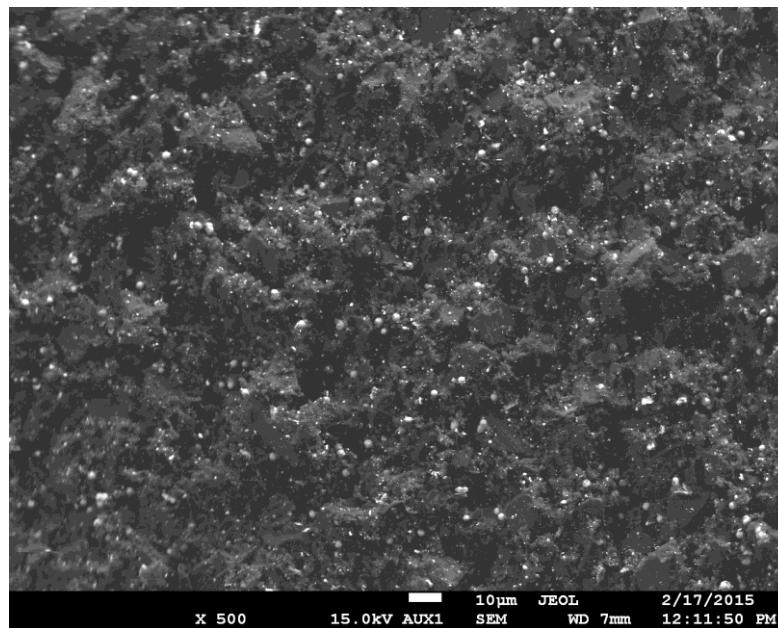
Figure B.18SEM image of mix 18
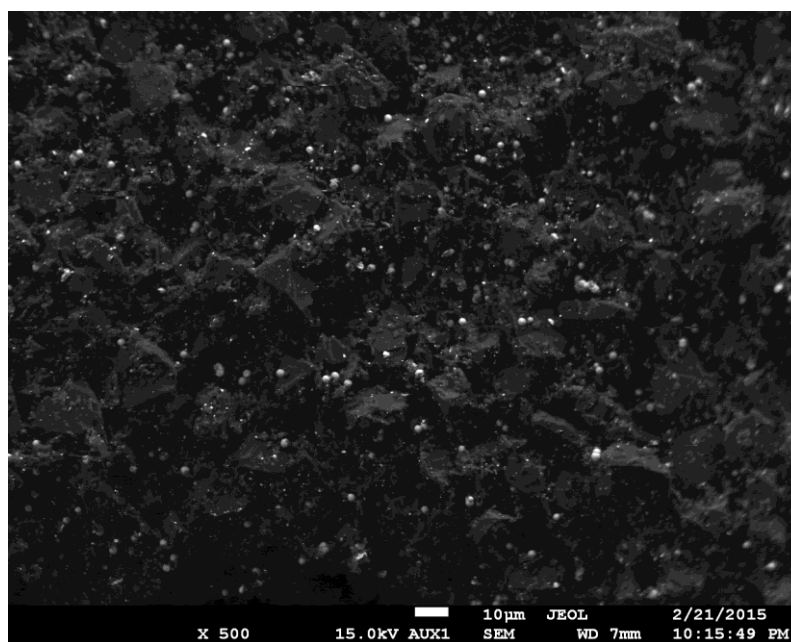
Figure B.19 SEM image of mix 19
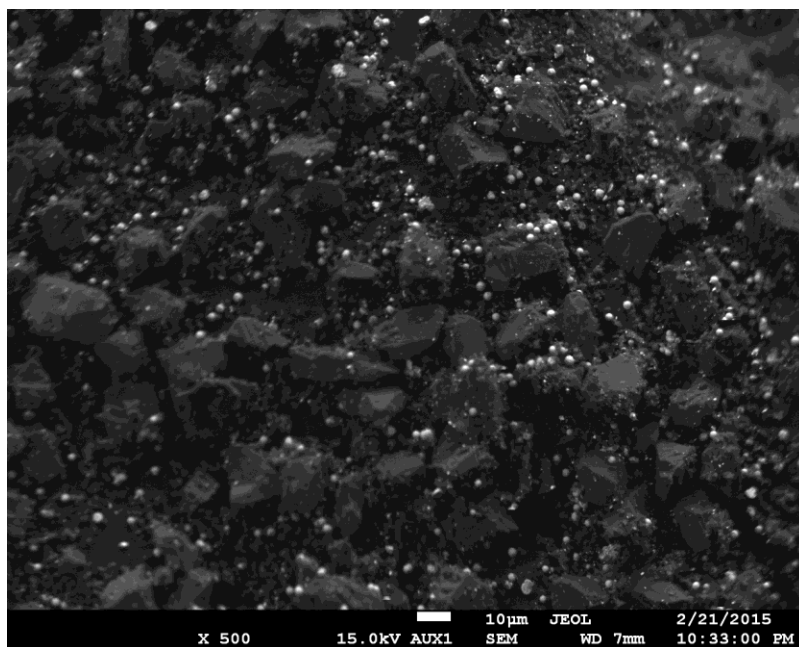
Figure B.20 SEM image of mix 20
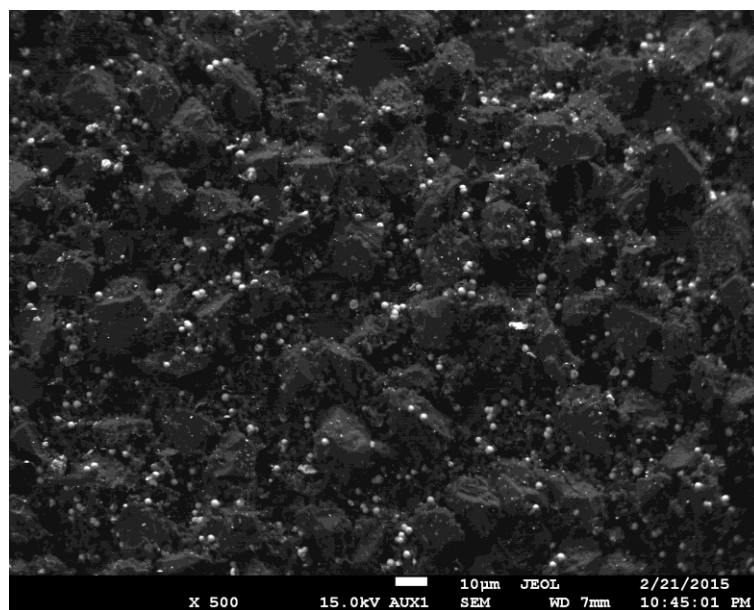
Figure B.21 SEM image of mix 21
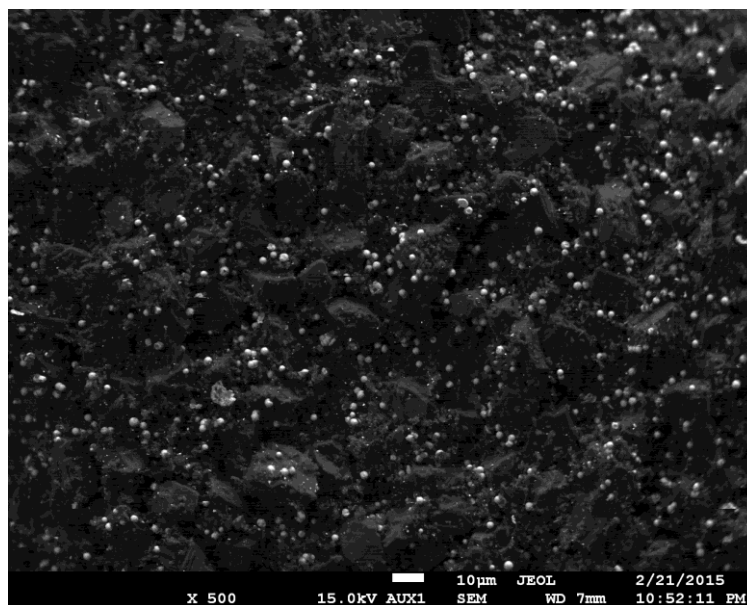
Figure B.22 SEM image of mix 22
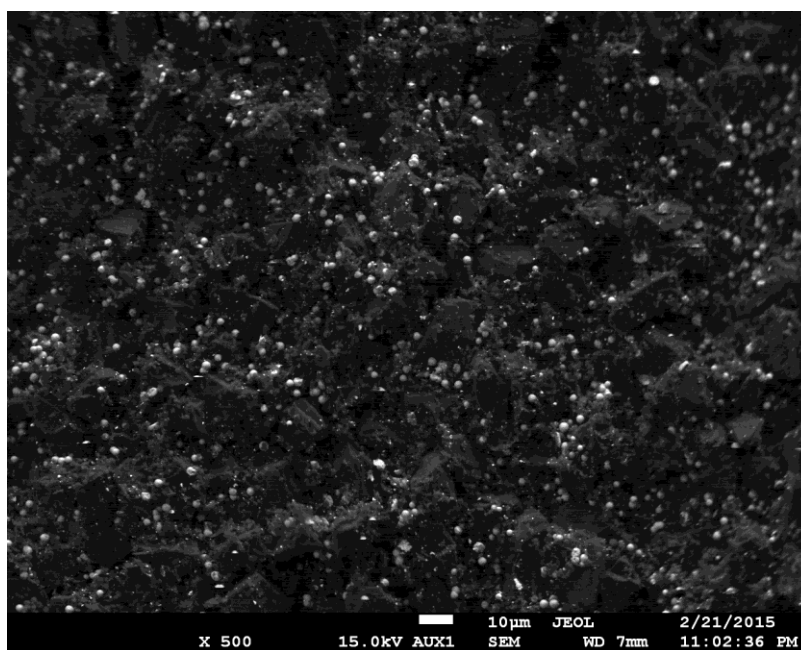
Figure B.23 SEM image of mix 23

APPENDIX C

VBX test wear progression images

This appendix contains the VBX test wear progression images of the tested variants.

0 pass

60 passes

3.69 mm²

2 mm

90 pass

113.89 mm²

2 mm
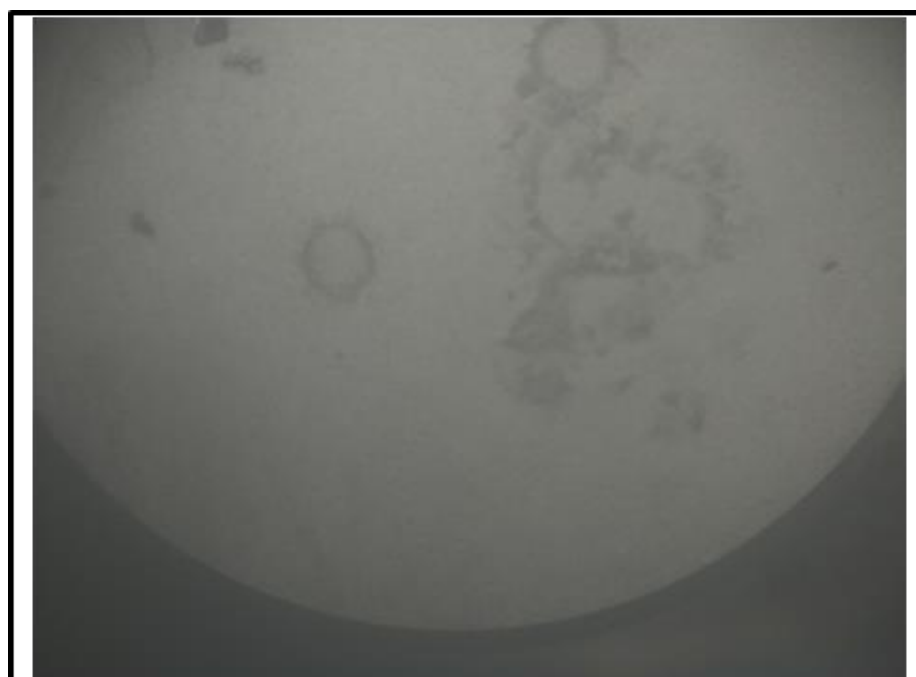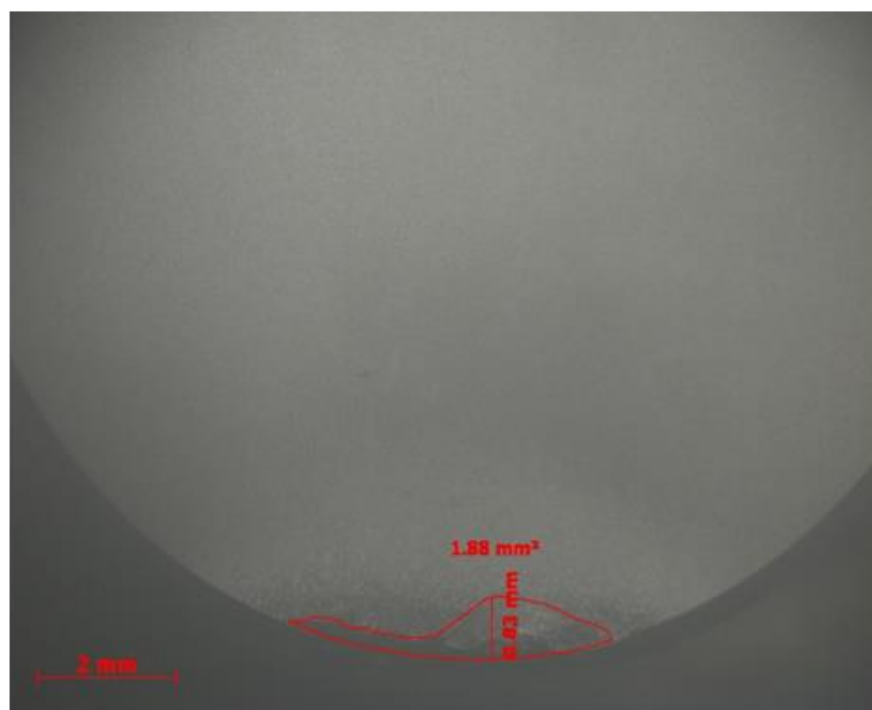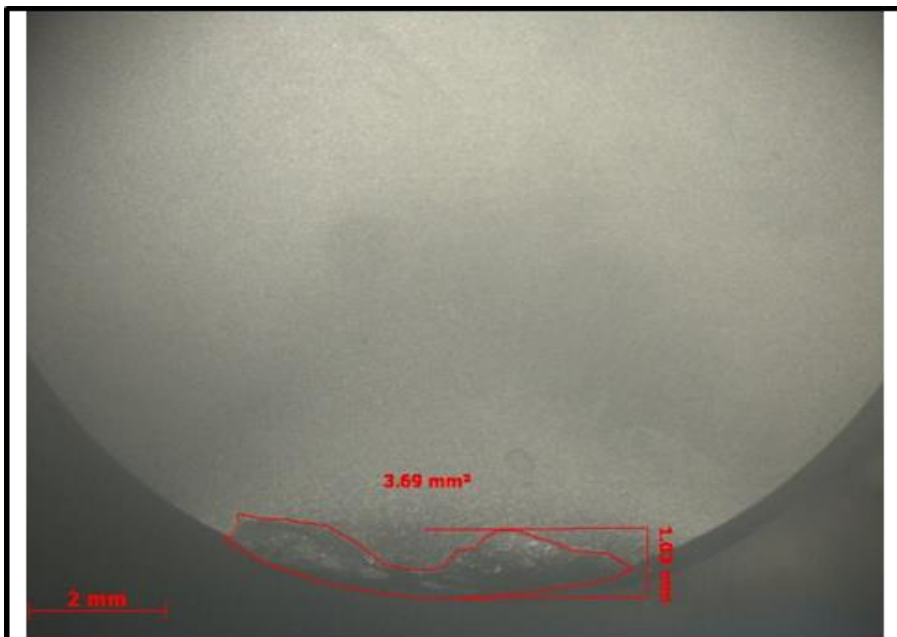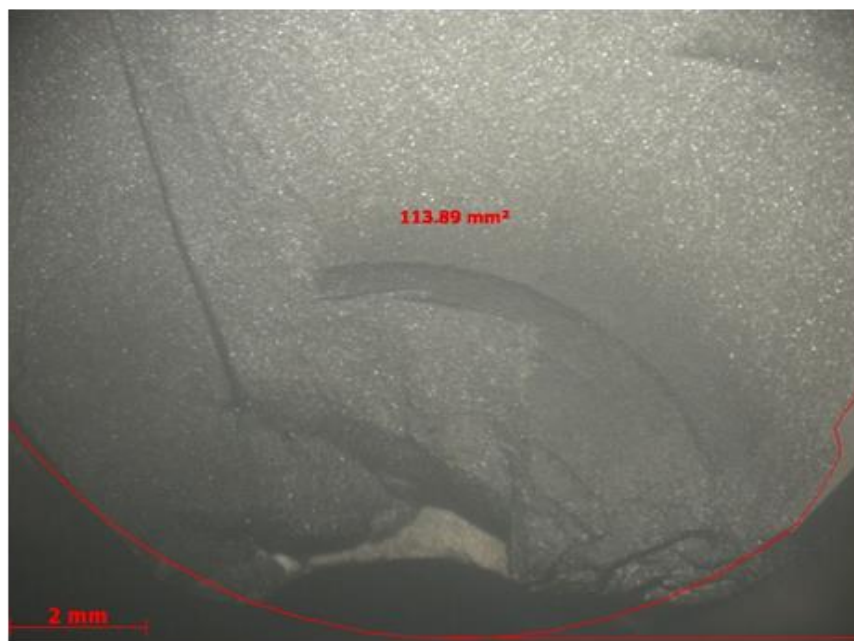
132 passes

Variant B: test 1

0 pass

60 passes

11.01 mm²

2 mm

120 pass

57.31 mm²

**8.51 mm**

2 mm
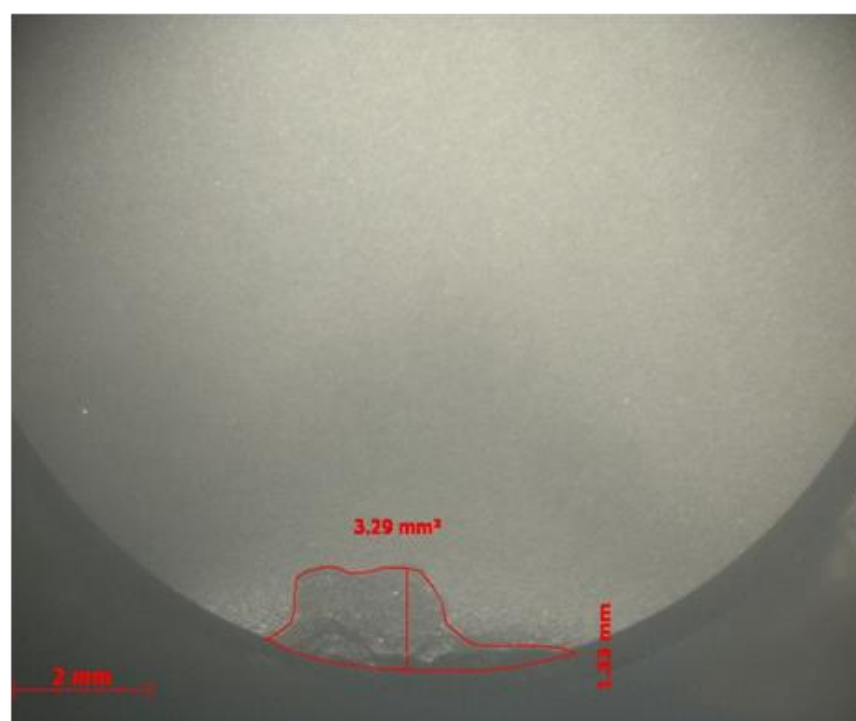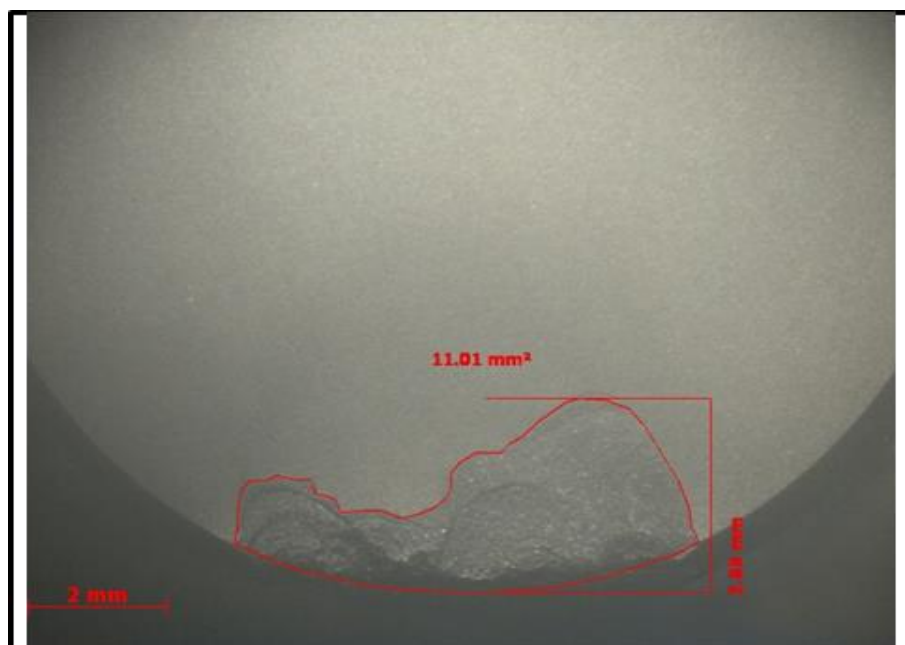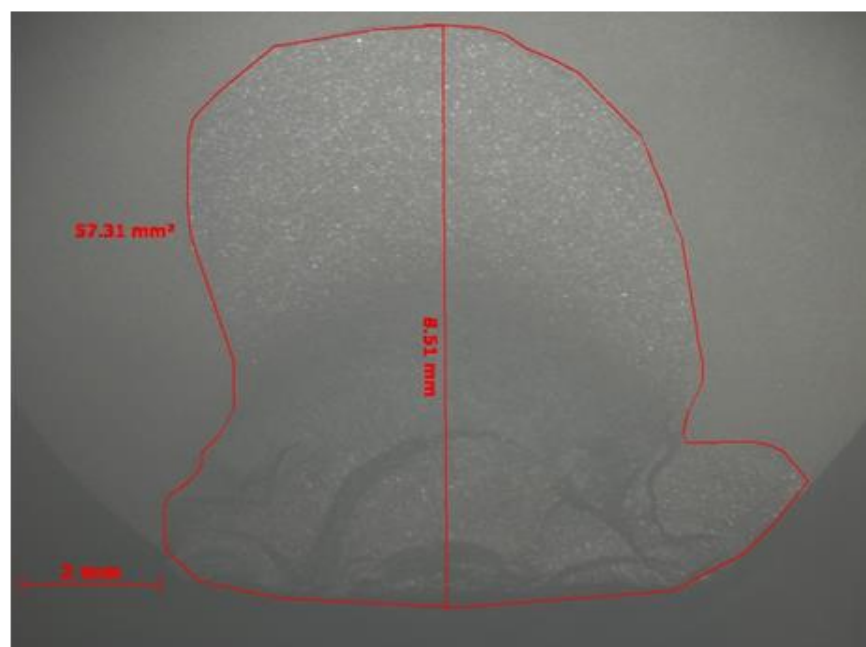
180 passes

Variant B: test 2
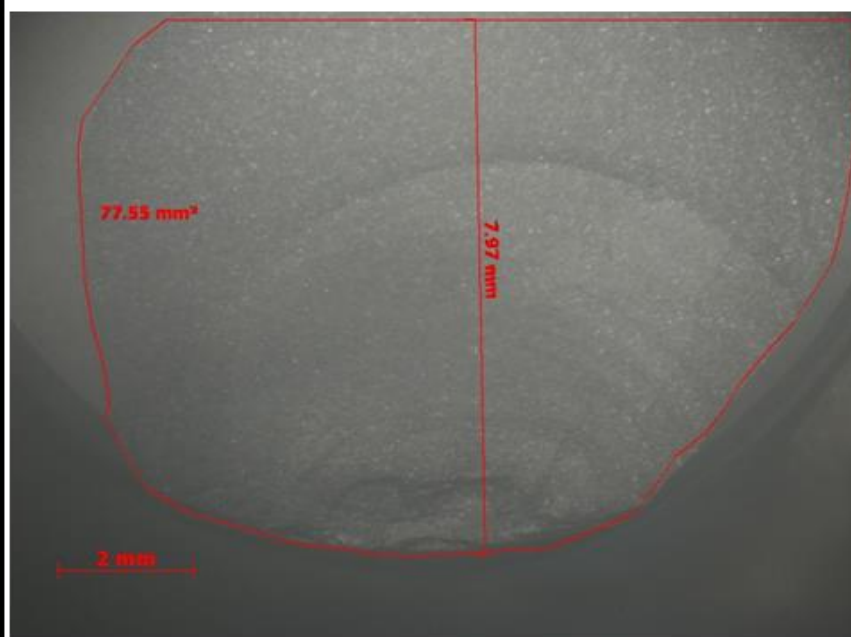
0 pass
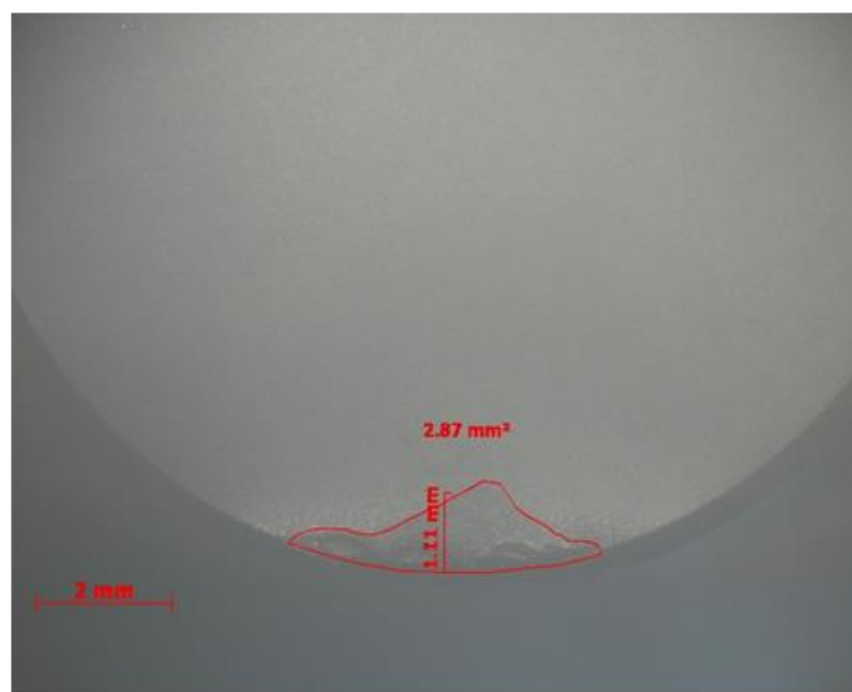
60 passes
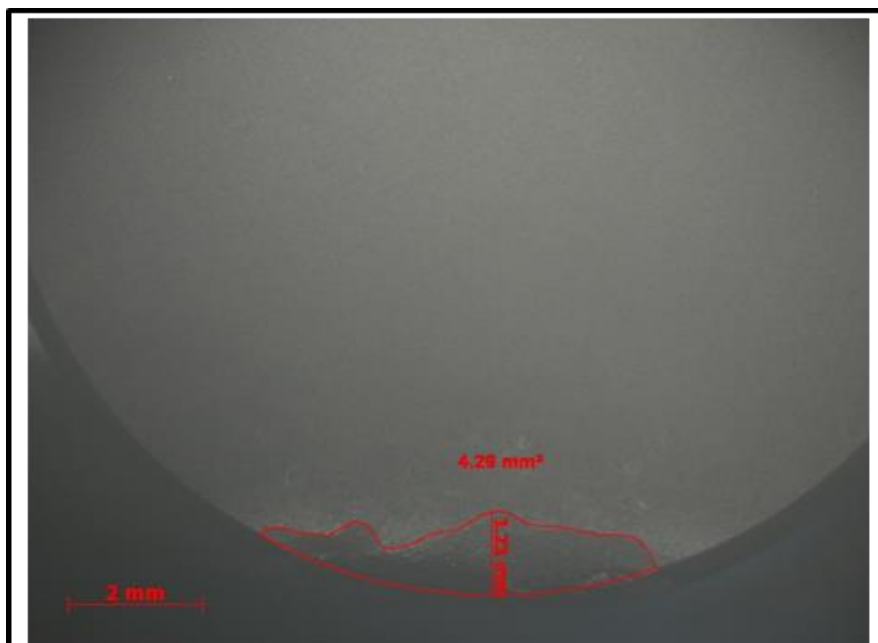
90 pass

118passes

Variant B: test 3

0 pass

10 passes

Variant D: test 1

0 pass

7 passes

Variant D: test 2

116

0 pass

24.39 mm²

2 mm
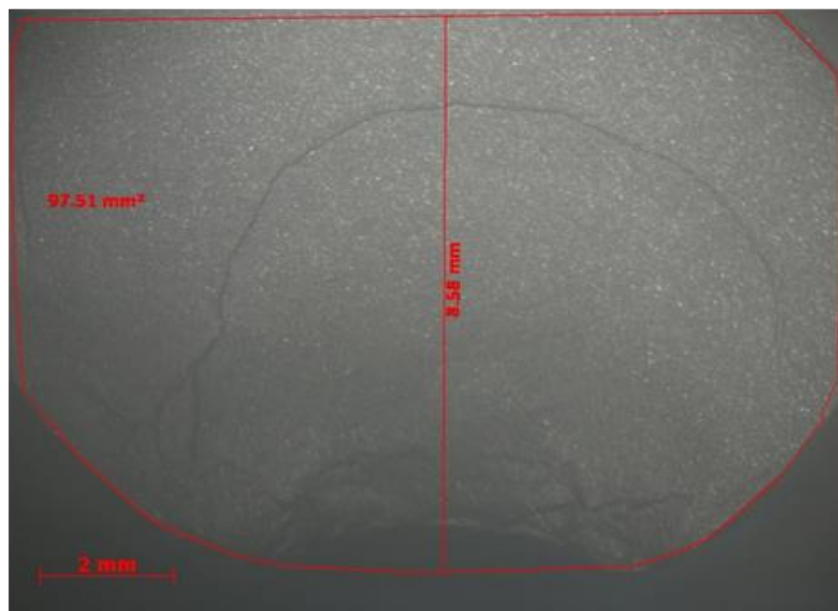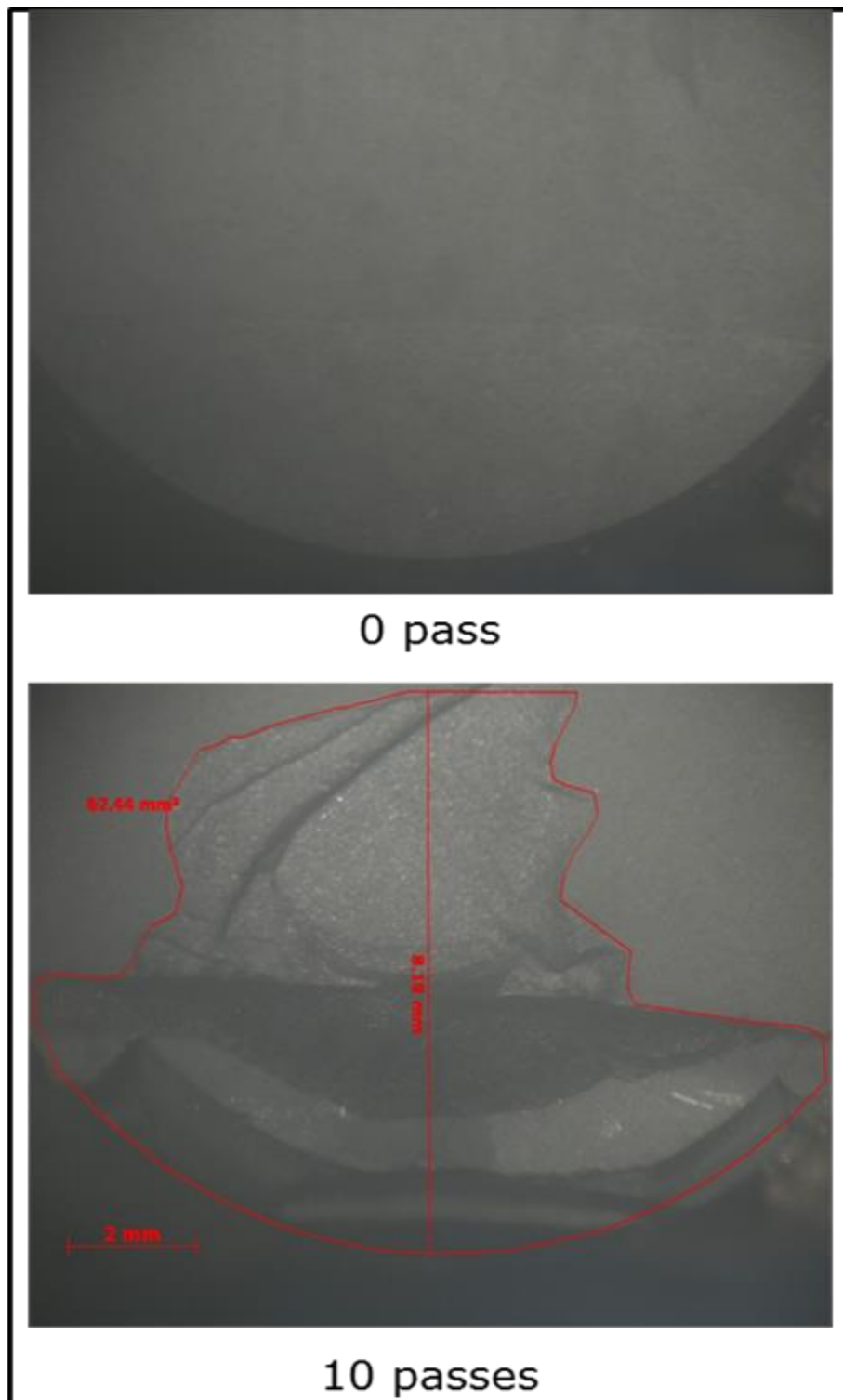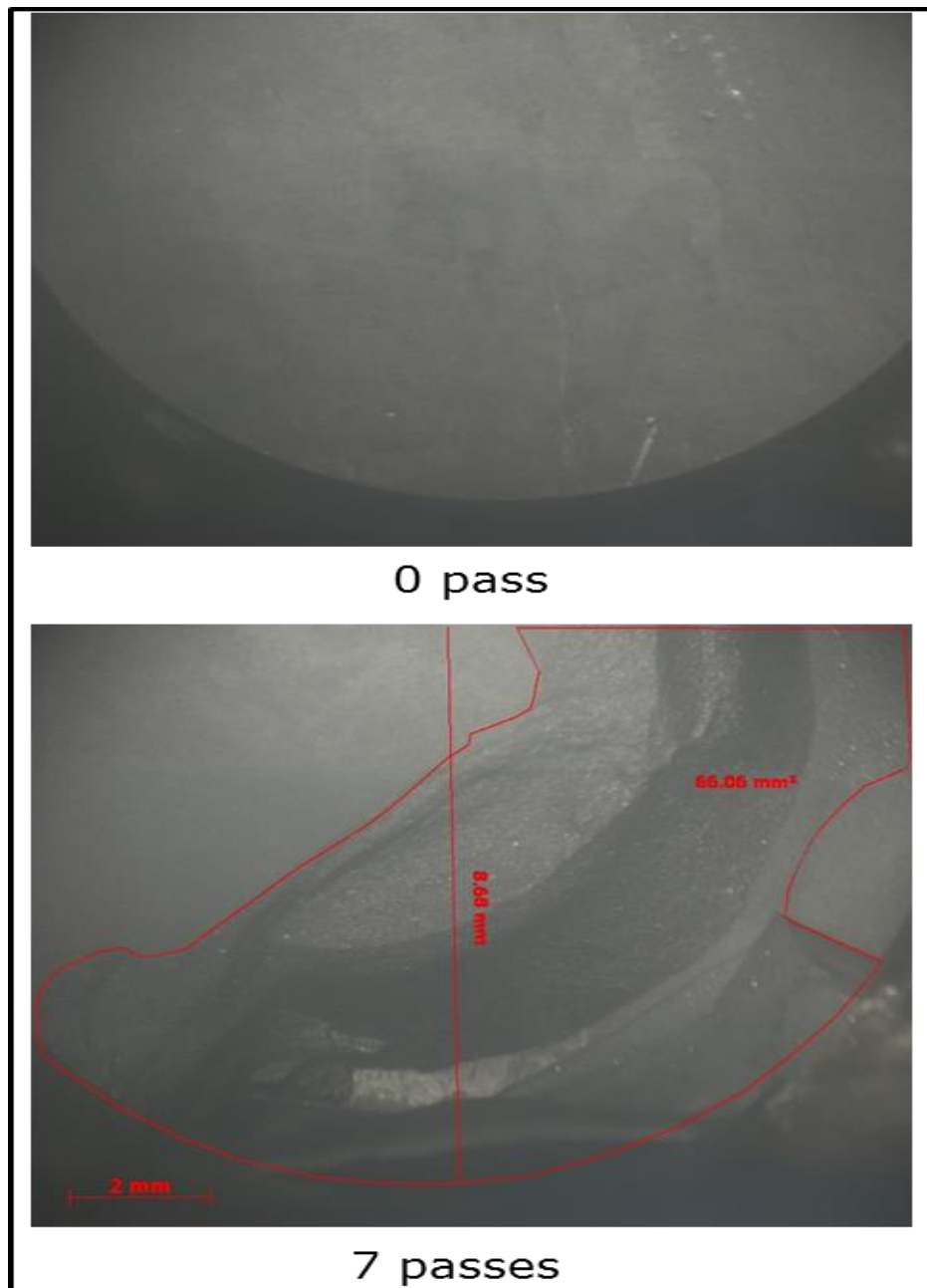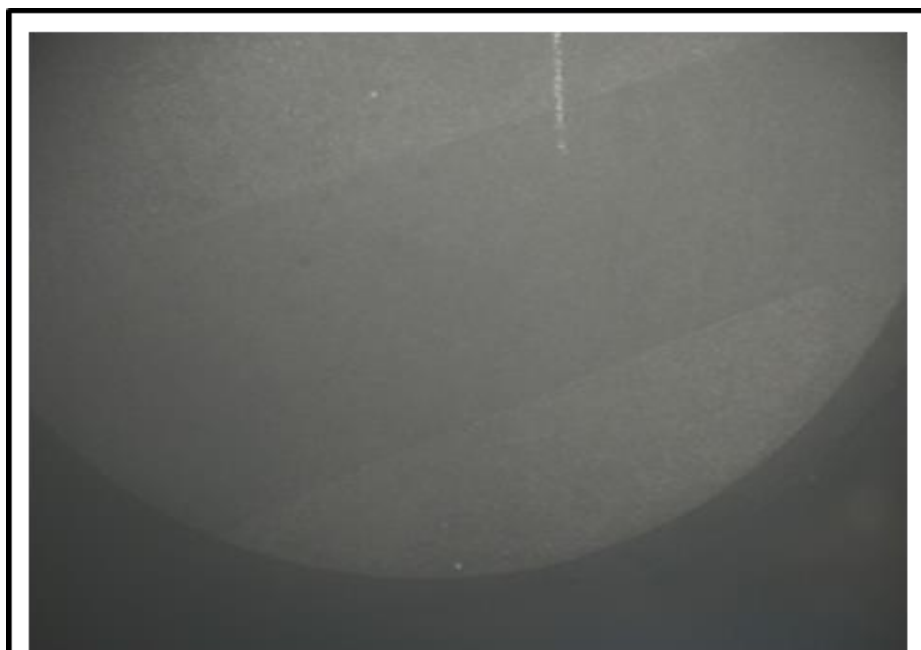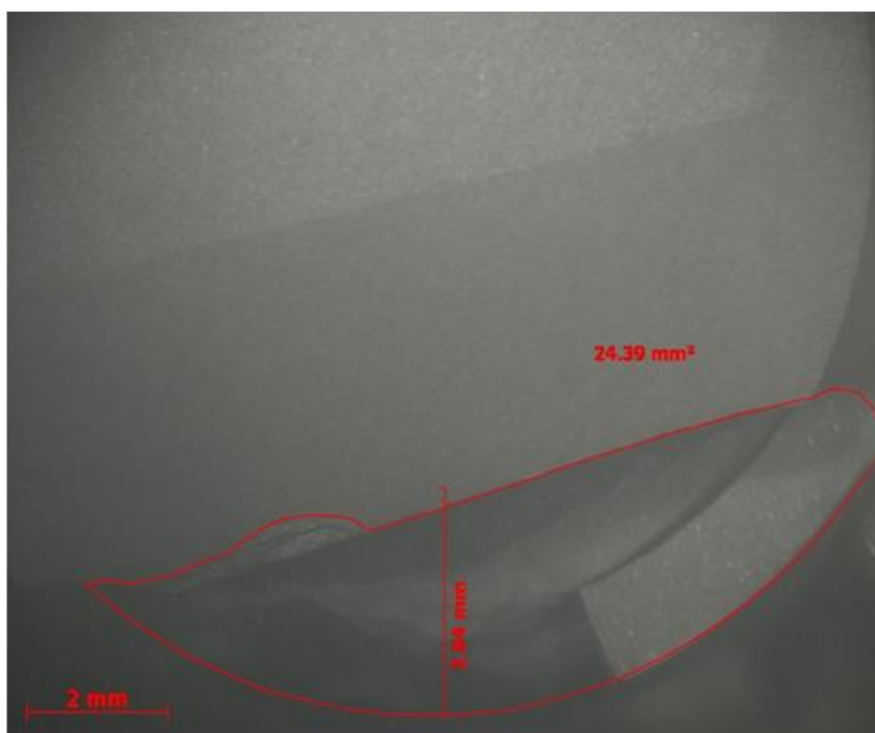
6 passes

Variant D: test 3

0 pass

90 passes

2.46 mm²

2 mm
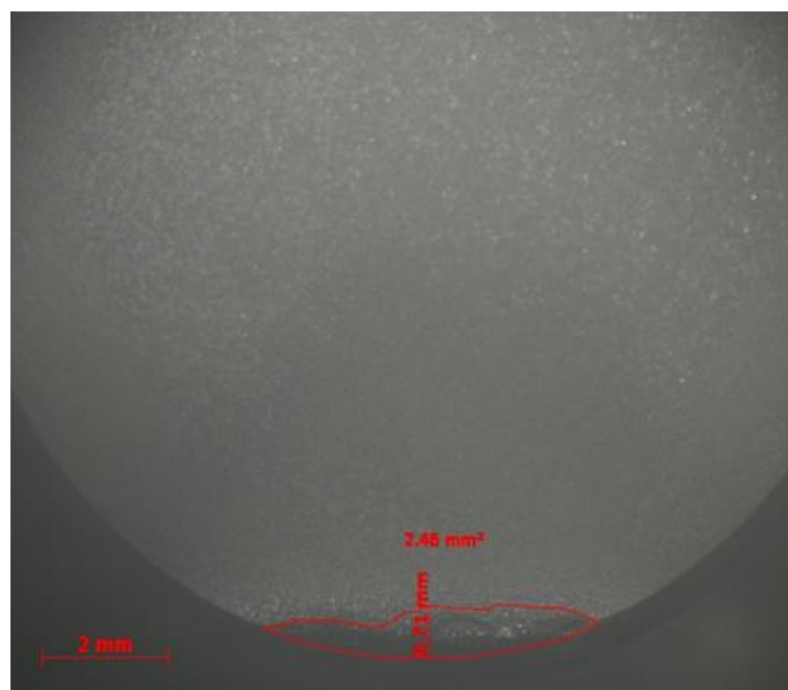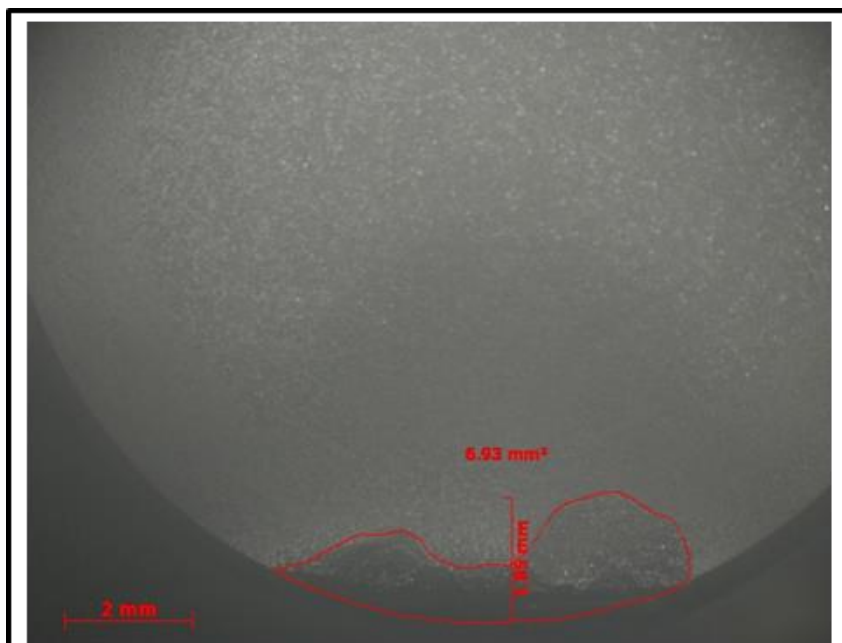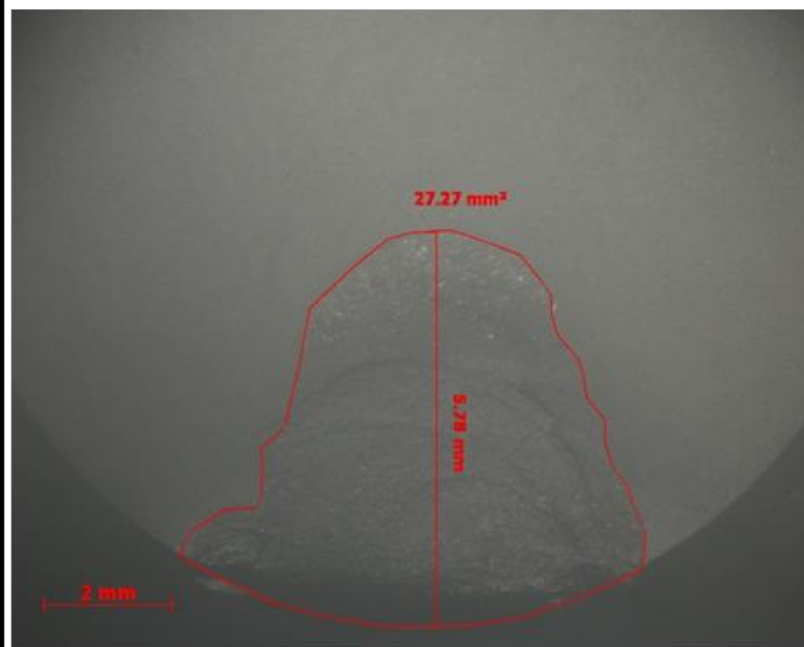
180 pass

270 passes

Variant C: test 1

0 pass

2.82 mm²

2 mm

90 passes

4.58 mm²

2 mm

120 pass

85.85 mm²

8.98 mm

2 mm
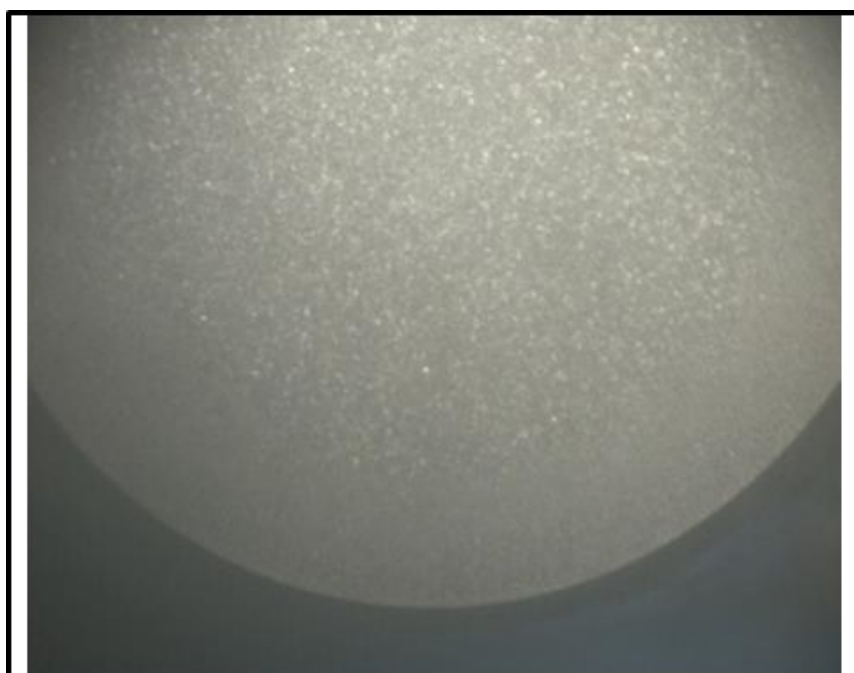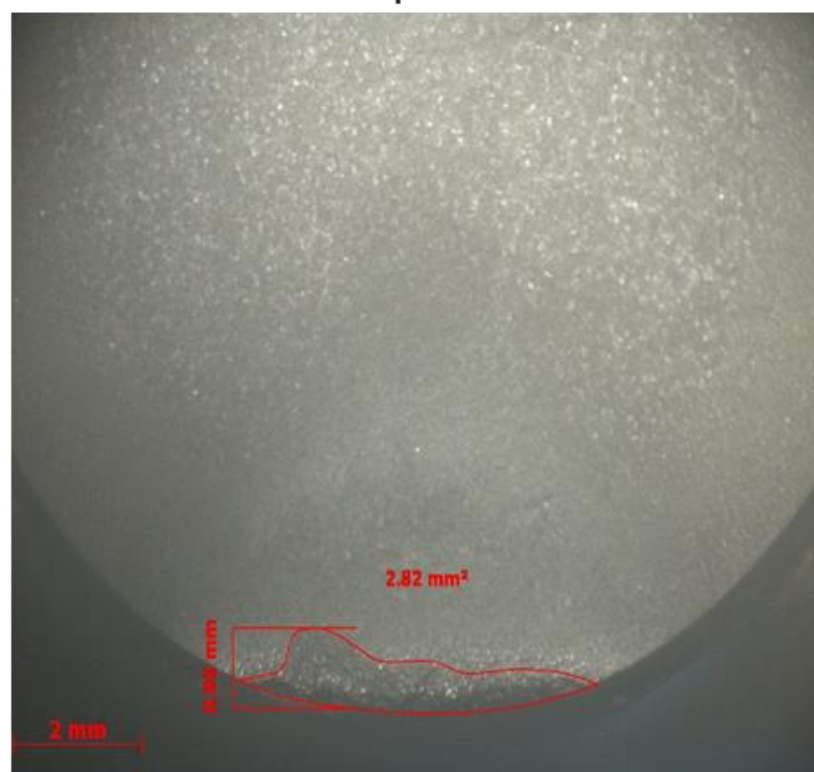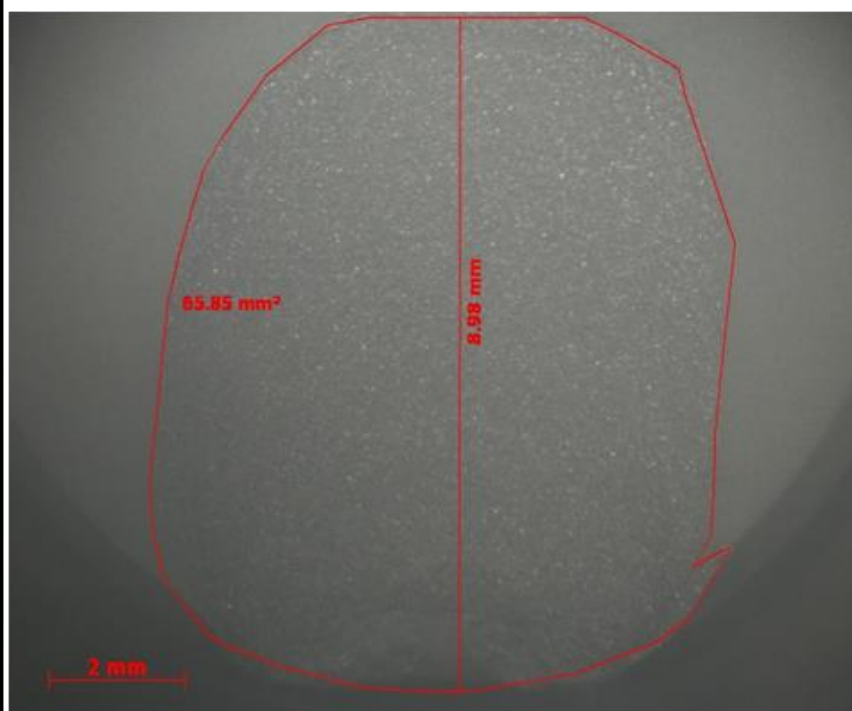
150 passes
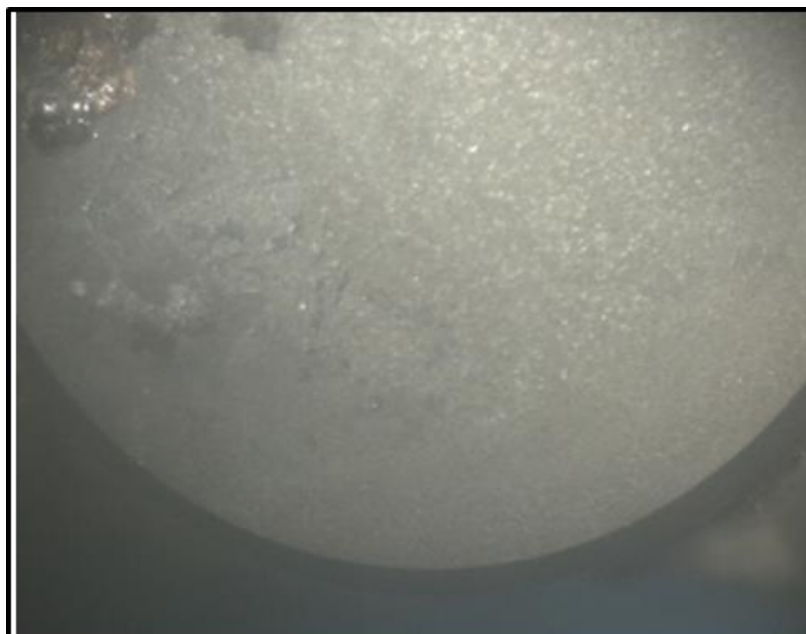
Variant C: test 2

0 pass
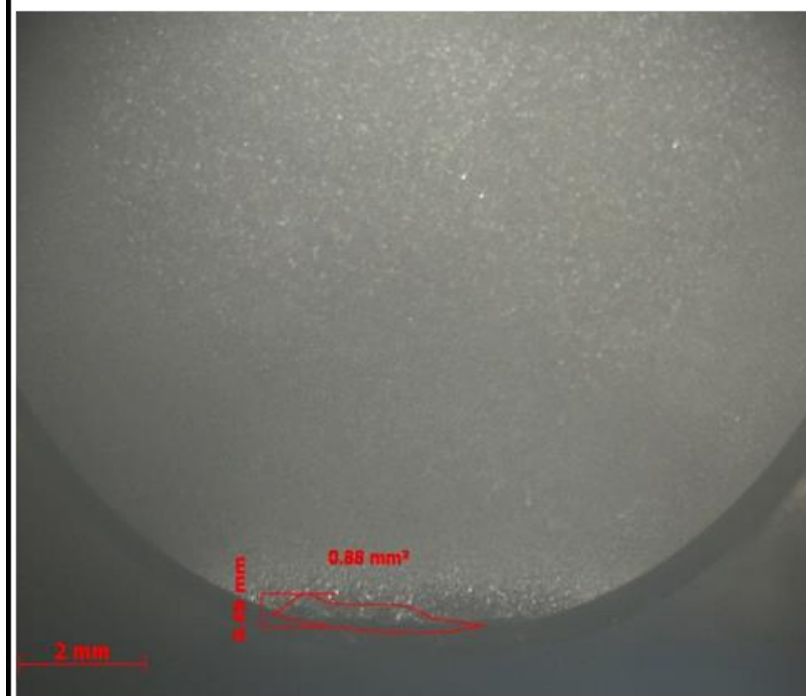
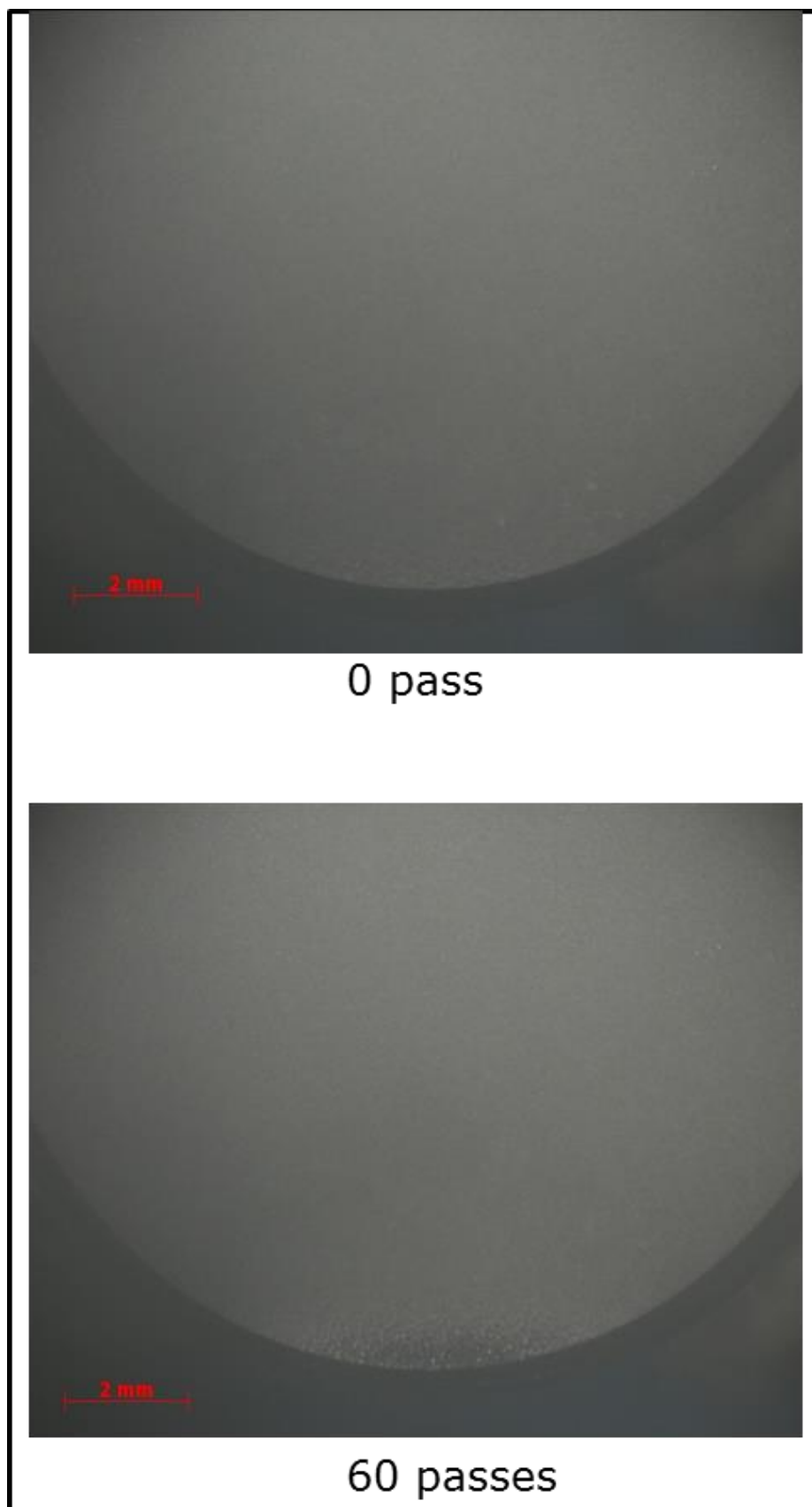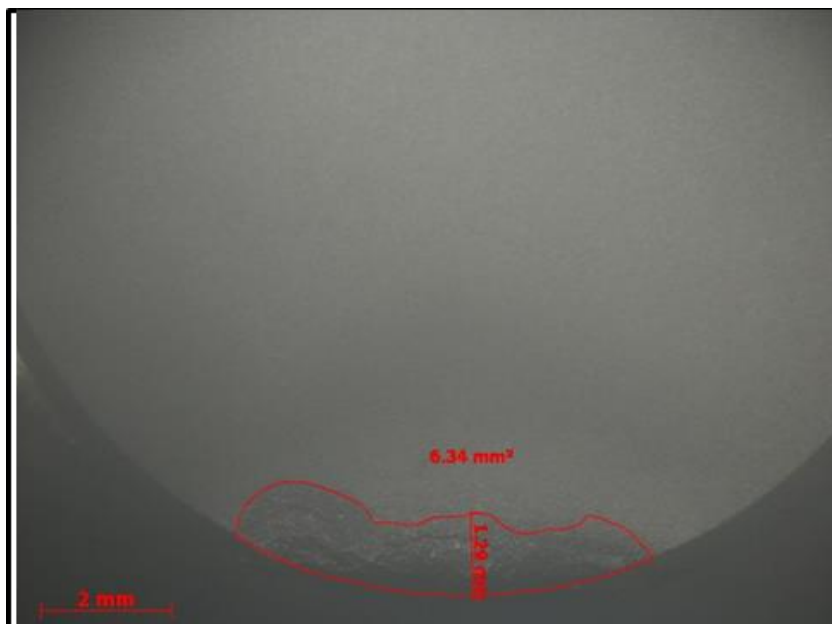90 passes
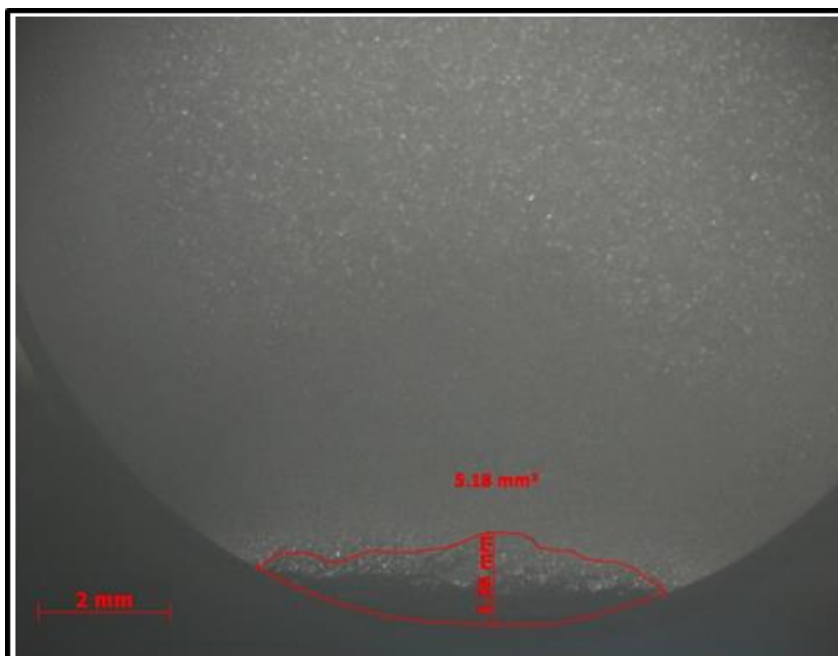
180 pass
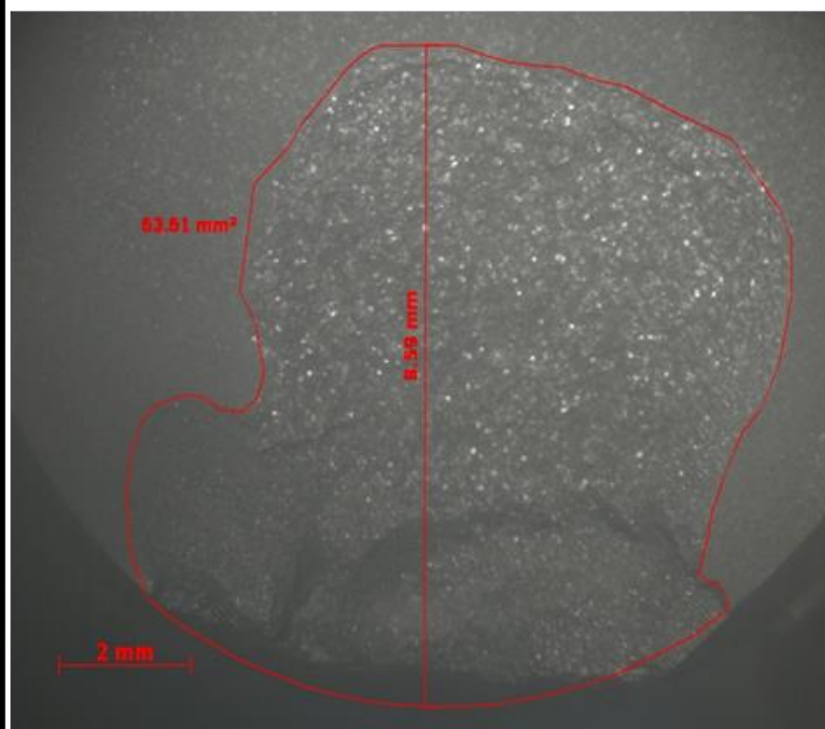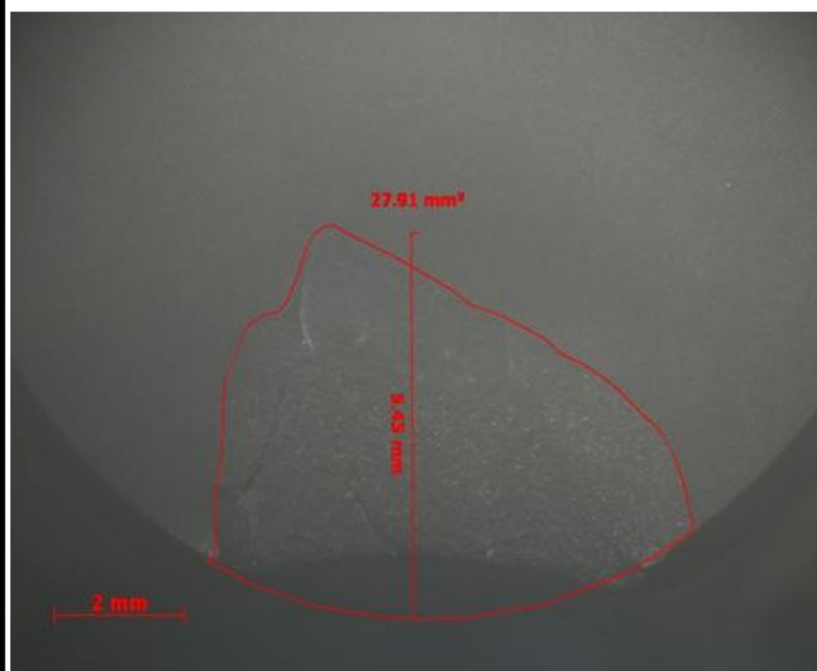
240 passes

Variant C: test 3

Variant A: test 1

2 mm

0 pass

2.31 mm²

2 mm
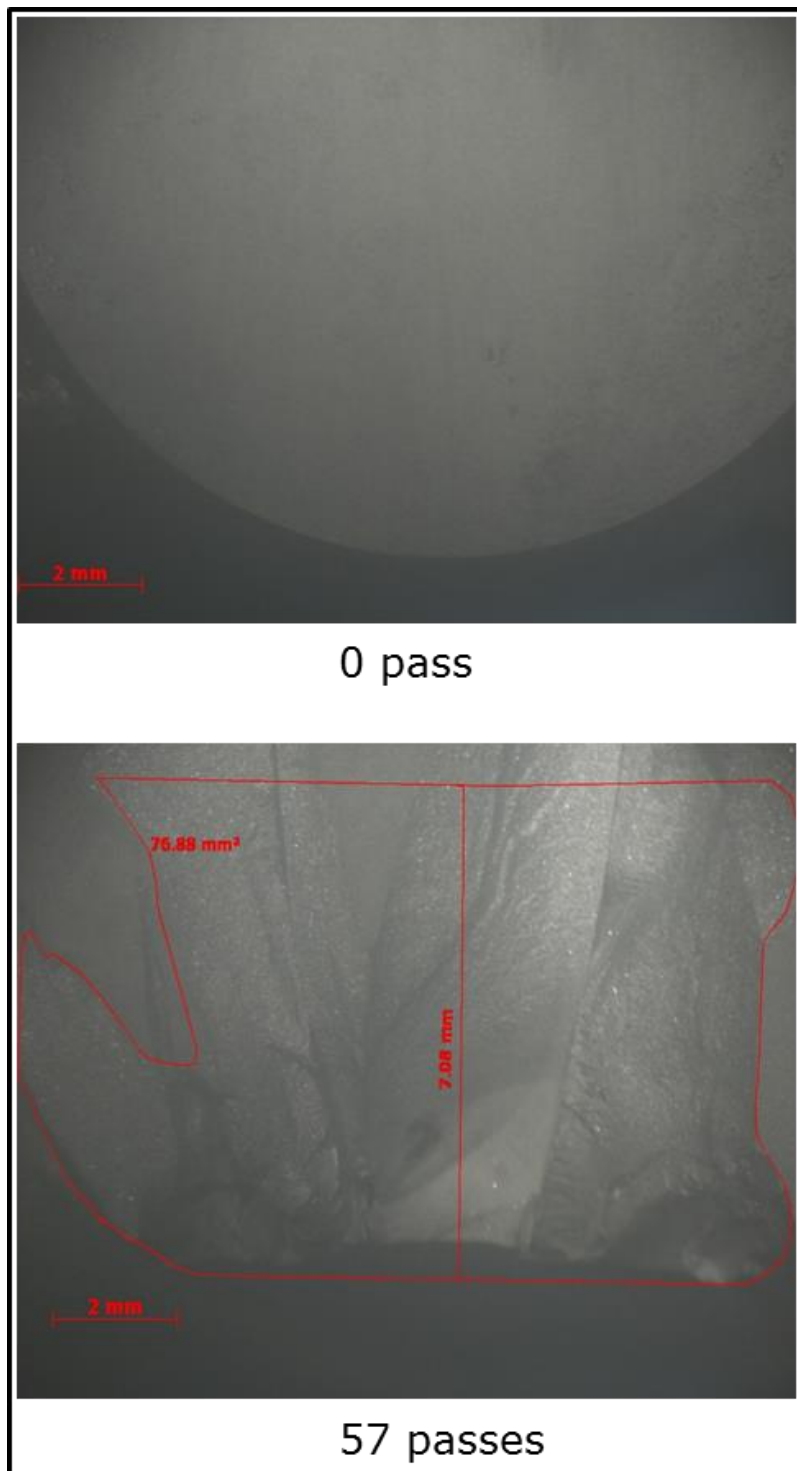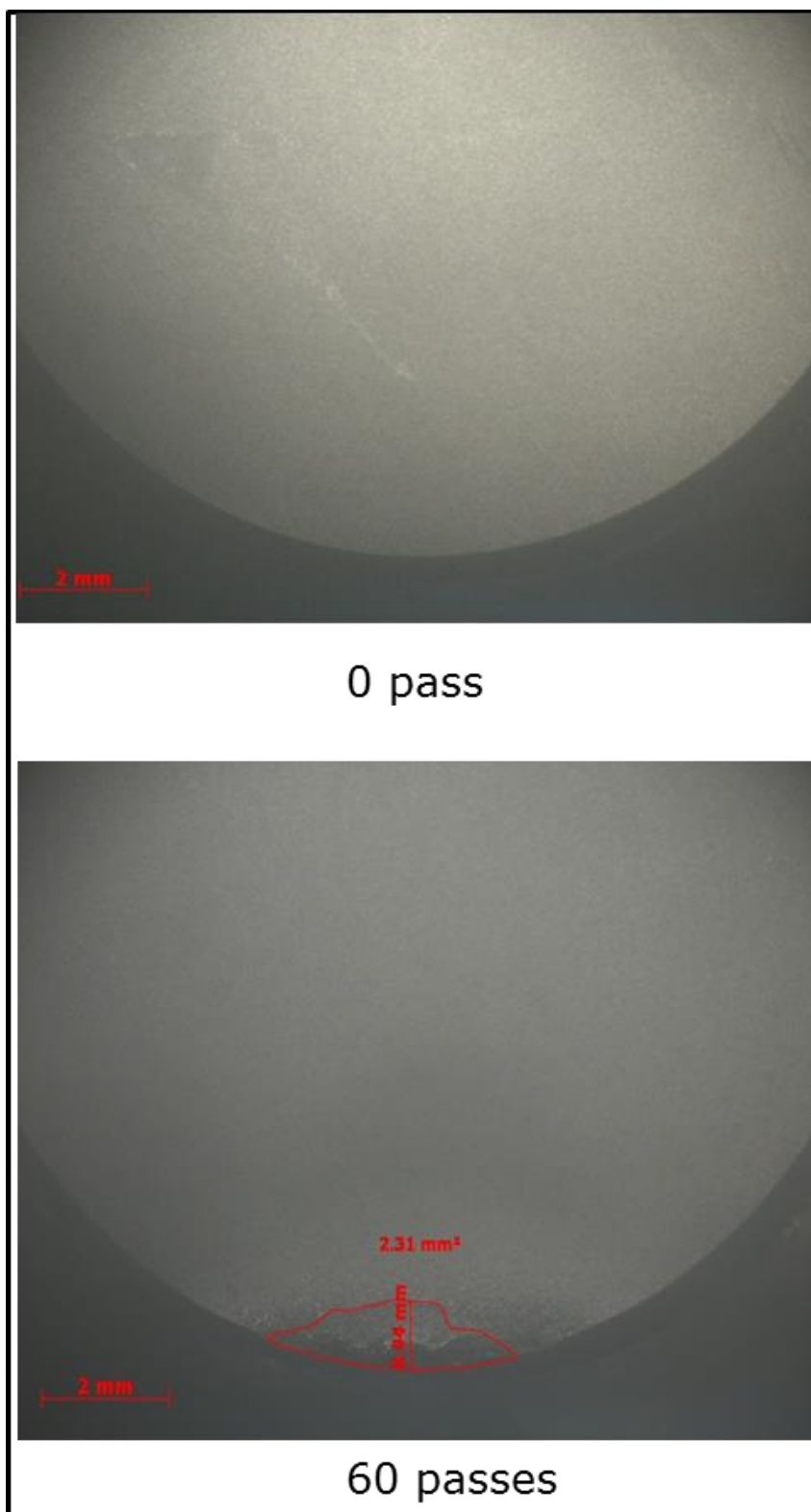
60 passes

90 pass

116 passes

Variant A test 2

0 pass

60 passes

90 pass

142 passes

Variant A: test 3