

# Deliverable 1.3: Prototypes and progress validation

Dissemination Level: Confidential (CO)

<u>Owner</u>	
Name:	Martin Lustfeld
Lead Beneficiary:	BOSCH
Phone:	
E-mail:	Martin.lustfeld@de.bosch.com
Context	
Author(s):	M. Lustfeld (Bosch); N. Dury (C4L); W. Scalbert (E6)
Work Package:	WP1
Task:	Task 1.3: Assessment and validation of technical progress
Document Status	
Version:	1.0
Last modified:	27/01/2017
Status:	Final
Approved by:	Marwan Abdou-Ahmed
Date Approved:	06/02/2017

**Declaration:** Any work or result described therein is genuinely a result of the Hiperdias project. Any other source will be properly referenced where and when relevant

# **Table of Contents**

1.	Scope
2.	Bosch KPI's (3D Silicon Processing)5
	2.1 KPI definition and assessment methods5
	2.2 KPI benchmarking6
	2.3 Relationship between productivity KPI1, surface roughness (KPI 4) and laser fluence
	2.4 Scalability of the productivity KPI10
3.	Class 4 Laser KPI's (Fine Cutting)13
	3.1 Definition of Benchmark Process
	3.2 Definition of goals
	3.3 Definition of measurement process
	3.4 Summary of KPI
4.	Element Six KPI's (Diamond Processing)
	4.1 Definition of Benchmark Process
	4.2 KPI Definition for Quantitative Progress Assessment
	4.3 Definition of Characterization and Measurement Methods
5.	Summary

# **Version History**

Version	Summary of Change	Written By	Approver	Date
0.1	First draft, BOSCH contribution	Lustfeld		13.12.2016
0.2	First draft, Element Six	W. Scalbert		03.01.2016
	Contribution			
0.21	First draft		F. Keating	13.01.2017
0.22	First draft		T. Scanlon	13.01.2017
0.3	Second draft	W. Scalbert		14.01.2017
0.4	Third draft	W. Scalbert		16.01.2017
0.5	C4L first draft	N. Dury		23.01.2017
0.6	Bosch update, summary,	M. Lustfeld		26.01.2017
	common formatting			
0.61	Layout correction	W.Scalbert		26.01.2017
0.62	Final Layout	M. Lustfeld		26.01.2017
0.7	Last input added	N. Dury		27.01.2017
0.8	Draft finalized & sent to	M. Lustfeld		27.01.2017
	coordinator for review			
1.0	First Issue		Marwan	06/02/2017
			Abdou-	
			Ahmed	

# 1. Scope

In order to quantitatively assess the progress of the laser machining processes within the course of HIPERDIAS, key performance indicators (KPI's) are defined. These relate both to quality and productivity requirements. Three applications are being pursued within HIPERDIAS (3D-processing of silicon, fine-cutting of metals, and laser ablation of diamond), focusing on different objectives not easily comparable to each other (Figure 1). Thus, for each application, individual KPI's have to be defined that will be tracked throughout the project. They support the decision-making regarding to the development and enhancement of the laser apparatus. Every single modification of the system and development effort should be reflected in an increase of the entirety of KPI's defined for each application.



Figure 1: HIPERDIAS project concept illustrating the relation of applications and process development

In this document, KPI's are defined and presented for each of the three HIPERDIAS applications. The application of KPI's for the assessment of progress of process development is illustrated by results of experiments most recently performed within HIPERDIAS. A short discussion and summary is given to compare these KPI's and to identify common objectives and discrepancies with respect to the development of the laser apparatus.

# 2. Bosch KPI's (3D Silicon Processing)

#### 2.1 KPI definition and assessment methods

For 3D-processing of silicon to be a viable manufacturing process, some requirements have to be met regarding to productivity and quality. These requirements have to be translated into KPI's that can be tracked throughout the project. There are thus productivity and quality KPI's. Furthermore, a KPI can either be of total or intrinsic nature. Exemplarily, the ablation rate of the laser system can either be expressed in total (mm<sup>3</sup>/s) or intrinsically referring to the laser power (mm<sup>3</sup>/J). The latter expression allows extrapolation of KPI's achieved with low-power experimental laser systems to the high-power prototype system pursued within HIPERDIAS.

Typical silicon structures considered for laser 3D-processing are characterized by lateral dimensions of up to several mm and a maximum depth of up to 1 mm, yielding an ablated volume on the order of magnitude of 10 mm<sup>3</sup>. About 700 such structures could be fit on a wafer that should be processed in a total time no longer than 2 h each. The corresponding average productivity KPI target is thus defined as  $\bar{V} = 1 \text{ mm}^3/\text{s}$ . Due to the fact that laser processing includes down times of laser beam repositioning and scanning acceleration, this average value requires a peak value three times as high based on processing experience. Thus, the targeted peak value is  $\dot{V}_{max} = 3 \text{ mm}^3/\text{s}$ .

Key Performance Indicator	Symbol	Unit	Target Value	Assessment methods
KPI1: average ablation rate	$\bar{V}$	mm³/s	≥1	white light interferometry
KPI2: peak ablation rate	<i>V̇</i> <sub>max</sub>	mm³/s	≥3	-
KPI3: shape deviation	$\delta_S$	μm	≤10 (waviness)	white light interferometry
KPI4: average surface roughness	Sa	μm	≤1	laser scanning microscopy
KPI5: thickness of surface damage	l <sub>d,sd</sub>	μm	≤1	energy-dispersive x-ray spectroscopy
KPI6: Surface defects > 1 μm	-	1/mm²	none	scanning electron microscopy
KPI7: min. achievable edge radius	r <sub>e</sub>	μm	≤ 200	optical microscopy
KPI8: max. edge-steepness	α <sub>e</sub>	degree	≥ 70	laser scanning microscopy

Table 1. Dosch Kri S. Dennition and minit values, and assessment methods	Table 1	: Bosch	KPI's: I	Definition	and lim	nit values,	and	assessment methods
--	---------	---------	----------	------------	---------	-------------	-----	--------------------

Regarding to the processing quality, shape tolerances should be reproducible within a tolerance of few µm. This includes a maximum target value in shape deviation due to waviness of  $\delta_S \leq 10 \ \mu m$  and a maximum tolerable average surface roughness of  $S_a \leq 1 \ \mu m$ . Additionally to geometric deviations, the wafer material may be modified by laser irradiation beneath the surface of the ablated structure. Such potential modifications include the oxidation of crystalline silicon which may be detrimental to the functionality of the structures. The maximum depth of such modifications should be less than  $l_{d,sd} \leq 1 \ \mu m$ . Intrinsically, geometries created by laser processing are limited with regard to the minimum achievable edge radius  $r_e$  and their maximum achievable edge steepness  $\alpha_e$ . These quantities depend on several laser beam characteristics such as the focal spot size and beam intensity. In practice,  $r_e \leq 200 \ \mu m$  and  $\alpha_e \geq 70^\circ$  are suitable limits for most potential

applications. However, surface defects such as cracks or pores of dimensions on the order of 1  $\mu$ m or larger correspond to non-functionality of the structure and thus have to be ruled out entirely.

The KPI's described above are required for productive industrial mass production and for meeting end-user-quality requirements of the Bosch demonstrator applications as summarized in Table 1.

In order to assess the KPI's, several different measurement systems were evaluated with respect to their accuracy and efficiency regarding to each KPI. The following methods were identified as suitable:

- White light interferometry: Allows fast profilometry of large (dimensions of several mm up to cm) ablated structures with high accuracy. Suitable to determine the total ablated volume of a sample (and thus, dividing by the ablation time, KPI1, the average ablation rate), as well as shape deviation (KPI3). Approximate values of the surface roughness (KPI4) and the wall steepness (KPI8) could also be obtained.
- Laser scanning microscopy: Profilometry of small sample areas (< 1 mm) at very high accuracy, thus suitable for very accurate measurement of the surface roughness (KPI4) and the wall steepness (KPI8).
- Scanning electron microscopy: Represents different materials in different grey shades. Very high spatial resolution and thus suitable to detect surface defects as small as 1 μm (KPI6).
- Energy-dispersive x-ray spectrometry: Detects chemical elements in the surficial area of a sample (resolution: about 5  $\mu$ m). This method can be used to detect surface contaminants such as oxygen (KPI5)
- Optical microscopy: Suitable for obtaining a general impression of the state of the sample as well as to quantify larger 2-dimensional features such as the edge radius (KPI7).

#### 2.2 KPI benchmarking

For benchmarking of the ablation process throughout the project, a model geometry was defined. The geometry covers a broad range of geometric features and machining challenges, such as a high total ablation volume, inclined planes, through holes, etc. with a high requirement of accuracy. The inclined plane especially requires minimum shape deviation with respect to the inclination angle, symmetry (left / right) and edge-steepness. The through holes mainly require high ablation rates, in order to remove the material until complete penetration of the wafer. The cross shape of the model geometry in addition allows for evaluating the quality of the beam deflection system, since a high number of corners / edges is required to be implemented with a high geometrical accuracy. This in turn requires highly precise positioning and fast switching of the laser beam. A depth profile of the model geometry is given in Figure 2a along with a microscopic image of the ablated geometry (Figure 2b).



Figure 2: Profile of model geometry (a) and structure machined into silicon wafer using parameters of Table 2 (b).

Process parameters	Symbol	Unit	Target Value	
wavelength	λ	nm	1030	(TRUMPF TruMicro 5050)
pulse duration	$ au_p$	ps	6	
repetition rate	f <sub>rep</sub>	kHz	400	
maximum power	P <sub>max</sub>	W	50	
focus radius	$d_0$	μm	70	
КРІ	Symbol	Unit	Measured	Target value
KPI1: average ablation rate	$\overline{V}$	mm³/s	0.045	1 (not met)
KPI4: average surface roughness	Sa	μm	1 (15 w/ defects)	< 1 (met except for defects)
KPI7: min. achievable edge radius	r <sub>e</sub>	μm	80	< 200 (met)
KPI8: max. edge-steepness	$\alpha_e$	degree	81	> 70 (met)

The microscopic image shows that the model geometry could be qualitatively reproduced by the laser process. All laser parameters along with selected KPI's are summarized in Table 2. In order to quantify KPI's 1,3,4, and 8, white light interferometry measurements were performed, the results of which are shown in Figure 3.



Figure 3: White light interferometry measurements of the ablated structure shown in Figure 2; a) model geometry and roughness values; b) edge steepness measurement

The KPI assessment immediately shows that the core productivity KPI1 (average ablated volume rate) was below the target value of 1 mm<sup>3</sup>/s. This, however, should have been primarily due to the comparably low power of the laser used in this work (P = 50 W). The intrinsic ablation rate was at 0.9 mm<sup>3</sup>/kJ and thus very close to the target average value of 1 mm<sup>3</sup>/kJ. If linear upscaling of these results to the target laser power of 1 kW was possible, the target productivity KPI would thus be reachable.

Considering the quality KPI4 (surface roughness), the target value of  $S_a < 1 \mu m$  was easily reached on most of the surface of the structure. However, few spots were detected at which the roughness severely deviated from this value. These deviations were due to surface defects of unknown cause. Currently, experiments are run with the objective of understanding the underlying physical mechanisms and to avoid this effect. Both the target values of KPI7 (edge radius) and KPI8 (wall steepness) were already met by the ablation process.

#### 2.3 Relationship between productivity KPI1, surface roughness (KPI 4) and laser fluence

Further fundamental experimental series were focused on determining both the intrinsic ablation rate and the surface roughness of different laser processes as a function of the laser fluence. For this purpose, solely square geometries with an edge radius of 1 mm each were ablated. The obtained values could be compared to both target KPI1 (the average ablation rate of 1 mm<sup>3</sup>/s) and target KPI2 (the peak ablation rate of 3 mm<sup>3</sup>/s) defined for the 1 kW system to be developed within HIPERDIAS. Within this section, these values are transformed to intrinsic quantities based on the target laser power of 1 kW (KPI1: 1 mm<sup>3</sup>/kW, KPI3: 3 mm<sup>3</sup>/kW) and compared to intrinsic quantities of these fundamental experiments.

In Table 3, Table 4 specifications of the three laser sources used for fundamental studies are summarized. Their major difference of interest in this section is the pulse duration ranging from 1 ps to 10 ps. Furthermore, the Picoblade laser is capable of running in burst mode, releasing a pair of pulses each clock step. The impact of using the burst mode on KPIs will be investigated in future work.

For the experimental series discussed in this section, the laser fluence was varied in a range between 0.1 and 3.0 J/cm<sup>2</sup> in steps of maximum 0.1 J/cm<sup>2</sup> with each laser source. Square geometries with an

Dissemination Level: Confidential

edge length of 1 mm were ablated for each fluence value. Subsequently, the depth of the structure was measured by microscopy to calculate the ablated volume. Using these data, the intrinsic ablation rate could be determined for every set of parameters as shown in Figure 4.

These results show:

- The intrinsic ablation rate heavily depends on the pulse duration. The shorter the pulses the higher the intrinsic ablation rate.
- The intrinsic ablation rate is a function of the laser fluence with an optimum being attained at a value in the range between 0.5 and 1.0 J/cm<sup>2</sup>.

Both results are in good agreement with the literature.

Table 5. Laser sources and respective realures used for fundamental experiments	Table 3: Laser sources and	d respective features	used for fundamental	experiments
---	----------------------------	-----------------------	----------------------	-------------

Laser source features	TruMicro	Picoblade	TruMicro FE
wavelength $\lambda$	1030 nm	1064 nm	1030 nm
pulse duration $\tau_{p}$	6 ps	10 ps	1 ps
repetition frequency $f_{_{\text{rep}}}$	400 kHz	1000 kHz	400 kHz
max. laser power P <sub>max</sub>	50 W	40 W	40 W
focal diameter d <sub>o</sub>	80 µm	55 µm	55 μm
burst mode	no	available	no

In comparison with the KPI's, the obtained intrinsic ablation rate is well above the required average value of 1 mm<sup>3</sup>/kJ, in particular for small pulse durations. On the contrary, the required peak ablation rate of 3 mm<sup>3</sup>/kJ was not reached in any configuration. Further process enhancement will be necessary to reach this KPI. A potential measure in this regard is using the burst mode of the laser source Picoblade to be elaborated in future work.



Figure 4: Intrinsic ablation rate of silicon ablation with different laser sources as a function of laser fluence

Additionally to productivity considerations, sample analyses revealed that the surface quality of the samples was immediately linked to the laser fluence as shown in Figure 5. For short laser pulses (1 ps), the roughness of the ablated surface increased approximately linearly with the laser fluence

starting at very low values, but crossing the quality KPI4 value at about 0.7 J/cm<sup>2</sup>. Micrographs in the insets illustrate the change in surface quality from very smooth to rough with many surface defects.



Figure 5: Roughness of ablated silicon surfaces as a function of laser fluence for different laser pulse durations.

At longer pulse duration (6 ps and 10 ps), the relationship between surface roughness and laser fluence evolves very differently. For a laser fluence below 0.7 J/cm<sup>2</sup>, the surface roughness steeply increases with increasing fluence. Crossing the fluence value of 0.7 J/cm<sup>2</sup>, however, the surface roughness drops to a value well below the quality KPI, representing a high-quality surface obtained by laser ablation. This abrupt change in quality may be explained by a change of the governing processing regime as investigated in current work. The first results presented in Figure 5 show, however, that physical mechanisms important for the KPI's are not linear in general and require careful investigation in the ongoing work.

In this section, intrinsic ablation rates (measured in mm<sup>3</sup>/kJ) were discussed. However, for industrial applications, the total ablation rate (measured in mm<sup>3</sup>/s) is the KPI decisive for process productivity. Whether or not the intrinsic values are scalable to higher laser power thus needs to be carefully investigated. The following section documents an attempt in this regard.

#### 2.4 Scalability of the productivity KPI

Upscaling of the process to higher values of laser power appears to be necessary to achieve the productivity KPI1. Therefore, a series of experiments were run using the high-power USP laser available at the IFSW (USTUTT). The objective of these experiments was to determine the maximum achievable ablation rate without regard to the other KPI's. The corresponding laser process parameters are summarized in Table 4.

For these experiments, square geometries with an edge length of 10 mm were ablated instead of the model geometry presented in Figure 2. The maximum experimental time per processing instance was 30 s. Upon processing, wafer failure by through-cracks was observed for certain parameter combinations that yielded high specific energy input into the wafer, corresponding to low feed velocities. At the highest attainable laser power of 670 W, a minimum feed velocity of 4 m/s was required to avoid such cracking. Furthermore, it could be observed that the surface roughness decreased for increasing feed velocity values, saturating at  $S_a$  of a few  $\mu$ m at  $v_s \ge 4$  m/s (Figure 6b).

**Dissemination Level: Confidential** 

#### Table 4: Laser process parameters using the IFSW high-power USP laser system

Process parameters	Symbol	Unit	Target Value	
wavelength	λ	nm	1030	Thin-disk multipass amplifier developed by IFSW in collaboration with TRUMPF (who provided the seed laser: trumircro 5050)
pulse duration	τ <sub>p</sub>	ps	6	
repetition rate	f <sub>rep</sub>	kHz	300	
maximum power	P <sub>max</sub>	W	670	
focus radius (x)	d <sub>fx</sub>	μm	140	(Scanlab IntelliScan 20)
focus radius (y)	d <sub>fy</sub>	μm	420	
focal length	F	μm	340	
beam quality factor	M <sup>2</sup>	-	3	
Si wafer diameter	D	mm	200	
Si wafer thickness	h	mm	1.35	
КРІ	Symbol	Unit	Measured	Target value
KPI1: average ablation rate	$\overline{V}$	mm³/s	0.29	1 (not met)

The maximum attainable average ablation rate was determined to be 0.29 mm<sup>3</sup>/s without wafer cracking. The system thus outperforms the experimental setup used for the model geometry by a factor of 6.4. However, the intrinsic ablation rate of 0.4 mm<sup>3</sup>/kJ is far below that of the former system. This discrepancy implies a lack of scalability between the two systems. A major contribution to this discrepancy may be due to scanner dynamics. At higher scanning velocities, down times between laser processing steps due to acceleration and return moves of the laser beam are much higher as a share of total processing time than at lower scanning speed. For an accurate comparison of both systems, the scanning speed should be chosen similar for both laser systems. However, reducing the scanning speed of the high-power laser system would require active cooling of the wafer in order to prevent cracking.



Figure 6: Measurement data of structures machined with the high-power USP laser system available at IFSW; a) surface roughness measured by white light interferometry on structures created at different feed velocities; b) corresponding surface roughness values

# 3. Class 4 Laser KPI's (Fine Cutting)

In the following paragraphs, key performance indicators (KPI) will be listed and defined in order to qualitatively and quantitatively assess and validate the technical progress of cutting of fine watch components using ultra-short pulsed Laser technology developed within HIPERDIAS associated to diverse cutting methods and optics. These KPI's shall assess productivity and quality performances achieved thanks to a high power femto laser source.

- 1. The cutting process with the HIPERDIAS Laser will be compared to the current laser process in use: fast cutting with fiber laser; cutting with USP laser and Scanner; Cutting with USP laser and trepanning optic.
- 2. KPI's will be defined accordingly to the performances achieved by the current cutting process. But also compared to alternative cutting technologies as EDM or milling. Nevertheless the laser process remains the benchmark for this application.
- 3. These KPI's will be measured to assess the technical progress along the course of the project; the same measurement method shall be used all along the project as well as for the benchmark process.
- 4. Besides the specific abilities of the new HIPERDIAS laser, the general scalability of the cutting process shall be evaluated: is the process finally limited by the laser, by the optical or motion system, or by the physic of the process itself. Answering this question will enable to give the direction of further research. Should the limitation be the 200W laser, further cutting trials should be done on the 1000W laser at the end of the project.

#### 3.1 Definition of Benchmark Process

#### Description of laser system used

#### Table 5: Specifications of the benchmarking laser

Laser specification	Symbol	Unit	Value	Remark
Wavelength	λ	nm	1030	Light Conversion Carbide
Pulse duration	τ <sub>p</sub>	fs	290-10'000	Tuneable pulse duration
Repetition rate max.	f <sub>rep</sub>	kHz	1000	
Maximum average power	P <sub>max</sub>	W	5	Eventually a 20W version of the laser will also be used (E <sub>max</sub> =200µJ)
Pulse energy max.	E <sub>max</sub>	μJ	85	@60kHz (5 µJ @1 MHz)
Beam quality factor	M <sup>2</sup>	-	1.1	
Focus radius (x)	d <sub>fx</sub>	μm	25	
Focus radius (y)	d <sub>fy</sub>	μm	25	
Focal length	F	μm	100	Scanlab ScanCube 14 and Intelliscan 14

Laser specification	Symbol	Unit	Value	Remark
Wavelength	λ	nm	1030	
Pulse duration	τ <sub>ρ</sub>	fs	290- 500	Tuneable pulse duration to fit different materials properties
Repetition rate max.	f <sub>rep</sub>	kHz	1000	
Average power	P <sub>max</sub>	w	5-20	Power for one working station. Please note that in order to reach the right process time system with energy sharing may be used
Pulse energy	E <sub>max</sub>	μJ	40-60	
Beam quality factor	M <sup>2</sup>	-	1.1	
Focus radius (x)	d <sub>fx</sub>	μm	15-30	
Focus radius (y)	d <sub>fy</sub>	μm	15-30	
Focal length	F	μm	80-160	

#### Table 6: Typical parameter needed for fine cutting with current systems

#### Description of benchmark process and product

Currently, the cutting process for gears and fine watch elements happens on 2 main levels:

- Laser cutting process: Either a gas assisted fiber laser cutting, scanner cutting, or trepanning optic cutting.
- Traditional cutting: EDM, milling or punching.

The benchmark for this application will be the laser cutting process performed with the current laser source: base trials and quality definition will be performed on a 5W and eventually a 20W femtosecond laser with pulse duration adjustment possibilities from 290fs up to 10ps.

Nevertheless, some comparison will be made with traditional cutting process, but on a rougher basis as C4L has only limited access to such processes.

The benchmark will be performed on parts made of brass, stainless steel and a third non-metallic material later defined according to its availability (ceramic, sapphire or carbon-fibre based). Table 7 summarizes the benchmarked process specifications.

It is important to note that with the current processes that are benchmarked, the main limitation is the impossibility to reach all specification thanks to one process only.

Trepanning optic cutting brings the best quality at the best speed, fibre laser cutting gives a high speed result but with too large tolerances and a quality requiring more post processing. Finally, scanner cutting gives reasonable quality and speed but with a taper that remains out of the accepted tolerances for some parts.

The goals will be to bring all the specifications within one single process thanks to the HIPEDIAS laser and therewith, raise the laser cutting standards.

Key Performance Indicator	Unit	current process value		
Benchmarking shape Standardized watch arm Standardized Gear	4	See The Light		
Part thickness	mm	0.1 – 0.3		
Part dimensions	mm	Gear diameter: 5-10 Watch arm length: ca. 20		
Material covered Metal,		c, sapphire, carbon		
	Non-metal par	t geometry may vary slightly in dimensions.		
General dimensions tolerances	μm	From ± 5 to ± 20		
Specific dimensions tolerances	μm	+- 5		
Smallest holes	μm	From 50 to 100		
Maximal side steepness (taper)	0 to 10°			
Average cutting speed	mm/min	USP laser: ≤50		
(relative to shape and thickness)		Fiber laser: 300 but with insufficient quality		
Shape deviation	μm	+- 5 according to laser process		
Surface roughness (non-functional)	μm	0.4 (N5)		
Surface roughness (functional)	μm	0.1 (N3)		
Surface roughness values indicated are measured after washing and post treatment.				
The goal of the new process will also be	to reduce this p	post processing		

Example of parts cut with fibre laser	Example of part cut with scanner and fs laser
Example of part cut with trepanning optic	Example of part cut with traditional technology and post processed
THESE PICTURES A	RE CONFIDENTIAL

#### Figure 7: Comparison of product appearance using different cutting processes

Project: HIPERDIAS Grant Agreement No: 687880

#### 3.2 Definition of goals

#### 3.2.1 Productivity goals

<u>Process time</u>: the goal with the HIPERDIAS Laser will be to reach the process speed of a fiber laser process, with the best quality of the femto laser process.

Examples of process time:

Fiber laser: 0.25mm – brass – 20mm long watch arm – 150W laser – 15s

Femtosecond laser: 0.25mm – brass – 20mm long watch arm – 20W laser – 40s – 150s

Handling time: NC

<u>Post Processing</u>: washing, vibratory grinding, electro-polishing tolerated.

#### 3.2.2 Quality goals

#### Table 8: Summary of quality specifications to be achieved

Key Performance Indicator	Unit	current process value
Benchmarking shape Standardized watch arms Standardized Gear	4	See The Light
Parts thickness	mm	0.1 - 0.3
Part dimensions	mm	Gear diameter: 5-10 Watch arm length: ca.20
Material covered	Brass, stainless	s steel, one non-metallic material to be defined
General dimensions tolerances	μm	From ± 5 to ± 20
Specific dimensions tolerances	μm	+-2
Smallest holes	μm	From 50 to 100
Maximal side steepness (taper)	Related to the	tolerances; 1° when not specified otherwise
Average cutting speed (relative to shape and thickness)	mm/min	≥300
Shape deviation	μm	+- 2
Surface roughness (non-functional)	μm	0.4 (N5)
Surface roughness (functional)	μm	0.1 (N3)

Grant Agreement No: 687880

#### **3.3 Definition of measurement process**

#### Surface roughness

A 3D Laser scanning microscope (VKX series from Keyence with x20 and x50 magnification) without filters will be used.

#### Tolerances

A 3D Laser scanning microscope (VKX series from Keyence with x10 magnification) and an optical microscope Leica, x4 magnification will be used.

#### Taper

Entrance side and exit side measurement with a 3D Laser scanning microscope (VKX series from Keyence with x10 magnification) and optical microscope Leica, x4 magnification.

#### 3.4 Summary of KPI

No	КРІ	KPI Values for success	Validation Status
		Expected : ≥ 0.400 mm/min	
1	Cutting speed	Validated : ≥ 300 mm/min	9
		Non validated : < 300 mm/min	
		Expected : washing	9
2	Post Processing/Cleaning	Validated : vibratory grinding (electro- polishing for some parts only)	$\bigcirc$
		Non validated : additional work/cost	
	Production Cost	Expected : ≤ 0.4	
4	In €, for 1 watch arm 0.15mm thick – 20mm long – batch of 1000	Validated : ≤ 0.45	9
		Non validated : > 0.45	

Grant Agreement No: 687880

No	КРІ	KPI Values for success	Validation Status
	Surface Roughness	Expected : Ra 0.1 (N3) / 0.4 (N5)	
5	(functional / non- functional)	Validated : Ra	0
		Non validated : Ra $\geq$ 1 $\mu$ m	•
		Expected: < +/- 2 μm	
6	Shape deviation	Validated : < +/- 5 μm	9
		Non validated : > +/- 5 $\mu$ m	
7	Taper	Expected : 0°	
		Validated : within dimension tolerances	$\bigcirc$
		Non validated : above dimension tolerances	•
		Expected : none	
8	Colouration	Validated : washable surface oxidation	$\bigcirc$
		Non validated : persistent surface oxidation	
	Untying of the part	Expected : fall down in US-bath	
9		Validated : fall down in US-bath with separation cut	0
		Non validated : mechanical removing	

Project: HIPERDIAS Grant Agreement No: 687880

## 4. Element Six KPI's (Diamond Processing)

In the following paragraphs, key performance indicators (KPI) will be listed and defined in order to quantitatively assess and validate the technical progress of the new PCD polishing process using the ultra-short pulsed Laser technology developed within HIPERDIAS. These KPI's will assess productivity and quality performances achieved by this new Laser-polishing process of diamond material.

- 1. This new Laser-polishing process will be compared to the current mechanical polishing process used in Element Six. So the current mechanical polishing process represents the benchmark process and must firstly be defined.
- 2. So KPI's can be defined accordingly to the performances achieved by the current polishing process. And the measure of success of the Laser-polishing process will be based on higher performances than the mechanical polishing process.
- 3. These KPI's will be measured to assess the technical progress along the course of the project; all characterisations and measurements methods used to measure those KPI's like testing structures, prototypes, quantifiable measurements and characterisation procedures must be clearly defined.

#### 4.1 Definition of Benchmark Process

#### 4.1.1 Description of benchmark process and product

Currently in Element Six, the polishing process follows a mechanical method of polishing involving a micro-chipping phenomenon through friction between the PCD surface to be polished and the diamond grit on the surface of rotating polishing wheel.

The polishing process in Element Six standing as benchmark process is the process used to polish the Element Six *Syndite* products representing our benchmarking product as defined in deliverable D1.1. The so called *Syndite* products are 76 mm diameter and 1.6 mm thick discs made of two layers: a 0.5 mm thick PCD layer and a carbide tungsten layer.

The polishing benchmark process is displayed in Figure 8. Five *Syndite* discs can sit on a diamond grit polishing wheel, so each machine can polish up to five *Syndite* discs at once.

#### Grant Agreement No: 687880

# Deliverable 1.3: Prototypes and progress validation Dissemination Level: Confidential





#### Figure 8: Picture of a polishing machine in Element Six

#### 4.1.2 Surface roughness

After polishing, the optimum global surface roughness should be less than 0.011  $\mu$ m:

• Ra = 0.011 μm

According to 3D Laser scanning measurements (VKX series from Keyence with x50 magnification) of a mirror polished surface from a Syndite product (Figure 99):

- Sa = 0.01 μm
- Sp = 0.02 μm



• Rz = 0.05 μm

- Sz = 0.12 μm
- Sv = 0.07 μm

Ra = 0.01 μm

Rq = 0.01 μm

Page 21 of 32

Grant Agreement No: 687880

• Rv = 0.03 μm

# Figure 9: Picture of a polished surface and surface roughness measurements

#### 4.1.3 Material removal rate

To achieve such surface roughness, the polishing process can take up to several hours. Currently the processing time for this operation is anywhere between 12-18 hours on one machine holding 5 discs.

Processing time = 15.0 hours for 5 discs

Within Element Six, the material removal rate is estimated to 1  $\mu$ m per hour across the all top surface of a diamond disc as the Syndite product has a standard size. So, for 15 hours of polishing, 15  $\mu$ m in depth are removed across the 76 mm surface of a disc to reach a mirror optical surface finish.

- Material removal = 68.0 mm<sup>3</sup> per disc
- Material removal rate = 1.26 x 10<sup>-3</sup> mm<sup>3</sup>/s per disc

So, in total, as polishing machines can process 5 discs at a time, the overall removal rate of the benchmark process is:

- Overall material removal rate =  $6.30 \times 10^{-3} \text{ mm}^3/\text{s}$ 

#### 4.1.4 Handling time

Removal rate of the process itself is low and explains the necessity of the research and development of a new process technology to replace this mechanical process. But, furthermore, time required to set up products and diamond wheel on the polishing machine is long. It requires a high amount of manual work: the wheel has to be levelled with a drop gage to ensure flatness and good stability of the polishing process. And, as the wheel collapses during the process, the polishing wheel must also be dressed and reset up every 3 hours. Meanwhile, the discs must be removed to reapply some fresh conductive heat grease behind the discs and their diamond surface must be cleaned up to ensure no contamination between fresh polishing wheel and disc surface.

Element Six has estimated the total handling time to:

- Total handling time = approximatively 75 min for 5 discs

#### 4.1.5 Post processing time

The discs must be cleaned out before inspection: the conductive grease wiped out of the back carbide tungsten face and around the edges, and the PCD top surface cleaned out with alcohol. It is once again a time consuming work.

Element Six has estimated the post processing time:

- Post processing time = approximatively 4 min per disc

#### 4.2 KPI Definition for Quantitative Progress Assessment

#### 4.2.1 KPI for Productivity Assessment

The main KPI to evaluate the productivity of the new Laser polishing process is obviously the material removal rate. It must at least be superior to overall material removal rate achieved by a polishing machine capable of polishing 5 discs at once, an inferior value would be considered as a failure of the project. It is expected to reach 0.150 mm<sup>3</sup>/s per disc, a value 10 times higher than current value would be accepted and validated by Element Six.

No	КРІ	KPI Values for success	Validation Status
1	Material Removal Rate	Expected : > 0.150 mm <sup>3</sup> /s per disc	9
		Validated : > 0.075 mm <sup>3</sup> /s per disc	<u> </u>
		Non validated : < 0.007mm <sup>3</sup> /s per disc	
2	Total Handling Time	Expected : < 10 min per disc	•
	-	Validated : < 20 min per disc	9
		Non validated : > 20 min per disc	
3	Post Processing/Cleaning	Expected : no post processing	9
	lime	Validated : quick surface cleaning	<u> </u>
	Non validated : No additional work/cost	•	
4	Production Running Cost *	Expected : < 5\$ per disc	•
		Validated : < 9\$ per disc	$\bigcirc$
		Non validated : > 18\$ per disc	

Table 9: Summary list of the KPI for productivity assessment

\* Cost calculated in US dollars according to Element Six standards

The handling time is an important factor and is expected to be considerably reduced compared to the very heavily manual benchmark process. Indeed, if the material removal rate is superior to the benchmark process but the new process requires a longer overall handling time, the project will not be validated. This handling time of the Laser polishing process includes set up time of disc/machine as long as topography measurements of the diamond surface. Measurement process and set up time shall not exceed 10 minutes per disc, leaving a topography measurement time of 5 minutes considering an optimal set up time of 5 minutes per disc.

The Laser polishing process could change the way the disc must be post processed/cleaned, so the current post processing must be reviewed and might have to be reconsidered depending on the surface state after Laser polishing process. The Laser polishing is expected to be free post

processing/cleaning. However a very simple and fast clean-up is tolerated like swiping the diamond surface with alcohol to remove the graphite dust/particles formed on the top surface of the disc after Laser-ablation of the diamond material (no water can be used or will cause stains on PCD surface). No additional manual work/cost to the current post processing must be added by switching from a mechanical to Laser technology process.

The production running cost is the final productivity KPI necessary to validate project and represents its most important measure of success. A total running cost of \$5 per disc is expected including post processing/cleaning, however a running cost below \$10 per disc would be accepted and validated. A running cost over \$18 would represent an irremediable failure of the all project to Element Six point of view.

#### 4.2.2 KPI for Quality Assessment

The main KPI to evaluate the quality of the Syndite product after the Laser ablation is the surface roughness. It will be the main parameter driving the development progress of the project. And its value through the project will condition the conduct of the project and the way of measuring the success of the projects.

- The PCD top surface is expected to have an optical mirror aspect (Ra < 0.011 μm) at the end of the Laser ablation, as currently reached by mechanical process. Nevertheless, optical mirror finish is not only defined by a very low surface roughness but also by the complete uniformity of its surface: the surface looks uniform without lines or traces of machining directions so it displays a high proportion of specular reflection and gives a mirror effect. This represents the ultimate objective and will fully validate the project as a success.</li>
- However, if an optical mirror finish cannot be directly achieved after Laser ablation, some mechanical polishing can be considered to follow the Laser ablation to finally achieve a polished surface state. Then this dual Laser/mechanical polishing process will be studied as one process and KPI will apply to the dual process.
- So a near optical mirror finish (Ra <  $0.100 \mu$ m) would be accepted and be validated as long as this dual Laser/mechanical polishing process meet all the other KPI. It will be studied as one process to measure all other productive and quality KPI to assess the success of the project.

Shape deviation was mentioned as a requirement in deliverable D1.1 and will stand as a KPI to be only applied in the case of a necessary mechanical polishing step following Laser ablation:

- For non-achievement of an optical mirror surface finish on diamond surface.
- For not meeting requirements in terms of diamond material chemical composition and microstructure (cf. KPI8 and KPI 9 descriptions).

Before mechanical polishing, the diamond surface must present a domed shape to allow high quality mechanical polishing: the ideal shape is a domed profile with a micrometric height (Figure 1010). In the case of a mechanical step following Laser ablation, the Laser process will have to machine a dome shape on the diamond surface while smoothing it. The ideal profile will have to be reidentified relative to the surface quality achieved after laser processing. This profile will remain a dome with a new ideal height to be studied. The new shape will have to be repeatedly achieved by the laser system within a 2  $\mu$ m accuracy as the new ideal dome height will remain in a micrometric range. A 5  $\mu$ m deviation from this identified new ideal height will still be accepted but inferior accuracy will not result in a quality mechanical polish.

Grant Agreement No: 687880

The polished surface quality is assessed by the importance of observable visual defects as listed in



Figure 10: Ideal Syndite surface profile

the internal Element Six document *Sn285 - 3* 001\_20160107 (the most significant visual defects are highlighted in deliverable D1.1). Visual defects are detected during quality inspection which occurs after Laser ablation and post processing of the discs, the discs are sorted out according to the defects severity. At the end, a pass rate, equal to the percentage of discs which pass the quality

inspection, is calculated and gives an assessment on the performed polished surface quality. So a pass rate will also be defined as a KPI for the project to assess the polished quality given by Laser ablation.

Low heat is expected to be applied on the diamond surface as the ultra-short Laser pulsed ablation should be a "cold" process. So the diamond material is expected to undergo neither thermal damage nor mechanical damage susceptible to create visual defects as severe as usually observed after mechanical:

- like cracks (due to high heat induced from friction between diamond grit and diamond disc)
- or pits and chipping (from high constraint mechanical force breaking and pulling out some material out of the surface).

In conclusion a 100% pass rate is expected. However material can sometimes be of lower quality: inhomogeneous polycrystalline diamond layer (diamond grain growth issues etc...), presence of some small surface defects (deep scratch, inclusions, etc...) etc... These flaws can jeopardize the process behaviour and result in a faulty polishing. So 10 % of process failure will be accepted as resulting from material faults: an overall rate of perfect polishing quality disc is tolerated down to 90% and process will still be considered as successful.

The diamond material is in reality an alloy carbon-cobalt, the cobalt makes the polycrystalline diamond electrically conductive. And this way, it allows Syndite discs to be manufactured by EDM.

However, before polishing, the chemical composition of diamond material is altered on the top surface due to previous machining steps, the surface suffers from a lack of cobalt after leaching of the cobalt atoms during past processes. Polishing allows to recover same chemical composition as bulk material on the top surface by removing a fine layer of material: typically 20  $\mu$ m are removed in average across the all surface of the diamond disc during mechanical polishing. This is sufficient to generate a surface with the original level of cobalt.

If the surface is too depleted in cobalt, the diamond disc cannot be post processed by EDM, a sufficient layer thickness has to be ablated without creating any major depletion of cobalt during the process. So the level of cobalt depletion on the surface of the disc is a primordial quality criteria of success; the percentage of depleted cobalt has to be the minimum possible so the disc: no cobalt depletion on the surface is preferable as achieved by the mechanical process and it must be less than 10% at least. Otherwise a final mechanical polishing will be necessary, no matter how low the surface roughness is, which will compromise the project.

Table 10: Summar	<pre>/ list of the KPI for quality</pre>	assessment
------------------	--	------------

No	КРІ	KPI Values for success	Validation Status
5	Surface Roughness	Expected : Sa < 0.010 $\mu$ m, Sz < 0.12 $\mu$ m $^{(1)}$	
		Validated : Ra < 0.100 $\mu m^{(2)}$	$\bigcirc$
		Non validated : Ra > 0.100 μm	
6	Shape deviation <sup>(3)</sup>	Expected : < +/- 2 μm	۲
		Validated : < +/- 5 μm	9
		Non validated : > +/- 5 $\mu$ m	•
7	Visual Defects <sup>(4)</sup>	Expected : pass rate 100%	
		Validated : pass rate > 90%	<u> </u>
		Non validated : pass rate < 90%	•
8	Cobalt Depletion	Expected : 0 %	9
		Validated : < 10 %	<u> </u>
		Non validated : > 10 %	•
9	Graphitization <sup>(5)</sup>	Expected : No micro-structure modification	•
		Validated : Meet portfolio	<u> </u>
		Non validated : Do not meet portfolio	
10	Colour <sup>(6)</sup>	Expected : L* < 15	9
		Non validated : L* > 15	•

<sup>(1)</sup> Results confirmed to be independent from direction of measurement to ensure true mirror optical finish

<sup>(2)</sup> Under condition of mechanical polishing time as defined in previous paragraph.

<sup>(3)</sup> Only applies if mechanical polishing is required after Laser processing

<sup>(4)</sup> cf document Sn285 - 3 001\_20160107 for the generic criteria utilised by the Element Six Advanced Materials Business Unit for the inspection of PCD material

<sup>(5)</sup> cf Element Six Advanced Materials Business Unit micro-structure portfolio for micro-structure assessments

<sup>(6)</sup> Only for CTB 010 material

Structure of the material has to be identified to control if the material did not undergo any allotropic transformation under ultra-short pulsed laser ablation, as diamond material easily transforms into graphite under thermic exposure. Important transformation of diamond into a graphite structure will heavily modify the properties of the material, the most important one being its hardness. The hardness drops after transformation of diamond into graphite which would cause the material to fail the wearing tests and be non-conform to specifications. So the level of graphitization of the diamond represents a KPI: the micro-structure of the alloy of polycrystalline diamond and cobalt must match the Element Six internal microstructure portfolio, this portfolio holds Element Six microstructure references evaluating the microstructures of the Element Six specific diamond materials.

Colour of the surface must respect a certain specification for marketing reasons, this specification applies only for one Syndite diamond grade: CTB 010. According to the CIE  $L^*a^*b^*$  standards, the lightness  $L^*$  (which represents the darkest black at  $L^* = 0$ , and the brightest white at  $L^* = 100$ ) across the CTB surface must be inferior to 15 uniformly.

#### 4.3 Definition of Characterization and Measurement Methods

A collaboration has been established between Element Six Ltd and the department of Material sciences of the University of Limerick. This collaboration will allow Element Six to use University of Limerick facilities and instruments to run precise characterizations of the samples resulting from the HIPERDIAS project. Measurement methods are detailed for every KPI:

#### **KPI1 – Material Removal Rate**

The material removal rate (KPI1) will be measured with two different methods depending on the samples size to be measured and the accuracy required:

- For 3 mm x3 mm squares :

3D Laser scanning microscopy VKX series from Keyence will be used to measure the depth of 3 mm x 3 mm squares ablated inside the 70 mm diameter discs (Figure 111).



Figure 11: Ablated squares on polycrystalline diamond

- For 70 mm diameter discs :

A Proforma 300i series from MTI Instrument is a device able to measure the thickness of Element Six Syndite disc up to a +/- 0.25  $\mu$ m accuracy across its all surface. The device allows to relocate a disc at its same location to measure thickness variation up to a 0.05  $\mu$ m resolution (figure 12). So Syndite disc thickness will be measured on Proforma device before and after Laser-ablation polishing to calculate thickness variation of ablated material during this process.



# Figure 12: Results from Proforma measurement (thickness variation = 11.22 μm, after 6 hours of mechanical polishing)

#### **KPI5 – Surface Roughness**

As a key parameter of success and critical to measure, surface roughness (KPI5) will be measured at Class 4 Laser's facilities with 3D Laser scanning microscope VKX series from Keyence as well as at the University of Limerick for scientific confirmation purposes where hybrid nanoscope will be used to validate the industrial measurements.

10% of the Syndite disc must be measured in five different locations and various directions (Centre, North, West, South, and East) for final acceptance. Results must be independent from direction of measurement to validate a true mirror optical finish has been achieved.

#### **KPI6 – Shape deviation**

Before mechanical polishing, the dome shape present on the diamond surface of the Syndite disc is controlled by interferometry: the interferometric measurement system TOPOS from the company Lamtech Lasermesstechnik can measure the waviness of the disc with an absolute accuracy up to 0.1  $\mu$ m over the entire measurement area (Figure 133). The measure gives a FLT value representing the maximum height difference of the disc waviness which will have to match the shape deviation requirements (KPI6). Numerous interferometric measures will be taken to ensure shape deviation compliance from the Laser system.

Dissemination Level: Confidential



Figure 13: Flatness measurement of domed surface of pre-polished Syndite disc using interferometric measurement device

#### **KPI7 – Visual Defects**

First the Laser polished Syndite discs must pass engineering qualifications: the engineering qualifications ensure the surface is fully uniform without any visible lay marks. The inspection process is performed by optical microscopy up to x 1,000 amplification.

Then the discs will follow same inspection quality route as the mechanical polished Syndite discs follow in Element Six production line (Figure 14: Production line of quality inspection of polished Syndite disc): any presence of visual defects will be observed on an x20 optical microscope as standard. Results from the x20 optical microscopy observation will provide the pass rate (KPI7).



Figure 14: Production line of quality inspection of polished Syndite disc

However the scientific study will evaluate the surface topography evolution through variation of all these Laser parameters and rastering modes using conventional microscopy, scanning electron microscopy and atomic force microscopy.

#### KPI8 and KPI9 – Cobalt Depletion and Graphitization

Chemical composition of the diamond surface is different from chemical composition of bulk diamond material. Material must be ablated until surface recovers same composition as bulk material. Ultra-short Laser pulsed ablation might also affect chemical composition of the diamond: chemical compounds can differently react to the heat induced by ultra-short pulses: the chemical compounds can be vaporized at different rates, their ratio on the material surface then varies; the structure of the chemical compounds can also be transformed under the heat accumulation into the material during the ablation process.

Then following measurement methods will be applied:

#### Grant Agreement No: 687880

Dissemination Level: Confidential

- EDS will measure any variations of percentage of chemical compounds (KPI8) and X ray diffraction will detect any changes of carbon structure (KPI9) to control if the ablation process does or does not conserve the chemical composition and structure of the ablated surface.
- Assuming the ablation process keeps chemical composition and structure intact: How much material has to be removed to recover a homogenous material?
- Successive EDS measurements of ablated samples with an increasing depth will allow to determine the minimal ablation depth
- Assuming the ablation process does not keep chemical composition and structure intact: what is the conclusion?
- Depending on the percentage of cobalt depletion and graphitisation (KPI7 and KPI8), the conclusion of these measurements will be that a final mechanical polishing is necessary after the ablation process

The use of a Laser system producing an average power of 1000W can produce thermal subsurface damage during ablation of such a thermal conductive material as diamond even though pulses are in the order of subpicoseconds. The heat dissipated into the material can create allotropic transformation of the carbon constituting the diamond: different carbon structures like graphite can grow.

Subsurface analyses will be conducted to study the heat dissipation into the material and the reaction of the diamond material to it: transmission electronic microscopy analyses will be carried out to detect percentage and depth into the material of other diamond structures (KPI9).

Grant Agreement No: 687880

Table 11: Summary list of measurement methods per KP	Table 11	: Summary	list of	measurement	methods	per	KPI
--	----------	-----------	---------	-------------	---------	-----	-----

No	КРІ	Measurement Method	Location of Measurement
1	Material Removal Rate	3D Laser scanning microscopy	Class 4 Laser AG
		Proforma 300i series	Element Six
5	Surface Roughness	3D Laser scanning microscopy	Class 4 Laser AG
	(Topograpny)	Hybrid nasoscope	Element Six*
6	Flatness	TOPOS interferometric measuring system	Element Six
7	Visual Defects	Optical microscope (up to x1,000 mag)	Class 4 Laser AG
	(Topography)	3D Laser scanning microscopy	Class 4 Laser AG
		Scanning electron microscopy	Element Six*
		Atomic force microscopy	Element Six*
8	Cobalt Depletion	Electron diffusion spectroscopy	Element Six*
	(Chemical Composition)	Transmission electron microscopy	Element Six*
9	Graphitization	X ray diffraction	Element Six*
	(Micro-structure)	Transmission electron microscopy	Element Six*
10	Colour	luminance meter	Element Six

\*in collaboration with the University of Limerick

## 5. Summary

Within this deliverable, the Key Performance Indicators (KPI's) for the three applications to be pursued by the end-users Bosch (3D silicon processing), Class 4 Lasers (fine cutting) and Element Six (diamond polishing) have been defined. Even though these applications are very different in nature, some of their KPI's are qualitatively comparable to each other:

- Productivity KPI's such as material removal rate or cutting speed
- Quality KPI's such as surface roughness, surface defects, and shape deviation
- Precision KPI's such as general tolerances and edge (side) steepness
- Cost KPI's such as the specific process cost
- Necessity of post processing.

For these KPI's, similar evaluation techniques can be applied (such as 3D scanning microscopy for surface roughness and tolerances) and the results can be compared across all applications. On the contrary, some of the KPI's are very specific to the application they track such as the graphitization of diamond surfaces for which according evaluation techniques have to be applied (such as X-ray diffraction analyses).

Furthermore, the definition of KPI's depends on the fundamental kind of process: In the case of finecutting and diamond polishing, established processes are already available to serve as benchmarking processes. The purpose of the KPI's mostly consists in deciding whether or not a laser process would beat the current process performance by a sufficient margin to justify a replacement. In the case of 3D silicon processing, high-throughput processes are not yet available at all. In this case the KPI's represent a general feasibility analysis to determine whether or not a process can be established that allows mass-manufacturing of new innovative products.