## THESIS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY

# **On Polishability of Tool Steels**

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#### **On Polishability of Tool Steels**

SABINA REBEGGIANI Göteborg 2013 ISBN 978-91-7385-828-1

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Doktorsavhandlingar vid Chalmers tekniska högskola Ny serie Nr 3509 ISSN 0346-718X

#### Published and distributed by

Chalmers University of Technology Department of Materials and Manufacturing Technology SE - 412 96 Göteborg Telephone: +46 31 772 1000 URL: www.chalmers.se

#### Printed in Sweden by

Chalmers Reproservice Göteborg, Sweden 2013 On Polishability of Tool Steels SABINA REBEGGIANI Department of Materials and Manufacturing Technology Chalmers University of Technology

## ABSTRACT

One of the world's fastest growing industries is the plastic industry. Today's ever increasing demands of high quality products, shorter lead times and reduced costs push development and research forwards. Moulds for plastic injection moulding need to have a functional surface to meet demands on demoulding and wear properties, but also to produce the required final surface quality, which for 'standard mould qualities' of high gloss applications means nearly defect free, shiny and smooth mould surfaces with roughness levels in the nm-range.

The aim of this thesis was to develop a metrology framework to quantitatively characterise these mould surfaces in order to gain better understanding of which defect structures are critical at injection moulding, and how these are correlated to material properties and operating conditions in surface preparation of tool steels. In practice this means to capture surface features of some few nm in height/depth up to some hundreds of microns in lateral dimension within insert areas of cm<sup>2</sup> and larger. Experiments combining polishers' experience with steel producers' as well as non-contact areal texture examinations of surface topography were performed to overcome and link practical skills to academic ones.

Based on areal surface metrology, defect classification and image analysis based surface characterisation, an evaluation procedure for polished tool steel surfaces was developed, initially tested and verified. The suggested method involves descriptions of relevant defect structures and acceptance levels for high gloss polished tool steels in the form of numerical parameter values based on interferometric measurements. It was also concluded that the cleanness of the steels was less important as long as it was kept within reasonable levels; the surface preparation strategy is a major factor influencing the mould surface quality, e.g. it was found that a 'several-step-strategy' was favourable to avoid defect structures; not all 'mirror-like' mould surfaces had desirable topographies for injection moulding, therefore a well-defined mould surface assessment with numerical values describing mould surface quality is necessary to secure effective mould surfaces.

#### **Keywords:**

Tool steels, polishability, surface characterisation, surface texture parameters, high gloss polishing, injection moulding

# ACKNOWLEDGEMENTS

I would like to express my thanks to all who helped and supported me during my doctoral studies and made this work realisable, especially;

- My academic supervisor Prof. BG Rosén, Halmstad University, for his supervision, guidance and never failing encouragement.
- My industrial supervisor Alf Sandberg, Uddeholms AB, for his invaluable support and belief in the project, and who persuaded me to join the project by insisting on a visit to Uddeholms AB in Hagfors.
- Prof. Lars Eriksson, Jönköping University, for giving me the designer's point of view.
- The members in the project team; Jens Grønbæk from Strecon A/S, Ralf Kiefer from Kiefer GmbH, Erik Madsen from Grundfos A/S, Palle Ranløv from Uddeholms A/S, Stefan Rosén from Toponova AB and Georg Zwick from Böhler-Uddeholm Deutschland GmbH.
- All staff at Uddeholms AB, Fraunhofer IPT, Huddersfield University and Aalen University with whom I have had inspiring discussions.
- Prof. Tom Thomas for his kind help and good advice.
- DigitalSurf for providing me the software MountainsMap Premium.
- Kiefer GmbH, Grundfos A/S, Strecon A/S, Svensk Industrigravyr AB, Primateria AB, Polérteknik ApS, ZygoLot and Bayer MaterialScience for taking part in the experimental work.
- Uddeholms AB, the Swedish Foundation for Strategic Research ProViking Research School, the Knowledge Foundation – CAPE Industrial Research School and the European Commission Seventh Framework – the poliMATIC project for their financial support.
- My colleagues in the Functional Surfaces Research Group at Halmstad University for providing a creative and pleasant atmosphere.
- My beloved family and dear husband Peter for their patience and unfailing support and my wonderful little daughter who ensures that I do not work (or sleep) too much.

I also want to thank the following copyright holders for permission to use their material;

- Elsevier Ltd for figure 2 and 7.
- Uddeholms AB for their pamphlet in Appendix I.

Sabina Rebeggiani Halmstad, March 2013

# LIST OF APPENDED PAPERS

# Tool steel polishing and topography characterisation S. Rebeggiani, A. Sandberg, B.-G. Rosén In: Proceedings of the Swedish Production Symposium 2007, Göteborg, Sweden (2007) *Contribution:* Initiated the study, took part of surface investigation, performed surface measurements, analysed surface data, wrote and presented the paper.

#### II. Surface characterization of high gloss polished tool steels

S. Rebeggiani, B.-G. Rosén, A. Sandberg

In: Proceedings of the 8<sup>th</sup> International Tooling Conference, Aachen, Germany (2009)

*Contribution:* Took part in the planning of the experiments, performed surface measurements, analysed and evaluated the experiment, wrote and presented the paper.

# III. A quantitative method to estimate high gloss polished tool steel surfacesS. Rebeggiani, B.-G. Rosén, A. Sandberg

In: Proceedings of the 13th International Conference on Metrology and Properties of Engineering Surfaces, London, UK (2011); and J. Phys.: Conf. Ser. 311 (2011), pp. 012004

*Contribution:* Initiated the study, selected and measured the samples, analysed surface data, suggested procedures for surface analysis, wrote and presented the paper.

IV. Factors influencing the surface quality of polished tool steels

S. Rebeggiani, B.-G. Rosén

Submitted to the Institute of Physics Journal: Surf. Topog.: Met. Props. (2013) *Contribution:* Took part in the planning of the experiments and the surface investigations, performed surface measurements, analysed surface data, and wrote the paper with advice from colleagues/supervisor regarding the structure of the article.

# V. Quantitative evaluation of the surface finish of high gloss polished tool steels

S. Rebeggiani, B.-G. Rosén

Submitted to: The 14th International Conference on Metrology and Properties of Engineering Surfaces, Taipei, Taiwan (2013)

*Contribution:* Took part in the planning of the experiments and the surface investigations, selected and measured the samples, analysed surface data, suggested procedures for surface analysis, and wrote the paper with advice from colleagues/supervisor regarding the structure of the article.

# **ADDITIONAL PUBLICATIONS**

- VI. *Measuring strategies for smooth tool steel surfaces* J. Berglund, P. Jonsson, S. Rebeggiani, B.-G. Rosén
   In: Proceedings of the XII. International Colloquium on Surfaces, Chemnitz, Germany (2008)
- VII. *Evaluation of a robot assisted polishing equipment*S. Rebeggiani, B.-G. Rosén
  In: Proceedings of the Swedish Production Symposium 2008, Stockholm, Sweden (2008)
- VIII. Towards robust polishing strategies for moulds and dies
  F. Klocke, B.-G. Rosén, B. Behrens S. Rebeggiani, R. Zunke
  In: Proceedings of the Swedish Production Symposium 2009, Göteborg, Sweden (2009)
- IX. *High gloss polishing of tool steels step by step*S. Rebeggiani, A. Sandberg, B.-G. Rosén
  In: Proceedings of the Swedish Production Symposium 2011, Lund, Sweden (2011)
- X. A step-by-step analysis of manual polishing sequences
   S. Rebeggiani, B.-G. Rosén
   In: Proceedings of the 9th International Tooling Conference, Leoben, Austria (2012)

# Polishability of tool steels – Characterisation of high gloss polished tool steels

S. Rebeggiani

Licentiate thesis, Chalmers University of Technology, Göteborg, Sweden (2009)

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# **1** Introduction

#### **1.1 Background**

One of the world's fastest growing industries is the plastic industry. Fast and automated production lines produce more or less complex plastic components in large volumes at low labour and material costs [1]. For injection moulding, individually produced moulds are needed – one for each component (unless multi-cavity moulds are used). Today's ever increasing demands for high quality products, shorter lead times and reduced costs push development and research forwards. As well as basic functions like shaping and cooling of the polymer melt, the mould needs to have a functional surface, e.g. to meet demands on demoulding and wear properties, functions that shall be combined with required final surface quality, which for 'standard mould qualities' of high gloss applications means nearly defect free, shiny and smooth mould surfaces with roughness levels in the nm-range.

The final finishing operations of the moulds are conventionally performed by manual polishers, who carefully polish the inserts into mirror-like appearances. The work is time-consuming and cumbersome, and wears hard on the polishers. Yet surface polishing is a relatively unexplored area; most knowledge is accumulated by individual polishers with long experience in the field, and literature covering the polishing process/mechanisms is rare. Since it has also become harder and harder to recruit new skilled polishers, a shift from manual to robot assisted polishing is to be expected. Automated systems should, among other things, lead to improved working environment for polishers, reduced production time, and increased reproducibility of mould surface quality due to less dependence on individual polishers.

But to manufacture a mould suitable for high gloss applications does not only depend on the surface preparation itself, the basis for how easy/good a tool steel is to polish and what surface quality can be expected, i.e. the tool steel polishability, is formed already during the production of the steel. Carbides, non-metallic inclusions, segregations...they all affect the material properties and it is of vital importance to understand their overall impact in order to handle the material selection to facilitate the manufacturing of moulds.

Another link to take into account in the process chain of plastic components (illustrated in figure 1) is the mould-users who, with their knowledge about polymers' different behaviours during processing, can affect the replicability from mould to plastic surface by adapting process parameters individually.



Figure 1: Involved parties in the process chain of plastic components.

In the end, it is the user who decides if the product properties meet desires and expectations or not, no matter the product specification. A way to stitch together all steps in the process chain and facilitate the communication between involved parties is to agree on a common standard to test and characterise the polishability of tool steels, and to quantitatively describe surface qualities and relevant defect structures.

#### **1.2** Aim of the thesis

The aim of the thesis was to develop a metrology framework to quantitatively characterise high gloss polished tool steels, and thereby to better understand;

- which defect structures are critical at injection moulding;
- how these defect structures are linked to material properties (mainly the content of non-metallic inclusions);
- how these defect structures are correlated to the surface preparation.

#### **1.3 Research approach**

This work is built up around the idea of the surface control loop (see figure 2) where the surface, rather than the product, is in focus. The control loop was introduced to emphasise the importance to link function, manufacturing and characterisation in order to understand and produce well functioning surfaces [2]. *Function* is suppose to answer what a surface shall do, e.g. to convey the right impression of a product [3], to work well in tribological contacts [4] or to facilitate bone growth [5]. *Manufacturing* is the knowledge of the process to achieve that surface, and *characterisation* how to describe and control desired surface function.



*Figure 2: The control loop illustrating the relationship between the three facets manufacturing, characterisation and function, from [2].* 

Figure 3 shows how the surface control loop has been connected to the process chain of plastic components (described in figure 1) in order to link all involved parties to the different facets presented in figure 2 and clearly show their interdependence.

Three nested loops are needed, interconnected in the function facets, to fully understand how to achieve a satisfactory result, i.e. a highly glossy surface with no or few surface defects. The three loops, which all consist of one manufacturing-, one function- and one characterisation facet each, are:

The Component loop (denoted *comp* for component) – the outermost loop considering the plastic components, i.e. the final parts. The plastic surface is strongly linked to the mould surface quality, and it is of great importance to know the expected surface finish of final products to be able to manufacture a suitable mould surface within the right quality level.

The Steel loop (denoted *steel*) – the innermost loop considering the tool steel, i.e. the material used for the manufacture of injection moulds. Process route, alloying elements and heat treatments all affect the polishability, which is only one of various steel properties needed for moulds.

The Mould loop (denoted *mould*) – the middle loop considering the transformation from bulk steel to a mould, which includes design, manufacturing, preparation, and assessment of the mould.



*Figure 3: The surface control loop adapted to the process chain for plastic components; FUNC – function, MANU – manufacturing, CHAR – characterisation.* 

All three loops have been addressed in the thesis, but a clear focus has been on the middle loop, the Mould loop, where the surface preparation – mainly manual polishing – and resulting surface quality has been studied. The other two loops are needed to be able to relate mould surface quality to final plastic components as well as to better understand why some steel grades are better suited for high gloss polishing than others – is it due to material or polishing aspects?

#### **1.4 Delimitations**

The steel grade selection was made by the steel producer to accurately represent different kinds of microstructures, but since moulds are impractical to handle due to their high weights, the majority of the tested materials in this thesis have been in the format of small, plane test samples.

All samples have been machine, manually or robot polished with the intention to achieve a mirror-like appearance, i.e. to required surface quality for moulds used for injection moulding of glossy plastic components. Only polishing techniques where fine abrasives mechanically modify the workpiece surface have been considered in this work. All other surface preparation techniques and stages to produce a mould, e.g. mould design and manufacturing of cooling channels, will be excluded or only briefly discussed.

Visual estimations of the surface quality of included samples were performed by experienced polishers. The results served as references and formed the basis for the defect classification and presented surface quality levels. Descriptions of surface defects which occurred due to worn or wrongly designed moulds or improper machine settings, and how to solve the consequent problems are described in e.g. [1,6] and are not included in this thesis. Also shape/form considerations are outside the scope of this work.

#### **1.5 Outline of the thesis**

The first part of the thesis, i.e. chapters 1 to 4, will introduce the reader to the surface control loop and how it is applied to the process chain of plastic components. Three loops have been linked to gain a better understanding of how the surface quality of final plastic components is related to injection moulding parameters as well as the mould surface quality, which in turn e.g. is related to its chemical composition and surface preparation.

The second part, chapters 5 and 6, discusses the results from appended papers put into the context of the surface control loop in accordance to figure 3.

*Chapter 1* introduces the reader to the industrial background of this thesis, including the aims, delimitations and research approach. The latter explains the surface control loop as a base for studies of tool steel polishability, and how it relates to the process chain of plastic components.

*Chapter 2* considers injection moulded plastic components. Mould surface quality is one factor affecting final products, and since the surface preparation of moulds is costly, it is desirable to manufacture moulds with the right quality level.

*Chapter 3* considers the steel, e.g. purity levels, and how such properties influence the mould quality.

*Chapter 4* considers the mould surface; the main parts are the preparation of mould surfaces focusing on abrasive polishing, and the characterisation of the same including both measurement techniques and how to extract relevant information from measurement data.

*Chapter 5* includes experimental data, results and discussion divided to connect to the three loops described in figure 3. Thus chapter 5.1 connects to the Component

loop, chapter 5.2 connects to the Steel loop, and chapter 5.3 connects to the Mould loop subdivided into surface preparation of moulds and resulting surface quality. *Chapter 6* summarises main conclusions, and presents suggestions for future activities within the field.

# 2 The surface control loop – the component

The outermost loop considers the production of injection moulded plastic components, see the green marked parts in figure 4. Main focus has been on the appearance of the plastic components and how it is related to the mould surface quality.



*Figure 4: The outermost loop considering the plastic component, including production, control, and function of the surface of the components.* 

#### **2.1** FUNC<sub>comp</sub> – function of component surfaces

In this context, the desired surface function is a high glossy appearance without disturbing defects. As a surface description in a specification, a 'shiny quality' is rather vague and numerical parameters, others than roughness parameters, are seldom used. But, a plastic surface can also include other types of functions than appearance, e.g. high reflection if used for head lights on cars, a certain texture to hide finger prints, or a suitable surface structure to be lacquered. No matter the function, measurable target values of final surface qualities, including acceptable defect levels, are preferable in order to obtain objective quality controls.

#### 2.2 MANU<sub>comp</sub> – injection moulding of components

Knowledge about the injection moulding technique in combination with thermal and rheological properties of the polymer is essential to control moulding parameters in order to satisfy requirements of e.g. reproducible dimensional accuracy and surface quality of produced components [7]. In fact more than 100 parameters, direct or indirect, influence the injection moulding process, thus a more practical approach to understand the relationship between these parameters and how they influence cost and quality aspects is to be preferred [8]. Four basic categories, all dependent on each other, cover all parameters; 1) temperature, considered to be the most important one, followed by 2) pressure, 3) time, and 4) distance. Surface defects occurred due to worn or wrongly designed moulds or improper machine settings, and how to solve consequent problems is described in e.g. [1,6]. However, defects transferred from the mould surface are just briefly discussed in the same.

#### 2.3 CHAR<sub>comp</sub> – characterisation of component surfaces

Since the final goal is to achieve high quality surfaces of the plastic components, it is of great importance to measure and analyse those surfaces to be able to distinguish between defects caused by moulding conditions, i.e. process parameters as well as polymer properties which are up to the mould-user to regulate, and defect structures coming with the mould surface. The latter are supposed to be avoided mainly by sufficient surface preparation in combination with properly chosen tool steels.

Similar measurement techniques to those used for included steel samples have been used to study the plastic components in this work. However, functional tests connected to specific applications are more common in industry, e.g. in-house measurements of the reflection capacity of produced headlights. Others use reference specimens for visual colour and gloss inspections, performed by trained personnel, to find the right appearance on components, which is of great importance for all high-quality products [9,10].

There are also standards for gloss, transmission and colour investigations. E.g. the DIN 67530, defining measurement and assessment of gloss for plane surfaces of paint coatings and plastics [11].

# **3** The surface control loop – the steel

The innermost loop considers the raw material, i.e. the steel, see the orange marked parts in figure 5. Choice of process route, alloying elements, and heat treatments all affect final tool steel properties. This work has focused on the polishability, i.e. how easy it is to reach a certain surface quality.



Figure 5: *The innermost loop considering the steel, including production, control, and function of the same.* 

#### **3.1** FUNC<sub>steel</sub> – function of steels

Tool steels must fulfil various functions, both to facilitate manufacturing (e.g. machinability) and process demands (e.g. wear resistance). To satisfy all desired tool steel properties concurrently is a difficult task; the list of essential characteristics is long and most often the requirements are incompatible – e.g. higher sulphur content will improve machinability but decrease polishability [12]. An overall material screen to exclude steels not suited for actual application, as well as taking price issues into account, is needed to properly choose the right tool steel, which serves as the base for mould-makers, polishers and the mould-users. Below, the major tool steel properties are listed [1,13-15]:

- Compressive strength to withstand the high clamping forces during moulding
- Thermal conductivity to control the cooling speed in the moulds
- Toughness to resist cracking and fractures
- Wear resistance to withstand surface damage over time
- *Corrosion resistance* to resist chemical reactions in cooling channels and withstand corrosive polymers
- Machinability to reduce cost and time at mould manufacturing
- *Heat-treating dimensional stability* to reduce shape/dimensional corrections after heat treatment
- Polishability to facilitate final surface preparation

## **3.2 MANU**<sub>steel</sub> – manufacturing of steels

The key factors contributing to high-quality tool steel production are [1,14,16]:

- *Process route* different techniques result in various degrees of non-metallic inclusion and segregation contents, and type of microstructure.
  - IC (conventional ingot cast) materials are steels with varying amounts of inclusions that are globular and/or elongated in the hot working direction, and various degrees of segregation patterns.
  - **ESR** (electro slag remelted) materials are in general rather pure steel grades with uniform microstructures, normally including small oxides.
  - VAR (vacuum arc remelted) materials are steels with well controlled microstructures with low contents of segregations and small and evenly distributed inclusions.
  - **PM** (powder metallurgy) materials are clean steels with fine and homogeneous microstructures, and small evenly distributed carbides.
- *Heat treatment* a way to tune mechanical properties of steel grades, e.g. to increase or decrease the hardness, the toughness or the strength.
- Composition alloying elements are added in various amounts to optimise certain material properties. Roberts et al. [14] point out that 'tool steels are ... complex microstructural systems, where every microstructural component of the system is affected by alloying and processing, and eventually plays a role in the performance of a steel under specific service conditions'. Table 1 lists common alloying elements and how they affect tool steel properties.

Alloying ele	ment	Effects on steel				
Aluminium	Al	Increase the possibilities to control the grain growth				
Carbon	С	Increase the hardness, wear resistance and tensile strength (ductility and weldability decrease with increased C)				
Chromium	Cr	Adds wear resistance, corrosion and oxidation resistance; carbide- former, increase hardenability, improve high-temperature strength				
Manganese	Mn	Adds strength and hardenability; eliminate high temperature brittleness (ductility and weldability decrease with increased Mn)				
Molybdenu m	Мо	Adds heat resistance and hardenability, carbide-former				
Nickel	Ni	Adds toughness and to some extent wear resistance, increase the hardenability and impact strength				
Nitrogen	Ν	Increase strength and corrosion resistance; nitride-former				
Silicon	Si	Adds strength and increase the hardness				
Sulphur	S	Increase machinability (ductility, notch impact and weldability decrease with increased S)				
Vanadium	V	Increase yield strength and tensile strength; refine grain structure, carbide-former (hardest of all carbides)				

*Table 1: List of common alloying elements and their effect on tool steel properties* [14,17-18].

Understanding of how these factors affect final surface qualities is of great importance to be able to tailor-make tool steels with required material properties and simultaneously facilitate mirror-like mould surfaces, i.e. the steel itself is one, but complex, factor to take into account to avoid material defects.

#### **3.3** CHAR<sub>steel</sub> – characterisation of steels

This facet considers both quality control leading to rejection of unacceptable steels, e.g. those with too high slag contents, and material development. Quality controls commonly involve ultrasonic tests of ingots/steel bars to detect larger defect structures and slag estimations and inspections of microstructures of test samples preferably taken from the centre parts of the heat treated steel bars where the slag content is expected to be the highest. Material development involves more detailed analysis, e.g. chemical analysis and measurements by scanning electron microscopes combined with energy-dispersive x-ray spectrometers enabling elemental analysis.

There are several standards to determine the cleanness of tool steels, e.g. ASTM E45-11a including methods to categorise inclusions by size, shape, concentration, and distribution [19], ASTM E2142 - 08 which, based on the former, sort inclusions into chemical classes based on automatic x-ray analysis [20], SS 111116 dividing inclusions into four classes depending on their length, width and quantity [21], and DIN EN 10247 based on an automated detection where size and shape of the inclusions are strictly mathematically defined [22]. DIN EN 10247 is expected to be more objective; inclusion classifications tend to vary slightly from person to person, and from day to day which leads to less reliable results [23].

# 4 The surface control loop – the mould

The middle loop considers the mould, i.e. the transformation from steel into a mould, see the yellow marked parts in figure 6. Main focus has been on the characterisation part, i.e. how to estimate, measure and numerically describe the surface quality of polished tool steels.





## 4.1 FUNC<sub>mould</sub> – function of mould surfaces

In this work the surface function is mainly discussed in terms of the appearance and surface quality of the mould, i.e. it attempts to answer questions like:

- what surface quality is acceptable for production of shiny plastic components?
- how can a polisher's qualitative estimations be described quantitatively, i.e. with measurable target values?
- how can such qualitative descriptions be presented in a common vocabulary in order to define different defect structures?

However, mould surfaces are mixtures of several essential functions, e.g. they need to have low adhesion to facilitate demoulding and to withstand corrosion and abrasive particles. In other words 'they are expected to provide reliable and fully repeatable function in spite of being under extreme loads during the moulding process, and long service life...', quoted from [1].

#### 4.2 MANU<sub>mould</sub> – preparation of mould surfaces

A 'successful mould making' is based on five essential factors [14]:

- high-quality steel,
- good mechanical design, e.g. avoidance of sharp corners,
- proper selection and application of the steel,
- correct heat treatments,

• proper finishing by grinding and electric discharge machining (EDM).

'Proper finishing' should be split into three parts – machining to reach basic the shape, pre-preparation of the surface and final surface preparation.

Pre-preparation of moulds, often synonymous with grinding, is the basis for a good final surface quality. Rougher surface features like deeper scratches and white layers, residues from EDM, need to be eliminated, and final mould dimensions must be secured. Also stress-relief treatments could be needed to reduce residual stresses introduced by grinding and/or EDM [14]. Thereafter the final surface preparation can begin, which for moulds for injection moulding of shiny plastic components today almost always means manual polishing due to complex mould geometries restricting the use of automatic polishing machines.

#### 4.2.1 Final surface preparation

Polishing is a widely used concept and is, in general terms, a process with the objective to produce an even surface with high gloss or light reflection for decorative or technical purposes by modifying the surface condition rather than the shape [24-25]. High gloss polishing of moulds commonly means abrasive machining, i.e. lapping and/or polishing. Experienced polishers prepare moulds by the use of a combination of rotary and reciprocating high speed hand-held tools, abrasive papers, polishing stones and self-made carriers in various materials in combination with diamond paste/suspensions in order to achieve the required mirror-like appearance. On-going developments on robot assisted polishing equipments, e.g. [26-28], are expected to further improve the reproducibility of mould surface qualities since automated techniques are less dependent on individual polishers, reduce production time and costs, and improve the working environment for polishers.

However, there is an abundance of other technologies to choose between, e.g.;

- Diamond turning often used for high-quality aspheric optical components [29];
- *CMP* (*Chemical-Mechanical Polishing*) commonly used in the semiconductor industry due to its ability to generate exceptionally flat and nearly damage free surface finish [30];
- *Laser polishing* which is a fast technique that, unfortunately, leaves a wavy structure in the surface [31-32];
- *EDM (Electro Discharge Machining)* hardly reaching mirror-like surfaces [33-34].

The theories behind these techniques differ due to their nature and need to be discussed separately. Therefore, this text only considers polishing as a process where fine abrasives mechanically modify the workpiece surface in purpose to achieve a mirror-like appearance.

Diverse hypothesis have been presented through the years, e.g. in Micrographia, written in the 1700th century, where studies of a razor edge in an old microscope

led to the following conclusion 'polish ... must consist of little hard rough particles, and each of them must cut its way, and consequently leave some kind of gutter or furrows behind it' [35], by Beilby stating that 'polish is the result of surface flow' i.e. a thin film is formed by mobile, solid molecules, originating from the tool or polishing agent, behaving as a liquid which fills in grooves and pits [36], and by Samuels who proved that the polishing process was a micro-cutting process and that polished metal surfaces were plastically deformed [37].

A complete model explaining the fundamentals of polishing does not exist; material removal behaviour is still poorly understood and predictive process models still wait to be developed [38]. Today, polishing is commonly treated as a wear process, which in general terms is described as the combination of abrasion, adhesion, surface fatigue and tribochemical mechanisms. Abrasion, assumed to be the most important one, is divided into four parts – micro-ploughing and -cutting caused by embedded abrasives, and micro-fatigue and -cracking caused by freely rolling grains [39-40]. But which factors/parameters affect what type/s of abrasion? How? And, how to include them in a model?

The attack angle, defined as 'the angle between the working face of a particle which emerges at a surface and the plane of the specimen', see figure 7, is important to include in theoretical models of abrasive processes [41]. To be able to describe the ratio of micro-cutting to micro-ploughing, the  $f_{ab}$  value was introduced [39,42], illustrated in figure 7. Only a fraction of the worn surface will be torn away by micro-cutting, the rest will be pushed to the groove sides by micro-ploughing due to plastic deformation. Further, material removal can be caused by micro-fatigue, i.e. low cycle fatigue, when material is pushed back and forth over the surface as a consequence of several discrete abrasions, which allows both surface topography and material properties to differ from initial conditions, pointed out by e.g. [43-44].



Figure 7: Left; a sketch from [41] showing the attack angle  $\alpha$ . Right; cross section of a groove showing Gahr's definition of the  $f_{ab}$ -value, from [39]. A value equal to one indicates pure cutting, a value equal to zero pure ploughing.

Results based on scratch tests to determine the  $f_{ab}$ -value for hardened and unhardened (hardness levels below 1300 MPa) steels showed that the transition

from micro-cutting to micro-ploughing was between  $60^{\circ}$  and  $30^{\circ}$  in case of the hardened steels, and around  $60^{\circ}$  in case of the unhardened steels, which is the assumed range of effective attack angles for most diamond abrasives [45]. Further, it was stated that it is 'not the hardness of the material, but the deformation capacity of the workpiece material which determines the level of resistance to material removal' [45], which in practice means that the material removal is higher for harder materials than for softer ones if the energy input, e.g. applied pressure, is low. Similar results were presented in e.g. [46] where the  $f_{ab}$  value was found to be dependent on material properties as well as operating conditions, and in [47] where in situ experiments showed that the cutting region increased with increased workpiece hardness.

Others have focused on abrasive properties; e.g. [48] who found that angular shaped abrasives gave rise to higher wear rates and more sharp and narrow grooves than rounded abrasives, and concluded that not only the abrasive hardness and shape, but also the abrasive rolling behaviour, i.e. to what degree sharp corners are in contact with the workpiece, affected the wear rate and so final surface conditions. [49] showed that high loads and low abrasive concentrations facilitated a grooving behaviour since most abrasives were embedded in the carrier, while low loads and high abrasive concentrations lead to a rolling behaviour due to free abrasives.

To summarise, specific models describing the mechanisms behind high gloss polishing do not exist, even though there are 'nearly 200 'wear equations' involving an enormous spectrum of material properties and operating conditions' trying to predict tribosystems – all failures, according to [50-51]. The reason might be the amount of interacting parameters, which are summarised and discussed with a more practical view of the polishing (and lapping) process in [38]. The process is described as a four component system – the workpiece, the lap (carrier), the granule (abrasive), and the carrier fluid (lubricant). This means that the polisher needs to find the best suited combinations for the actual workpiece material, for each step of surface preparation, in a jungle of parameters/factors;

- *abrasives* (type of material/chemical composition, grain size, size distribution, shape, concentration, in a fluid/paste);
- *lubricant* (coolant, to prevent grain agglomeration);
- *carriers* (type of material, hardness, geometry, structure/porosity/texture to e.g. enhance fluid/abrasive transport);
- *process parameters* such as contact forces, time, environment (temperature, humidity, cleanness), tool direction (linear/circular, frequency, rpm), tool holder, technique (e.g. manual and/or hand-held devices) etc.

#### 4.3 CHAR<sub>mould</sub> – characterisation of mould surfaces

There is a broad range of measurement equipment and analysis techniques available to quantify engineered surface topographies - for industry as well as for research – the choice of which should be determined by the output of interest. Mechanical profilers for quality controls in production lines are most often enough for continuous go/no go inspections, whereas more accurate instruments are needed for failure analyses. The performance of different measurement equipment can be compared using an amplitude-wavelength space graph [52], which gives more detailed information of the effective working range of measurement devices.

Surface inspections of moulds consider shape/form, waviness, roughness and local defect structures. In practice this means that surface features of some few nm (in height/depth) up to some hundreds of microns (in lateral dimension) should be detected within insert areas of cm<sup>2</sup> and larger. Experienced polishers have trained their ability to assess surface qualities through the years and identify barely visible surface features very quickly even on larger moulds.

Standards to classify typical surface imperfections with general characteristics and parameters are e.g. ISO 8785 defining terms and parameters for universal use [53], ISO 10110-7 specifying acceptance levels for optical elements and systems [54], and ANSI/OEOSC OP1.002, the American counterpart, including illustrations and methods for inspection of optical components [55]. An established 'defect standard' that classifies, describes and quantifies unwanted surface features for moulds could not be found by the author.

#### 4.3.1 Instrumentation

More advanced surface (and sub-surface) measurement devices have existed for commercial use since the beginning of 1990, but is still sparsely utilized in the field. Table 2 summaries a selection of commercial techniques, commented in the view of being used for high gloss polished tool steels. In common for all techniques, irrespective of if it is based on a tactile or an optical probe, is the small measurement area which is << th the mould area, i.e. it is of great importance to know why, what and where to measure in order to get representative surface values. Scatterometers are included as these techniques provide relatively large measurement areas and are insensitive to vibrations which means they are well suited for in-line applications.

Instrumentation		Resolution [m]	Advantages/Drawbacks	Comments
Line- profiling methods	Contact instrument: stylus	xy: 10 <sup>-6</sup> -10 <sup>-4</sup> z: 10 <sup>-9</sup>	Insensitive to tilted samples, time-consuming areal measuring, risk of surface damage, fragile stylus/pick- up	Commonly used in industry as 2D profiler
	Atomic force microscopy (AFM)	xy: 10 <sup>-10</sup> z: 10 <sup>-12</sup>	High resolution, time- consuming, noise sensitive, fragile stylus/pick-up, limited surface height	Small measurement area
Areal topography measuring methods	Non-contact instrument: phase shifting interferometric microscopy	xy: 10 <sup>-6</sup> z: 10 <sup>-10</sup>	Short measurement time, relatively small measurement areas; sensitive to vibrations	Limited workpiece dimension
	Non-contact instrument: focus variation	xy: 10 <sup>-6</sup> z: 10 <sup>-7</sup>	Large depth of focus, problem with artefacts	Known as confocal microscopy
	Scanning electron microscopy (SEM)	xy: 10 <sup>-9</sup> z: 10 <sup>-9</sup>	Ability to image undercuts, large depth of focus, no height information, work in vacuum, limited workpiece dimension	Stereoscopic imaging can provide areal topography imaging; EDS provides localised chemical information
Area- integrating methods	Scatterometer: Total integrated scattering (TIS)	xy: 10 <sup>-6</sup> z: 10 <sup>-10</sup>	No size limit on samples, short measurement time, comparably large measurement area, rms<< illuminating wavelength	Roughness values need to be correlated to profile methods, large measurement area

Table 2: Summary of a selection of instrumentation for surface measurements;
listed resolutions should be interpreted as general guidelines rather than fixed
values true for all instruments within respective category. The information is
collected from [56-60].

#### Mechanical stylus (profiler)

Profilers are doubtless the most common measurement devices for surface roughness estimations, probably due to their simplicity to handle. Basically, vertical displacements of a stylus attached on a cantilever traversing the surface are amplified and presented as a profile [57]. Such technique facilitates surface measurements inside holes and of any sized workpiece provided there is space for the cantilever. Handheld units generate profile measurements, including roughness parameters, in a few minutes providing a 'receipt' of the surface quality. It should be noticed that those parameter values are linked to ISO standards, e.g. ISO 4287 [61] defining terms and parameters for surface determination by profile methods

and so the measurement length specifies the cut-off and filtering process, i.e. roughness parameters generated by profilers are often by default based on modified surface data.

In the aspect of mirror-like surfaces, and the search for defects and textures, 2D analyses are not enough since it is nearly impossible to distinguish between different types of features, such as a pit or a valley, if they are captured at all [57,59], and consequently profilers should be rarely used in that context. It is stated that noise levels better than 0.1 nm (rms – root-mean-square) are needed to provide reliable results for high gloss polished surfaces [62], a level that far from all used profilers in industry reach.

Mechanical profilers can also be used for areal measurements [63], but since they often need several hours to complete one single measurement other techniques are preferred [59].

#### Interferometer

This technique builds up areal topographic images based on interference patterns arising when two reflected light beams, one from the sample and one from the reference mirror, originated from a single light beam are recombined. It is their different travelling distances within the instrument, i.e. the relative phase between them, that give rise to interference patterns [56]. The phase shifting interferometry, commonly used for smooth surfaces, uses a monochromatic light source resulting in an excellent resolution allowing detecting features down to 1  $\mu$ m in lateral dimension and down to sub-nm in height. However, these are sensitive to vibrations, and are limited in their ability to measure steep slopes and height variations larger than the wavelength of the actual light source [57].

#### Scanning electron microscope

A focused electron beam raster-scans the surface; the energetic electrons interact with the atoms in the sample within a few nm to several  $\mu$ m of the surface, i.e. scattering events take place (primary electrons loose energy and/or change direction). Emitted signals in the form of e.g. secondary electrons and back-scattered electrons are then 'collected' by different sensors to produce surface images for topographical or chemical contrasts [56]. Combined with an energy dispersive x-ray spectrometer (EDS), even elemental analysis is allowed.

SEM images include no height data but, by combining two or three images oriented at slightly different angles, high-resolution topographic images can be reconstructed into 3D images and thus analysed as such, see e.g. [64-65].

#### <u>Scatterometer</u>

Unlike simple glossmeters, which measure the reflection in defined angles given in so called gloss units, scatterometers detect and collect diffusely and specularly reflected light, which based on scalar scattering theory, is converted into surface roughness, commonly rms values. One established technique, mainly for smooth surfaces (nm-roughness), is based on total integrated scatter (TIS) measurements, which is the light scattered into a hemisphere above the measured sample divided by the total light reflected by the surface [58].

A type of scatterometer has been developed at Halmstad University in purpose to be used for in-situ surface quality control for manual as well as automated polishing processes. The technology is patent pending, but a brief explanation of this new method 'for quantitative measurement of surface accuracy' is presented in [66].

#### 4.3.2 Surface characterisation methods

This chapter summarise possible tools for surface characterisation rather than defined methods for the same. The chapter is divided into two parts; qualitative methods describing surface quality based on subjective estimations represented by various non-measurable characteristics, and quantitative methods describing surface quality based on measurable attributes represented by numerical parameters.

#### **Qualitative methods**

Visual- and parametric comparisons with help of reference samples are widely used in industry, e.g. in workshops for assessments of machined surfaces providing both visual and tactile estimations in combination with known roughness values. Other methods are based on surface observations using optical microscopy, e.g. to identify and classify porosity in number and size or to control specific surface structures. Below the use of comparison specimens for mould finishing will be discussed, as well as a polishability evaluation based on pull-outs developed by Uddeholms AB.

#### Comparison specimen

Comparison standards, providing both visual and tactile (fingernail) assessments of machined surfaces, can be found in various collections. E.g. the Roughness Comparison Specimens including differently machined surfaces with given roughness values [67] and the Pocket S.P.I. Mold Finishing Guide, a finish gauge in pocket format offering cross-references between average roughness values and the S.P.I. scale [68], (see figure 8). Other alternatives include both metal and plastic surfaces, e.g. the Mold Finish Comparison Kit, a small box providing steel and plastic comparison surfaces in combination with manufacturing hints and average roughness values [69] and Scala Zanola, a manual including suggested finishing procedures with expected roughness levels with metal and plastic reference samples [70]. Many polishing shops also manufacture their own specimen to facilitate surface quality discussions with customers – customers have demands of component appearance, the polisher has the knowhow to achieve it. However, comparison specimens require experienced operators to get reliable results and, they do not include any criteria for defect assessment.



*Figure 8: The Pocket S.P.I. Mold Finishing Guide providing short guidelines for surface preparation, expected surface finish and average roughness values.* 

#### Polishability evaluation based on pull-outs

A practical way to test polishability is to examine standard polished workpieces using optical microscopy to detect the degree of porosity. The evaluation procedure described here was developed at Uddeholms AB in Hagfors [71].

Polishing parameters should be set to facilitate pull-outs, i.e. from the sample surface torn out grains or particles. The sample surfaces are then classified into five levels based on the occurrence of detected pull-outs and their size; from 1 - few small pores leading to high polishability, to 5 - many pores of all sizes leading to low polishability. Only pull-outs with diameters  $\geq 10 \ \mu m$  are included, which means that both smaller holes/pores and other types of defects are excluded by this evaluation procedure. The pull-out detection is performed by experienced employees, but is nevertheless subjective and the results vary slightly from person to person.

#### **<u>Ouantitative methods/tools</u>**

There are many reasons to translate human interpretation into numbers, e.g. to facilitate discussions regarding surface quality and to standardise surface criteria in specifications. Numbers, i.e. parameters, are used to reduce and summarise relevant information needed to describe the surface quality which in turn can be used to control manufacturing processes, and to correlate the surface topography to its functional properties [72]. Collected data most often need to be processed in sufficient ways to be useful, e.g. to separate adequate surface features from noise and superfluous data. Figure 9 attempts to give an overview of the steps used to divide and sort surface data in order to be able to describe desired surface properties/functions with relevant numerical values. Each step is described in more detail below.



*Figure 9: An overview of different steps for processing of measured surface data. Gray fields consider the stage of surface data as denoted in ISO 25178-2 [73].* 

#### <u>Measuring</u>

Different types of instruments are based on different physical principles and thus resulting images/profiles are affected in various ways. Mechanical profilers are e.g. limited by their tip shape/radii [59], while optical instruments are e.g. limited by their lens system, defined by the Airy disc/pattern, and imaging ability according to the optical properties of the measurand substrate, such as the complex refractive index [56,74].

Except for the choice of instrument and decisions of where and when to measure selected samples/products, measurement data are dependent on the instrumentation setup and actual conditions e.g.

- the *calibration* status which secures accurate output data (i.e. the traceability [59]),
- the sampling which determines the distance between measured points,
- the number of measurements which is fixed if standard methods are used. It is also important to cover a sufficient part of the surface to get representative mean values; 5-10 measurements on uncoated steel sheets were found to give stable mean values for most surface texture parameters (±10% at the 95% confidence level) [75],
- the choice of *magnification*, which determines what features can be captured (partly connected to the sampling),
- environmental factors which include e.g. humidity and temperature,

- the *cleanness* of the sample surface which is crucial to avoid misleading surface information caused by artefacts like dust and debris as well as cleaners/detergents or oil left on the sample surface.

#### Pre-conditioning

Measured data are most often processed in different ways before further analysis to remove irrelevant features/components, such as measurement noise and non-measured points, which actually is a way to add information – non-measured points are replaced by valid values either by constant numbers or values defined according to the neighbourhood.

#### Form removing

An F-operator, defined as an 'operator which removes form from the primary surface' [73], is applied to remove the nominal form since the amplitudes of these long wavelengths disturb topographical studies of parameter values; commonly a polynomial is fitted to and subtracted from the measured surface.

#### Determining significant features/components

There are several tools, single filters or more advanced multistep methods, to use in order to determine and separate significant features and surface components from insignificant ones, i.e. to find functional features and the scale of interest.

In this work, mainly robust Gaussian filters have been used. They are based on wavelength separation, and unlike Gaussian filters, they take features like grooves and peaks into account [76]. Morphological filters are based on structuring elements, e.g. discs or spheres, traversing the surface image from above or below, and could therefore be preferable for function related analysis of surfaces [56].

Feature characterisation is well described by five stages in the newly released standard for surface texture determination, ISO 25 178-2 [73]. Insignificant points are excluded from the surface image by a segmentation process identifying the features/motifs of interest, e.g. hills or dales, ending up in new data sets containing reduced surface information.

Other methods include e.g. the relative area analysis, a type of scale-sensitive fractal analysis where triangular tiles at different scales represent the areal scale of observation. The purpose is to link that scale to surface functions and process parameters in order to predict surface behaviour and describe surface topographies with few, but relevant parameters capturing the essence of the same [73,77-78]. Wavelet analysis, i.e. space-frequency analysis, provides decompositions of the wavelength components which mean that each surface component can be studied at separate scales [79].

Image processing is used either to improve or otherwise change the visual appearance of images, or to prepare images for further analysis of relevant features [72]. Sobel edge operators for edge detection, mentioned in Figure 9, is just one of

many tools that can be used to reduce or rearrange measured data to visualise and sort out surface characteristics.

#### Quantification

To quantitatively describe surfaces simply means to put numbers on them. The ISO 17825-2 [73] distinguishes between two parameter classes, the field and the feature parameters. Field parameters are based on statistics involving all measured data points, e.g. height and average parameters related to surface amplitude, and areal parameters based on the areal material ratio of the surface. Feature parameters are based on statistics from a sub-set of the measured data points as they are defined to describe significant feature attributes like their height, volume or distribution [80].

Comparisons between parameter values based on measurements on real samples in production and values stated in e.g. polishing guides, must be made with prudence since measurement data, modified or not, vary significantly according to actual setup and post-processes. This means it is of great importance to consider used measurement equipment and followed data processing to be able to draw correct conclusions out of the surface analysis, i.e. to use a well considered metrology framework.

## **5** Results and discussion

In the following subchapters empirical results from the five appended papers, and additional results, will be summarised and put into context of the surface control loop presented in figure 3 in order to clarify the relationship between the studies.

The chapter will be divided according to the three loops; chapter 5.1 connects to the Component loop, chapter 5.2 to the Steel loop, and chapter 5.3 to the Mould loop. Each chapter ends with a sum-up of the chapter linking back to the surface control loop.

#### 5.1 The Component loop

#### Paper II and additional results

The Component loop considers the production of injection moulded plastic parts (figure 10); from process parameters through estimations of final surface functions. In this work the main focus has been on the appearance of the plastic components and how it is related to the mould surface quality. The purpose was to study to what extent defect structures were transferred from the mould surface into the plastic surface, i.e. the function of the plastic components was linked to

the function of the mould and the steel by the characterisation facets. Also parts of the manufacturing facets were involved in the tests in terms of varied mould temperature during injection moulding.



Figure 10: The Component loop.

#### 5.1.1 Experimental

Injection moulded plastic plaques in the size of 150x105x4 mm were produced in three different mould inserts; one out of an ESR material, and two out of PM materials. The insert cavities were divided into two fields, one half was manually polished into an 'optimal' finish, i.e. the very best result achieved by adapted techniques, the other one was manually polished to a 'standard' finish, i.e. the result achieved using a general polishing technique.

Areal topography measurements were mainly performed with a NewView 7100 white light interferometer, quoted vertical resolution: 0.1 nm; sampling: 0.22  $\mu$ m [81]. Relocated measurements of inserts and corresponding plastic plaques were analysed with the proprietary software MountainsMap Premium [82].

#### 5.1.2 Results and discussion

The difference in surface quality between the two fields was measurable both on the plastic plaques and the moulds; the 'standard' fields on the moulds were measured to include more peaks and valleys than the 'optimal' fields, differences that were transferred into the plastic plaque surfaces.

It was also made clear that holes down to some few microns in diameter were replicated into the plastic plaques. The overlaid profiles in figure 11 shows how two smaller holes (diameter of approximately 8 and 3  $\mu$ m) were transferred into the plastics; the holes were hardly detected on the Makrolon types whereas the others replicated the holes in various grades, differences which were also visible to the naked eye. It was expected that the Pocan type (black line in figure 11) should have a better replica-behaviour than the others due to its longer solidification time, but it was hardly measurable in the performed study.



Figure 11: The table lists included plastic materials and their colour marking in the graph showing relocated profiles of two detected pin-holes on the mould, and how they are transferred into the plastic plaques. The mould-profile is inverted, and dashed, since the interferometer failed to measure inside the holes; only the 300/90 and 280/90 samples are included.

Finer grooves from the mould surfaces were easily detected on the Makrolon plaque, while only larger grooves were captured on the Novodur and Bayblend plaques (figure 12).

<sup>&</sup>lt;sup>1</sup>Bayer MaterialSience AG, Germany, <u>www.bayermaterialscience.com</u> <sup>2</sup>Inoes, United Kingdom, <u>www.ineos.com</u>

<sup>&</sup>lt;sup>3</sup>Lanxess Deutschland GmbH, Germany, <u>www.lanxess.com</u>



*Figure 12: Images illustrating the difference in groove replication between different polymer types.* 

It could also be concluded that lower mould temperatures were preferable to lessen the replication of defect structures originated from the mould surface, see figure 13. However, the expected difference between the two Makrolon types was not detected; Makrolon 2447 has lower viscosity than Makrolon 2647 and should therefore reproduce surface structures better according to the supplier.



Figure 13: Relocated profiles crossing a 75 µm defect (in diameter) transferred from one of the PM inserts. Makrolon AL2647 - Bright green, and Makrolon AL2447 – Dark green; dashed lines – higher mould temperature.

The microstructure itself seemed to be transferred into the plastic surfaces. Figure 14 shows an example of the ESR insert and one from the PM insert; the interferometer image of the Makrolon plate from the PM insert clearly shows up a surface pattern similar to the carbide structure from the mould.



*Figure 14: The microstructure seemed to be replicated into the plastic plaques; micrographs and interferometric images are not relocated.* 

Gloss and transmission measurements were performed on four of the Makrolon AL 2647 plates, but no significant difference between the two fields of the mould were detected, and the results are therefore not further discussed.

#### 5.1.3 Summing up the Component loop

It can be concluded that different plastic materials and injection moulding parameters gave rise to various surface qualities, i.e. besides the mould surface quality, the plastic material selection and injection moulding parameters are crucial factors to reach high surface quality. This means that knowledge linked to all three loops is needed to optimise the production of plastic components and simultaneously achieve required surface quality;

- the steel microstructure seemed to be replicated into the plastic plaques, i.e. the choice of process route within MANU<sub>steel</sub> affects the overall finish of the plastic components,
- defects such as scratches and holes with diameters down to 2-3 microns were replicated into the plaques, thus need to be taken into account in mould surface assessments, i.e. a well defined CHAR<sub>mould</sub> facet with numerical values describing mould surface quality should be helpful to secure 'the right' surface quality level,
- lower mould temperatures lessen the replication of pin-holes, as well as properly chosen polymer types, i.e. the MANU<sub>comp</sub> affected the grade of replicability.

#### 5.2 The Steel loop

#### Paper II and IV

This loop considers the process to manufacture tool steels, and involves choice of process route, alloving elements, and heat treatments which all affect final tool steel properties (figure 15). This work aimed to study how steel mainly properties, non-metallic inclusions, affect the polishability and occurrence of various the defect structures, i.e. to link the steel function to the mould manufacturing – the

surface preparation - by the characterisation facets. Measured steel properties are aimed to be linked to mould surface qualities, which will be discussed in chapter 5.3 - The Mould loop.



Figure 15: The Steel loop.

#### 5.2.1 Experimental

An energy dispersive x-ray spectroscopy analysis was performed at Uddeholms AB in Hagfors; oxides and sulphides larger than 2.80  $\mu$ m, and nitrides and carbides larger than 4.0  $\mu$ m (equivalent diameter) were automatically registered. In the following text they will be referred to as particles/mm<sup>2</sup>.

36 steel grades, representing different processes routes, ingot sizes, and various heat treatments, i.e. steel grades with various microstructures and cleanness, were included. All samples were similarly prepared in an automatic polishing machine using three steps with soft carriers combined with diamond slurries (grain size 9, 3 and 1  $\mu$ m, respectively). Initial surfaces were ground.

#### 5.2.2 Results and discussion

Figure 16 summarises the results; as expected, PM materials have the lowest amount of particles and IC materials the highest. Most of the ESR and PM materials do include carbides (since they increase the wear resistance), but these were too small to be registered in the EDS-analysis.

It should be noticed that residues left by torn-out particles will be registered and classified as existing particles, which means that the method is supposed to be insensitive to defects caused by pull-outs.

This work does not differentiate between different types of oxides, i.e. inclusions bonded with oxygen, but the composition might influence the occurrence of defect structures. Figure 17 gives an overview of the size distribution of the oxides, and it is clear that smaller oxides are dominant.



*Figure 16: Particles per mm<sup>2</sup> (log-scale) for steel grades produced by different process routes.* 



*Figure 17: Oxide distribution for steel grades produced by different process routes to show the dominance of smaller oxides.* 

#### 5.2.3 Summing up the Steel loop

The PM materials were measured to have the lowest particle content, the IC and CC materials the highest. However, most ESR and PM materials do include carbides which were too small to be registered in the EDS-analysis, but yet accountable in terms of influence factors for tool steel properties, e.g. the polishability. The relationship between the steel manufacturing/function and the Mould loop will be further discussed in the next chapters, especially the occurrence of smaller particles since these have been a growing problem during recent years due to increasing demands on quality levels of steels as well as mould and plastic surfaces.

#### 5.3 The Mould loop

Paper I, III, IV and V

The Mould loop (figure 18) covers most of this work – the characterisation and manufacturing facets of moulds. The chapter is therefore divided into two; 'manufacturing of moulds' with the purpose to study the influence of the polishing strategy on surface quality, and 'characterisation of moulds' with the purpose to define quality levels, describe relevant defect structures for high gloss polished tool steel surfaces, and suggest a quantitative evaluation method for the same. This means that the Mould loop needs to be linked to the Steel loop to study the relationship between steel properties, surface preparation and mould surface quality, and to the Component loop to know which defect structures are of relevance for injection moulding of glossy plastic components.



Figure 18: The Mould loop.

#### 5.3.1 Experimental

The results will be based on four experimental set ups; the first includes the same steel grades as the ones included in the energy dispersive x-ray spectroscopy analysis discussed in chapter 5.2, the second includes 22 of these steel grades, the third and fourth includes just a few steel grades prepared in different polishing shops.

#### Surface preparation

The first set up was based on the same steel grades as those included in chapter 5.2.1. They were polished in an automatic polishing machine using three steps with soft carriers combined with a diamond slurry (grain size 9, 3 and 1  $\mu$ m); process parameters were set to facilitate pull-outs. Initial surfaces were ground.

The second set up was based on manually polished samples, prepared in a wellknown polishing shop. A typical surface preparation sequence includes one to six steps with stones or abrasive papers with decreasing grain sizes, and three to five steps with various carriers and diamond abrasives. Initial surfaces were ground.

The third set up includes samples polished by various polishing techniques, most often not specified in detail, and served as 'indicators' of delivered surface quality levels. In total eleven different polishers/automated polishing techniques were included in the work.

The fourth set up includes samples that were pre-ground (with  $Al_2O_3$ , 60 Mesh) before they were delivered to different polishing shops for stepwise surface preparation. The samples were divided into smaller fields, one for each preparation step, so that the development of the process could be studied. The polishers were told to achieve a surface quality corresponding to that needed for injection moulding of shiny plastic components using their own chosen abrasive preparation techniques.

#### Surface investigation

Presented particle contents originate from the energy dispersive x-ray spectroscopy analysis described in chapter 5.2.

A visual estimation of the samples included in the second set up was performed by the polisher, who ranked the samples according to the grade of pin-holes/pitting and raisings (outwardly directed defects [53]); referred to as the 'Visual estimation: Surface quality' in the text. 1 corresponds to a surface with no pin-holes and raisings, 9 to a surface with pin-holes and raisings all over. The polisher also ranked the samples according to the time and steps needed to achieve high gloss, further referred to as 'Ranking: easiness to grind & polish', where 1 means easy to polish, and 7 means hard to polish.

The polishability evaluation based on pull-outs is based on the samples included in the first set up. An optical microscope was used to examine the sample surfaces, which were classified into five levels;  $1 - \text{few small pores leading to high polishability, and 5 - many pores of all sizes leading to low polishability. The evaluation is described in more detail in chapter 4.3.2.$ 

A Phase Shift Technology MicroXam interferometer with a quoted vertical resolution of 0.1 nm and a sampling interval of 1  $\mu$ m was used to measure and examine the surfaces at different magnifications. The surface topography analysis, made with proprietary software MountainsMap Premium, was based on measurements with the objective magnification of x100 (approx. measurement area: 800x600  $\mu$ m); all images were levelled with respect to the least square plane and a

form removing step (polynomial fitting of order 2) was applied to get rid of any remaining curvatures. Parameter values are based on 15 measurements unless declared otherwise.

#### 5.3.2 Results and discussion – Manufacturing of moulds (MANU<sub>mould</sub>) Paper IV

ESR samples prepared by different techniques appeared to have the same surface finish, but closer investigations clearly showed quality variations (see figure 19); e.g. Polisher Rl should have used a longer time and/or other tools to get rid of remaining scratches, whilst Polisher B seems to have used too soft carriers and/or too long time and low pressure ending up with a wavy structure - due to ploughing effects? One of the participating polishers stressed the importance to make use of the cutting effect of abrasives, i.e. to avoid rolling and smearing effects, another to change polishing direction between *and* within steps to secure elimination of grooves from previous preparation steps.



Figure 19: Interferometric images of ESR samples prepared by different polishers/techniques to illustrate quality variations of similar looking steel surfaces.

Step-wised polished ESR and PM samples indicated that (paper IV);

- Different surface preparation strategies can lead to equal final surface qualities;
- *Similar preparation techniques* can be used for different types of steel grades to achieve equal surface qualities. The polishers adapted their preparation strategies to the different steel grades, and it was clear that some succeeded better than others. Yet it remains to study how any changes of their strategies will affect final surface qualities; for instance, what if polisher C2 uses half the

time at each step keeping the other parameters unchanged (see figure 21)?

- Several preparation steps are favourable in order to reach good surface qualities;
- Even the *best suited steel grades* for high gloss polishing can be ruined by wrongly chosen preparation strategies.

It is tempting to believe that cleaner steel grades should be easier to high gloss polish into good surface qualities, but the studies during this work did not show such a tendency (figure 20).

Also the hardness seemed to have minor influence on 'the easiness to grind and polish' (figure 20) even though materials with higher hardness levels often are reported to result in better surface qualities, as in [45] where surface qualities of hardened and unhardened (hardness levels below 1300 MPa) tool steels were compared. Since all samples included in this study (except one) could be considered to be 'materials with higher hardness levels', the hardness should not be a major factor affecting the surface quality.



Figure 20: Summary of the results based on the ranking 'Easiness to grind and polish' made by the polisher; the numbers in the table correspond to the amount of samples that were graded as 1- easy to grind/polish, to 7- hard to grind/polish. The graphs show that the particle density and the hardness had minor influence on the easiness to grind and polish the steels.

The occurrence of defect structures is widely discussed, and there are as many theories as performers within the field. The mould inserts (used in the test with the plastic plaques discussed in chapter 5.1.1-2) clearly showed that the same piece of

tool steel achieved different surface qualities due to various degrees of adapted polishing techniques. Pitting is one type of defect structure causing lots of trouble in industry, so how to avoid it? During the experiments, pitting tended to show up when softer carriers were introduced; some of the polishers observed that it occurred when too soft tools and too high speeds were applied. Also 'orange peel' was said to be caused by too soft tools, but when spending too much time on the last steps. Probably steel grades with less homogeneous microstructures, e.g. with segregations, tend to form wavy defect structures more easily than those without.

Initial experiments with a 6-axis robot for automated surface preparation showed promising results on a PM material; two steps with rotating tools (carrier: polymer foam, abrasives: diamond fluids) generated similar surface roughness compared to Polisher A using fewer steps but somewhat longer time (Figure 21). As can be seen, grooves in various dimensions ruined the surface quality.



Figure 21: Left: average surface roughness represented by the Sq value (Rootmean-square deviation of the surface) vs. the accumulated time (in minutes) after each step. Right: interferometric image of a PM sample prepared by a robot assisted polishing process denoted Polisher Rr.

Models to optimise polishing processes, based on relationships between surface roughness and number of cycles/passes to predict the resulting surface roughness and so when it was time to switch to finer abrasives have been presented in [83-84]. Others have analysed the texture direction to decide when it was time to shift from one preparation step to the next one; the ratio of old grooves to current polishing direction was considered [85]. However local defects, like deeper grooves or holes, can be left on the surface even if the overall surface roughness has been reached, see e.g. figures 19 and 21. This means that measurement techniques sensitive to local surface deviations are needed to secure defect free mould surfaces. The importance to recognise defects and their influence on functional properties, and the relation of the defined defects to surface roughness and waviness is also pointed out by [56].

To summarise, there are various combinations of tools and process parameters leading to satisfactory, and even excellent, surface qualities, see also [86-87], which means that specific polishing instructions are not easily defined. However, it is reasonable to specify suitable tools and process parameter ranges to reach good surface quality, i.e. to describe 'basic rules' behind surface preparation and the capability of different steel grades. Beyond this, it is up to the operator/polisher to adapt preparation techniques to the actual mould – both material properties and mould geometries require adapted polishing strategies, e.g. are larger tools needed for large mould surfaces such as moulds for bumpers. Guides assisting and piloting troubleshooting could also be helpful, e.g. the Trouble-Shooting tool provided by Struers A/S describing artefacts and giving stepwise instructions for possible preparation improvements to avoid occurred defects [88], but again it is the experienced polishers who hold the most valuable information. No wonder the polishing so far has been more of an art than a science...

#### 5.3.3 Summing up MANU<sub>mould</sub>

The surface preparation strategy turned out to be a major factor influencing the mould surface quality, whilst the hardness and cleanness of the steels were less important – as long as they were kept on reasonable levels. Even though it seemed to be impossible to give detailed surface preparation instructions, it was concluded that;

- similar preparation techniques can be used to produce high-quality surfaces on different steel grades.
- a 'several-step-strategy' was favourable.
- the mould surface quality needs to be studied in detail, i.e. CHAR<sub>mould</sub> is of great importance to secure proper mould surfaces with today's demands on roughness levels down to nanometers and nearly defect free surfaces – not all 'mirror-like' surfaces had desirable topographies for injection moulding.
- neither the hardness nor the cleanness of the steels seemed to influence the easiness to prepare the steel surfaces in any clear direction; even the best suited steel grades could be ruined by wrongly chosen preparation strategies.

In other words there is a strong link between the manufacturing of moulds and the mould surface quality, and thus to the Component loop in terms of the surface quality of moulded plastic components.

#### 5.3.4 Results and discussion – Characterisation of moulds (CHAR<sub>mould</sub>) Paper I, III, V

This chapter starts with the qualitative assessments as they form the basis for the suggested quantitative surface evaluation. Paper I motivates the choice of phase shifting interferometric microscopy as a technique to measure the surface

topography, while papers III and V mainly describe the development of the suggested evaluation method.

#### Polishability evaluation based on pull-outs

Figure 22 summarises the result which, again, shows that the cleaner the steel the fewer amounts of pull-outs (and so the better surface quality). However, the method had become less reliable due to increasingly cleaner steels which were evaluated as 1 even though they differed in surface quality. Smaller pores and other types of defect structures, such as raisings and wavy structures, that were all visible to the naked eye, did not fit into the defined scale.



Figure 22: Summary of the results based on the polishability evaluation. Steel grades with particle contents > 30 particles/mm<sup>2</sup> are excluded; 1 = good polishability, 5 = bad polishability.

#### Visual estimation: Surface quality

Manually polished samples were ranked, according to the grade of pin-holes/pitting and raisings (outwardly directed defects [53]) visible to the naked eye, in a well-known polishing shop. Figure 23 summarises the results; low amounts of particles (oxides, nitrides, sulphides and carbides) facilitated good surface qualities (i.e. surfaces with no/low amounts of pin-holes and raisings) but did not guarantee high surface quality.



Figure 23: Summary of the results based on the visual estimation. Steel grades with  $particle \ contents > 30 \ particles/mm^2 \ are \ excluded.$ 

#### Defect classification

During the project it was found that no universal definitions of defect structures existed. Steel producers as well as polishers and mould-users relied on their own descriptions and misunderstandings were common. Therefore, a defect chart [89] was developed in cooperation with Uddeholms AB to collect relevant defect structures in order to visualize, describe and name them using already established concepts. Figure 24 shows the defect classification which forms the base for the defect extraction in the quantitative evaluation; the full defect chart can be found in Appendix I.



Figure 24: A schematic view of included defect types, from left: inwardly directed defects, outwardly directed defects, areas that appear different compared to their surroundings, and wavy textures, from paper III.

#### Suggestion of a quantitative surface evaluation

The suggested evaluation procedure is based on a stepwise analysis to be able to extract relevant defect structures (a detailed description can be found in paper V). It is based on interferometric measurements aiming to capture the defects presented in

figure 24 and, by numerical parameters, sort the surfaces into three quality levels; good, accepted and non-accepted surface quality (see table 3). The levels were defined based on the visual estimation, and thus chosen to suit criteria for high gloss polished tool steel surfaces. Other fields of application have different requirements and included parameters need to be revised. Table 3 summarises results based on a selection of samples representing various quality levels.

Surface quality level		Field parameters				Non-standardised parameters			
		Sq	Str	Sk	Sk <sup>IV</sup>	AreaR	HeightR	AreaRe	DepthRe
		nm	-	nm	nm	μm <sup>2</sup>	nm	μm <sup>2</sup>	nm
Good surface quality		< 12	> 0.4	< 25	-	< 30	< 5	< 30	< 5
Accepted surface quality		12-20		25-35	-	30-200	5-15	30-100	5-15
Non-accepted surface quality		> 20	< 0.4	> 35	-	> 200	> 15	> 100	> 15
Steel Visually estimated grade surface quality									
РМ	Surface in very good condition, no pitting	7	0.6	17	12	14	3	2	1
PM 2	Surface in bad condition ('milky spots' all over), pitting	8	0.6	20	15	1	1	36	35
ESR 1	Surface in very good condition, min. pitting	4	0.6	10	6	23	13	18	14
ESR 2	Surface in good condition, but with severe pitting	5	0.4	13	6	16	5	53	28
ESR 3	Waviness	13	0.2	36	12	74	9	3	2
ESR 4	Surface in good condition, small grooves & scratches	10	0.2	26	10	6	3	15	9
IC 1	Surface in bad condition, severe pitting	10	0.3	19	9	256	20	98	69
IC 2	Surface in good condition, min. pitting and holes	8	0.4	16	8	65	18	148	64
Unknown	Orange peel & pitting detected	22	0.5	57	8	83	3	22	19

Table 3: Acceptance	levels and	d results	based	on a	selection	of	`samples.
1							1

As noticed in table 3, the PM 2, representing the group 'areas that appear different compared to their surroundings', cannot be sorted out by the presented procedure. Lack of samples and the fact that those defect structures tend to differ less in height/depth from the rest of the surface made them more problematic to capture. However, feature characterisation seemed to give more promising results (see paper

V); the 'milky spot' regions stand out by small, clustered motifs (hills), whilst the neighbourhood has randomly distributed motifs, see figure 25a. The percentage of motifs within the defined areas, i.e. the squares shown in figure 25b, could be used as acceptance levels for surface specifications.



Figure 25: a) Images based on the motif analysis of the PM 2 sample with 'milky spots' and an ESR sample. b) Results based on 12 squares (sized 200x200  $\mu$ m).

Attempts to link inclusion type and size to extracted defects gave variable results (see paper V), and need to be further investigated; relocated measurements are needed to 'tune' particle contents to extracted defects since the size and number of defects depend on the procedure used, e.g. larger recessions are likely to originate even from smaller oxides. Probably, the idea to correlate particle contents to final surface quality should be reformulated to instead correlate particle contents to the sensitivity of defect occurrence for different steel grades.

#### 5.3.5 Summing up CHAR<sub>mould</sub>

Based on the results from the characterisation of the plastic plaques, the  $CHAR_{comp}$ , and the visual surface estimation, the 'qualitative part' of the  $CHAR_{mould}$ , a quantitative surface evaluation could be developed. The procedure includes choice of measurement equipment as well as data processing and numerical parameters describing acceptance levels for high gloss polished tool steel surfaces, i.e. a metrology framework to quantify surface quality.

The suggested method also relates to steel properties, the  $FUNC_{steel}$ , in terms of particle contents; steel grades with higher particle contents were measured to have higher defect levels. However, correlations between particle contents, detected defects and final surface quality need to be further investigated since also the surface preparation strongly impacts on how particles present occur as defects.

# **6** Conclusions

The overall objective with this thesis was to develop a metrology framework to quantitatively characterise high gloss polished tool steels, i.e. to find a method to put numerical parameters on the surfaces.

An extended version of the surface control loop was used as a base for this work. To be adapted to the process chain of injection moulded plastic components, three nested loops were needed – the Steel, the Mould, and the Component loops – in order to clarify the relationship between their function facets. Studies within all three characterisation facets were performed to be able to distinguish between surface defects which occurred during the different manufacturing facets and in which facet they could be avoided; e.g. thin scratches present on the plastic surface can be caused by a damaged or improper polished mould surface (FUNC<sub>mould</sub>), but also by wrongly chosen process parameters (MANU<sub>comp</sub>), and if excellent surface quality is required (FUNC<sub>comp</sub>), i.e. the results striven for, a proper steel grade (FUNC<sub>steel</sub>) should be selected to facilitate the surface preparation of the mould (MANU<sub>mould</sub>) and secure a 'defect free' mould surface (FUNC<sub>mould</sub>).

Based on areal surface metrology, defect classification and image analysis based surface characterisation, an evaluation procedure for polished tool steel surfaces could be developed, initially tested and verified in this thesis. The suggested method involves descriptions of relevant defect structures and acceptance levels for high gloss polished tool steels in the form of numerical parameter values based on interferometric measurements.

It could also be concluded within each of the three loops that;

- the degrees of cleanness of the steels were less important as long as they were kept on reasonable levels (MANU<sub>steel</sub>);
- the surface preparation strategy turned out to be a major factor influencing the mould surface quality (MANU<sub>mould</sub> vs. FUNC<sub>mould</sub>), e.g. it was found that a 'several-step-strategy' was favourable to avoid defect structures.
- not all 'mirror-like' mould surfaces had desirable topographies for injection moulding (FUNC<sub>mould</sub>), i.e. a well defined mould surface assessment with numerical values describing mould surface quality (CHAR<sub>mould</sub>) should be helpful to secure proper mould surfaces with today's demands on roughness levels down to nanometers and nearly defect free surfaces to reach 'the right' surface quality of the components (FUNC<sub>comp</sub>).

#### 6.1 Future work

Future work within this field would include further development of automated polishing equipments, both for industrial and research use. The latter should facilitate more detailed studies of polishing mechanisms and how various steel grades are affected by different combinations of process parameters. E.g. it should be of interest to provoke the occurrence of defect structures, such as pitting and orange peel, in order to assess the sensibility of different steel grades.

In-line measurement techniques need to be developed, and the correlation between surface defects transferred from mould surfaces to those occurring due to moulding parameters and polymer properties need to be studied to be able to reach higher surface qualities with less labour/work. One possible solution could be to use test moulds with artificial defect structures in order to study and test the replicability of different polymers and moulding conditions.

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# **APPENDIX I**

Defect chart and hints for high gloss polishing of steel surfaces

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# DEFECT CHART AND HINTS FOR HIGH GLOSS POLISHING OF STEEL SURFACES





#### UDDEHOLM DEFECT CHART -

#### PITTING

Scattered (pin) holes dispersed over the majority of the surface.

#### HINTS

- Shorten the polishing time (use enough but short steps)
- Use lower pressure
- Use harder carriers/tools combination diamond paste and lubricants is important
- Avoid unidirectional movements during preparation of the surfaces
- Dry the workpiece and store properly to avoid corrosion attacks on the surface
- If the pitting defects only appears in a local area on the surfaces it probably due to impurities in the material

#### COMET TAILS

Scattered holes with a tail, dispersed over the majority of the surface.

#### HINTS

- Avoid unidirectional movements
- Use higher rotational speed if manual polishing





#### 3D MEASUREMENT AND PROFILE



0 100 200 300 400 500 600 700 800 µm



HOLE

Smaller irregular or circular shaped cavity, e.g. pores, pinholes and imprints by abrasives.

#### HINTS

- Choose a cleaner steel i.e. ESR steel grade
- Use softer carriers/tools (without lint)
- Use lower pressure
- Napless polishing cloths reduce the risk for pull-outs
- Use a fluoride-free polishing cloth

#### **GROOVE** (scratches)

Longitudinal recession with rounded/flat bottom.

#### HINTS

- Clean the workpiece, tools etc. between every polishing step; remaining abrasives can scratch the surface by accident
- Be sure that marks left from previous preparation steps (e.g. turning or grinding marks) are removed
- Check if the hardness is too low









#### UDDEHOLM DEFECT CHART -

#### RELIEF

Hill-like formations in all kind of geometries covering the majority of the surface.

#### HINTS

- Choose a cleaner steel i.e.ESR steel grade
- Use harder carriers/tools
- Choose a more homogeneous steel material. Softer areas tend to be more polished than harder ones (pre-stage to orange peel)
- Decrease the polishing time (use enough but short steps)
- Use lower pressure

#### PEAK/RAISING

Small outwardly directed feature, often irregularly shaped, e.g. bare laid inclusions.

#### HINTS

- Choose a cleaner steel material
- Clean the workpiece to avoid surface contamination
- Use lower pressure, larger abrasive sizes, polishing cloths with higher resilience and/or a lubricant with higher viscosity to avoid embedded abrasives

#### **ORANGE PEEL**

Randomly, smooth valleys and hills covering the majority of the surface.

#### **HINTS**

- Shorten the polishing time (use enough but short steps)
- Use harder carriers/tools
- Use lower pressure
- Increase the lubrication in order to cool down the surface

#### **WAVINESS**

Longitudinal, smooth valleys and hills covering the majority of the surface.

#### HINTS

- Work with tools that have a good contact to the surface
- If waviness occurs go back to the first polishing step and change to a larger tool that fits better to the geometry of the surface to be polished





**₩** 0.5 mm

#### **3D MEASUREMENT** AND PROFILE



700 800 µm







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#### UDDEHOLM DEFECT CHART -

#### DISCOLORATION/ **STAINING**



Discoloured areas; e.g. "milky spots".

#### HINTS

- Inhomogeneous microstructure is adverse
- · Clean and dry the workpiece immediately after each preparation step, avoid hot water
- · Compressed air can contain oil or water, which might affect the surface
- · Cover the surface after polishing and store properly
- · Avoid overheating during previous preparation steps which get visible during the polishing process

#### HAZE



Areas with lower gloss than the surrounding ("silvery frosted appearance").

#### HINTS

- · Choose steel with homogenous material properties (e.g. without grain clusters in different directions and/or hardness variations)
- Might be correlated to previous processing (e.g. milling or welding operations)
- · Last polishing step discarded/cancelled
- Unclean surface (insuffizient carrier, wrong lubrication and diamond paste)

## **BURN MARK**

Physical destruction due too high surface temp. during surface preparation. On the sample surface three different defects are shown e.g. dark bluish areas from high pressure during polishing, point shaped burns caused by EDM process and linear and laminar burns caused by grinding, welding or other operations.



#### HINTS

- Use lubrication in order to cool down the workpiece during surface preparation
- Use lower pressure and/or speed during surface preparation

#### CRACK

Linear recession with a sharp bottom.

#### HINTS

· Crack result from surface tensions build up during the manufacturing process, i.e. change the preparation and/or the manufacturing process



0.5 mm

0.5 mm

#### **3D MEASUREMENT** AND PROFILE





200 300 400 500 600

700 800 Lin

100







# Introduction

This chart aims to give an overview of common defect structures, their size/shape and some "hints" to reduce/avoid them.

#### Name and description

PITTING

Scattered (pin) holes dispersed over the majority of the surface.



of defect

Туре

#### Avoiding strategies

#### HINTS

- Shorten the polishing time (use enough but short steps)
- Use lower pressure
- Use harder carriers/tools combination diamond paste and lubricants is important
- · Avoid unidirectional movements during preparation of the surfaces
- Dry the workpiece and store properly to avoid corrosion attacks on the surface
- If the pitting defects only appears in a local area on the surfaces it probably due to impurities in the material

# Picture of the defect



#### 3D measurement and profile



# DEFECT CLASSIFICATION

Inwardly directed imperfection



- Pitting
- Comet tails
- Hole
- Scratches/groove
- Crack

Outwardly directed imperfection



Relief

- Peak
- Discoloration
- Haze

Areas that appear different

compared to the surrounding

Burn mark

Wavy surface structure



- Orange peel
- Waviness

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