Wear Mechanisms in Press Hardening of Boron Steel

Identification and Laboratory Study

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Deep in the human unconscious is a pervasive need for a logical universe that makes sense. But the real universe is always one step beyond logic.

"The Sayings of Muad'Dib" by the Princess Irulan
Frank Herbert *in* Dune

Abstract

Press hardening is a hot sheet metal forming technique which allows producing lightweight components with complex geometry and outstanding mechanical properties. For this reason, use of press hardened components is steadily increasing in the automotive industry.

One of the factors affecting the competitiveness of press hardening is tool wear. Press hardening dies require intensive maintenance, which increases production costs and limits the efficiency of the process. The present thesis deals with wear on press hardening tools, following a methodology of integral analysis beginning at the industrial system and ending with laboratory tribological studies.

As a first step, a non destructive methodology for the analysis of industrial forming tools is developed and applied to the characterization of wear in industrial press hardening dies. Results obtained are later used to develop laboratory tests to study the most relevant tribological mechanisms and obtain full comprehension of the tool-component system. Finally, detailed study is performed on surface engineering techniques applicable to press hardening tools, including the effect on wear of tool surface finish and the performance of engineered surfaces.

Results of this work show that tool wear appears from a complex combination of chemical and mechanical interaction. The relative importance of these wear micromechanisms can be influenced by modifying the chemical, topographical and mechanical characteristics of the tool surface.

Keywords: Press Hardening, Hot Stamping, Wear, Boron Steel, Tribology, Surface Replication, High Temperature, PVD Coatings.

Resum

El procés d'estampació en calent és una tècnica de conformació de xapa metàl·lica a alta temperatura que permet obtenir components de baix pes, gran complexitat geomètrica i excel·lents propietats mecàniques. Per aquesta raó, l'estampació en calent és un procés particularment atractiu per a la indústria de l'automoció.

Un dels factors que afecten la competitivitat de l'estampació en calent és el desgast de les eines. Les matrius utilitzades requereixen un manteniment intensiu, cosa que afecta negativament l'eficiència del procés. El focus d'aquesta tesi és l'estudi del desgast en estampació en calent seguint una metodologia de treball integral que abarca des de l'anàlisi d'eines industrials fins al desenvolupament d'assaigs tribològics al laboratori.

El primer pas d'aquest treball és el desenvolupament d'una tècnica d'assaig no destructiva, que s'ha utilitzat per a caracteritzar el desgast en eines industrials reals. Els resultats obtinguts s'utilitzaren per a desenvolupar assaigs tribològics específics, que permetéssin estudiar els principals micromecanismes de desgast que apareixen a les eines industrials i aconseguir així una major comprensió dels fenòmens involucrats. Finalment, s'ha estudiat l'efecte de tècniques d'enginyeria de superfícies aplicables al sistema, incloent l'efecte de l'acabat superficial de l'eina sobre els mecanismes de desgast i el rendiment de superfícies modificades amb tractaments superficials.

Els resultats d'aquesta tesi mostren que el desgast a l'estampació en calent apareix per una combinació de factors d'interacció química i mecànica. La importància relativa d'aquests factors pot modular-se modificant les característiques químiques, mecàniques i topogràfiques de la superfície de l'eina.

Paraules clau: Estampació en Calent, Desgast, Acer al Bor, Tribologia, Replicació de Superfícies, Alta Temperatura, Recobriments PVD.

Preface

This dissertation is presented for the degree of Doctor at the Universitat Politècnica de Catalunya. It summarizes the research carried out by the author under the supervision of Dr Maria Dolors Riera and Dr Daniel Casellas at Fundació CTM Centre Tecnològic from September 2010 to June 2015.

This thesis is presented as a compilation of published articles accompanied by an introductory memory. The document includes seven chapters and two appendices.

Chapter 1 offers an introduction to the current state-of-the-art in the topics of press hardening, tribology and non-destructive inspection of industrial forming tools. Chapter 2 summarizes the objectives of this thesis, as well as the limitations in its scope. The main materials and treatments studied, as well as the test methodologies employed are described in Chapter 3. In Chapter 4, a brief summary of the most salient results in the appended articles is offered. Chapter 5 consists in a discussion about the wear micromechanisms acting in press hardening, based in the results exposed in the previous chapter. Finally, Chapter 6 presents the main conclusions of this thesis, and Chapter 7 proposes possible future investigation lines.

Appendix A includes the four articles which compose the main body of work of this thesis. Paper A deals with the characterization of a non-destructive technique for the analysis of wear in industrial tools based in surface replication, as well as the application of this methodology to the inspection of press hardening tools. Paper B presents a summary of laboratory tests investigating the different wear mechanisms appearing on the aluminium-tool steel system, which is further elaborated in Paper C. Results of these articles will be used to discuss wear micromechanisms in press hardening and their implications. Finally, Paper D presents a robust, scratch tests-based methodology for the mechanical characterization of coated systems to be used in tooling applications.

Last of all, two contributions of the author have been included in Appendix B as complementary information for the reader. These works present further exploration of the concepts developed in this thesis, exposed in the CHS2 series of conferences.

Acknowledgements

As clichéd as it sounds, this thesis is not only a summary of five years of research, but the closure to a chapter of my life. I would not have arrived here if it weren't for the contribution of many, many people. While it is almost impossible to acknowledge everything and everyone that has helped me arrive here, the next paragraphs attempt to include at least the main contributions.

First of all I want to thank Dr Maria Dolors Riera, my tesis director, for her interest and energy which made this thesis really take off and be finished in time. Thanks to Dr Daniel Casellas, co-director of this thesis and responsible for the Area of Materials Technology of CTM. Thanks for your trust, for your implication and for counting with me in this project that is CTM. Thanks also to Dr Jaume Caro, for his care and effort in revising this thesis, and for helping me improve it. I want to thank specially Dr Montse Vilaseca, my supervisor and responsible of tribology at CTM. Thanks for the day-to-day support, for introducing me to tribology and for, essentially, teaching me how to work. *Gràcies per tot.* Finally, I want to acknowledge the role of Prof. Luís Llanes, the one to introduce me at CTM: without his trust and his aid, all of this would have never happened.

Most of the research in this thesis has taken shape inside the frames of funded research projects, both state-wide (Forma0) and at the European level (FP7 TailorTool, RFCS TestTool and, less directly, RFCS Zincobor). I also want to thank acknowledge the funding received from the Catalan government through AGAUR under the scholarship 2010TEM24.

I want to thank our partners in these research projects, for their collaboration, and particularly to Gestamp and Volkswagen for their interest and availability, and for offering us the opportunity to study real production tools. I am also very grateful to the Machine Element division from the University of Luleå and particularly Prof. Braham Prakash for letting me stay almost two months with them, learning from one of the strongest tribology research groups in Europe. Thank to all of you for the collaboration, discussion and work together, and for taking me at your home as if it were mine.

My most sincere thanks to all the people in CTM for working with me in all of this, from sample preparation to SEM imaging to discussion of results, but also for the moral support I received during this last writing stage. Thanks particularly to my car partners and to the lunch-at-13h people. I couldn't have done this without all the ~~useful~~ ~~positive~~ utterly pointless daily discussion about topics healthily unrelated to press hardening, tools, wear or PhDs: without you I would have lost my head *way* before finishing. Thanks to the *Tribo-team* of Nuri and Giselle for taking weight off my shoulders these last couple monts, to Raül for the day-to-day support and psychotherapy and to the three of you for being a small family to me.

About that, thanks of course to my *actual* family, *Gemma, Cris i sobretot papa i mama; tot el que us hauria d'agraïr no hi cap aquí. Gràcies per haver-me donat tot el que he necessitat i una mica més, per animar-me a estudiar i per fer-ho possible. Si no fos per vosaltres, mai no hauria arribat aquí.*

And finally, thanks to Helena *per fer tot aquest camí amb mi tots aquests anys. Per acompanyar-me per les muntanyes i els deserts; per la paciéncia i pel suport; per l'humor, i per ajudar-me a mantenir els peus a terra.*
I gràcies Miquel, per ensenyar-me el que mai ningú m'havia ensenyat enlloc.

Last of all: to you, reader. After all, what sense is in a book that nobody will ever read? I hope that you can find in this thesis the answers you are looking for or, at least, a good place to begin your own research.

Vilanova i la Geltrú, October 2015

Appended Works

Appended Articles

These four articles constitute the main body of work of this thesis. They can be found in Appendix A.

Paper A

M. Vilaseca, J. Pujante, G. Ramírez, D. Casellas. *Adhesive wear analysis of PVD coated and uncoated hot stamping production tools.* Wear 308 (2013) pp. 148-154

Paper B

J. Pujante, L. Pelcastre, M. Vilaseca, D. Casellas, B. Prakash. *Investigations into wear and galling mechanism of aluminium alloy-tool steel tribopair at different temperatures.* Wear 308 (2013) pp. 193-198

Paper C

J. Pujante, M. Vilaseca, D. Casellas, M.D. Riera. *The Role of Adhesive Forces and Mechanical Interaction on Material Transfer in Hot Forming of Aluminium.* Tribology Letters 59 (2015) 1-10

Paper D

J. Pujante, M. Vilaseca, D. Casellas, M.D. Riera. *High temperature scratch testing of hard PVD coatings deposited on surface treated tool steel.* Surface and Coatings Technology 254 (2014) 352-357

Other Appended Works

These two papers contain work done in the context of this thesis, and were published in conference proceedings. They are included in Annex B.

Paper I

J. Pujante, M. Vilaseca, K. Eriksson, J. Clobes, M. Alsmann, D. Casellas. *Wear Mechanism Identification on Hot Stamping Tools.* In: *Proceedings of the 3rd International Conference on Hot Sheet Metal Forming of High-Performance Steel CHS2 2011*, Verlag Wissenschaftliche Scripten (2011) ISBN 978-3-942267-17-5, pp. 377-384.

Paper II

J. Pujante, G. Ramirez, A. Ademaj, K. Steinhoff, C. Dessain, M. Vilaseca, D. Casellas. *Measurement of Adhesive Wear on Hot Forming Tools.* In: Ed. Mats Oldenburg, Braham Prakash, Kurt Steinhoff, *Proceedings of the 4th International Conference on Hot Sheet Metal Forming of High-Performance Steel CHS2 2013.* Verlag Wissenschaftliche Scripten (2013) ISBN 978-3-942267-82-3, pp 371-378.

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Chapter 1

Introduction

1.1 Press Hardening

1.1.1 The process

Press hardening, also known as hot stamping, is a hot sheet metal forming process that allows obtaining lightweight components with complex shape and very high mechanical properties. It is defined as a “non-isothermal forming process for sheet metals, where forming and quenching take place in the same forming step” [1].

Authors Karbasian and Tekkaya [2] offered a comprehensive review of the main aspects of press hardening, later expanded by Naganathan and Penter in 2012 [3]. The main advantage of press hardening is that it allows producing components with very high mechanical properties (Table 1.1) while avoiding the problems, such as spring back on the component or damage including fracture on the tools, associated to cold forming of Ultra-High Strength Steels (UHSS) [4].

Table 1.1: Typical properties of press hardened boron steels [5].

	Yield Strength [MPa]	Tensile Strength [MPa]	Elongation [%]
Soft Annealed	350-550	500-700	>10
Press Hardened	1100	1500	6

Press hardening can be performed in two variants known as the direct and indirect methods.

In the direct method (Figure 1.1 a), blanks are austenitised at a temperature between 900 and 950 ºC for 4-10 minutes inside a furnace. Afterwards, an automated system transfers the austenitised blank to a set of cooled dies where it is formed in a single stroke while its temperature is inside the 650 850 ºC range. After forming, the dies are kept close and pressure is applied for a short period of time (typically 5 to 15 seconds). During this step, the cooled dies quench the formed component at a cooling rate between 50 to 100 ºC/s, ensuring full martensitic microstructure. The finished component is then extracted from the die. The total cycle time including transfer, forming and quenching typically takes 15 to 25 s [3].

In the indirect method (Figure 1.1 b) the blank is cold stamped in a conventional process to approximately 90 to 95 % of its final shape. Afterwards, this preform is austenitised as in the direct method and transferred to a press where it will be given its final form and quenched. While this method introduces an additional process step, it allows production of parts with increased complexity.

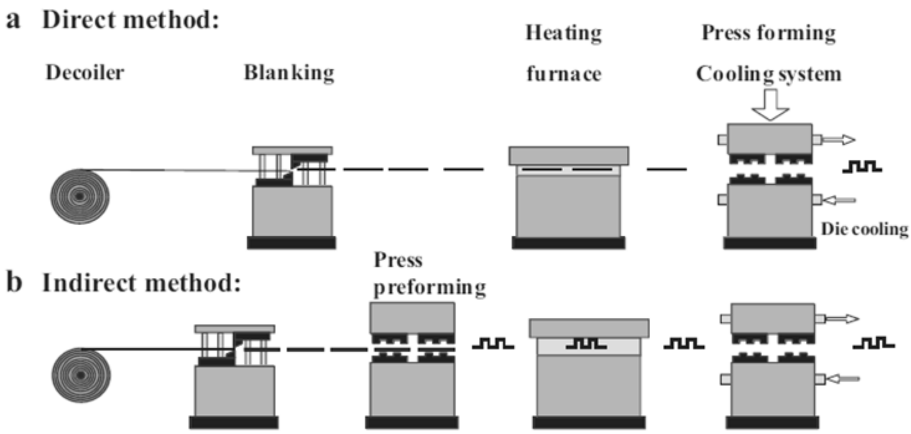


Figure 1.1: a) Direct and b) Indirect methods of hot stamping [6].

Press hardening was developed in 1977 in Sweden [2], originally applied to the production of agricultural implements. Its potential for the automotive industry was soon realised, and press hardened components were applied in passenger vehicles as soon as 1984 in the Saab 9000 [3].

Press hardening components are particularly interesting for structural and security car components, where they have become mainstream. As of 2015, the technology has become an industry standard for B-Pillars, but it is also widespread in A-Pillar and C-Pillar production as well as other components of the security cage [4, 7] (Figure 1.2).

The rising relevance of press hardening can be observed in both the industry and in international research. According to Karbasian and Tekkaya [2], the total production of press hardened parts increased from 3 million in 1987 to 8 million in 1997 to approximately 107 million in 2007. In 2011, a study by Schupfer and Steinhoff [9] foresaw a total demand of more than 600 million parts in 2015, well over the installed production capacity in that moment. Most of this growth was foreseen to come from the Asia-Pacific region, which was starting to invest in the technology following the lead of European manufacturers. The same authors predicted that this growing trend would not stabilize until after 2020, when press hardened components will have become standard practice in the industry for safety and structure parts.

While these predictions may seem adventurous, it is true that a strong trend in implementing press hardening has been maintained in the industry even after the advent of the economic crisis. For instance, the 6th generation of Volkswagen Golf, designed in 2008, contained a 6 % of UHSS components in its body. The

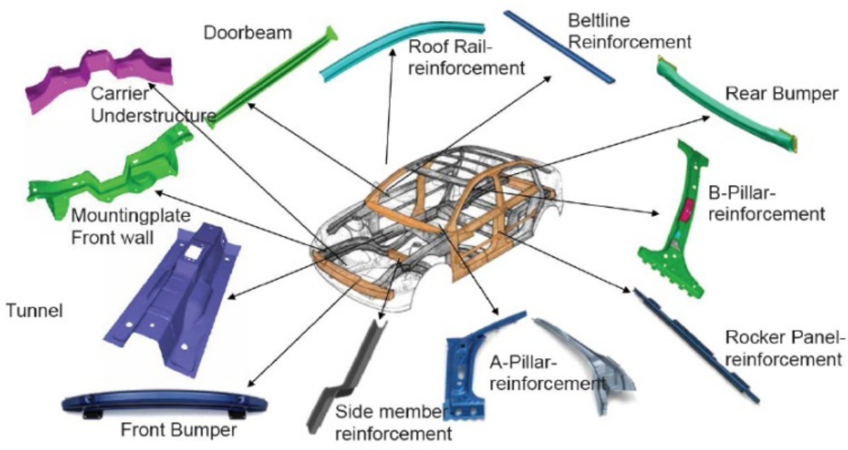


Figure 1.2: Components manufactured by Hot Stamping [3, 8].

2012 iteration (Golf VII) increases this share to 28 % of the body, with 24 % corresponding to press hardened parts. As of November 2015, the most iconic example is the newest Volvo CX90: press hardened steel composes 38 % of the body weight [10], the highest proportion in a passenger car.

In terms of research, press hardening has spurred research in fields as diverse as microstructural evolution [11–13], heat transfer and its implications [14, 15], development of new steel grades and coatings [6, 16], tribology and maintenance of tooling systems [17–19], innovative improvements of the technology [7, 20] and even plant layout and equipment.

As a reference, the number of contributions to the CHS2 Conference (International Conference on Sheet Metal Forming of High Performance Steel, a series of conferences focussed in the press hardening industry) has increased from 37 papers in its first edition in 2008 [21] to 81 in its 5th edition in 2015 [22].

1.1.2 Use of coatings on press hardening sheet steel

Press hardening can be performed on uncoated sheet steel. In this case, austenitisation needs to be done in protective atmosphere to avoid oxidation and scaling: presence of oxides on the sheet metal affects surface quality and tool wear, but also heat transfer [6, 15]. Decarburization is also a problem due to the high specific surface of the sheet metal.

These problems can be partially prevented by using protective atmosphere in the austenitisation furnace [6]. Even in this case, certain degree of oxidation and decarburisation (up to 60 µm depth) takes place during blank transfer from the furnace to the press [23]. Therefore, after production, components need to be shot blasted in order to correct these issues. For this reason, even though press hardening is also performed on uncoated material, the use coated sheet metal has become the industry standard.

AlSi coating in press hardening of Boron Steel The most commonly used press hardening material is aluminised boron steel sheet, produced by ArcelorMittal under the trademark USIBOR (USIBOR 1500P and USIBOR AlSi) and by other companies such as TKS and Nippon Steel under ArcelorMittal license [24]. This sheet steel is coated with a layer of Al-10 % Si with a melting point of approximately 600 ºC applied by continuous hot dipping and with a thickness between 20 and 36 µm, depending on specifications [25].

During heat treatment, high melting point Al-Si-Fe intermetallics form in the coating-substrate interface and grow into the coating; as a result, the coating does not melt. Full alloying of the coating requires 300-360 s and results in a complex sub-layer structure of four intermetallic phases plus a metallic Fe-based diffusion layer in the coating-substrate interface [25–27]. These intermetallics have been reported to be hard and brittle [28, 29].

The use of AlSi coated steel sheet prevents decarburisation and scaling of the steel sheet. It also provides corrosion protection, although limited to barrier protection [24]. Additionally, the coating generates a rough surface after heat treatments that results in great paintability even without additional treatment [25].

However, these materials show some limitations. First of all, use of the AlSi coated sheet metal results in forming constraints. The AlSi coating can achieve less deformation than the sheet metal below at room temperature. Therefore, these materials cannot be used in the indirect press hardening method, as the coating breaks during the cold stamping stage [2]. Moreover, the coating also imposes limitations on the direct press hardening process: blank heating temperature cannot exceed 12 ºC/s or the coating will melt [30], and development of the coating intermetallics requires austenitisation time of 360 seconds [27].

A second concern is that, while this coating offers barrier corrosion protection, it does not provide cathodic protection, something offered by Zn-based coatings [16, 24]. Finally, the presence of the coating dominates tool-workpiece contact during forming, and greatly affects the wear mechanisms observed. This will be further discussed in section 1.1.3. For all these reasons, there is interest in developing alternative coatings [31].

Zn-based coatings In the last years, interest of Zinc-based coatings for press hardening has increased in the automobile industry in the form of galvanized and galvannealed boron steel. Zinc-based coatings provide cathodic corrosion protection, enabling using press hardened components in corrosion-intensive applications such as the underside of a vehicle [16].

Application of Zn-based coatings has been hampered by the phenomenon of Liquid Metal Embrittlement, where liquid Zinc interacts with the boron steel substrate. The result is that large cracks (up to more than 100 µm deep) appear on the components during forming [6, 32, 33].

Liquid Metal Embrittlement can be avoided by modifying austenitisation parameters in order to increase the iron contents in the coating. However, this affects negatively the corrosion resistance of the coating [33]. For this reason, Zinc-based coatings are mainly restricted to indirect press hardening, where only limited deformation takes place at high temperature and liquid metal embrittlement is not a concern [2].

Other coatings While AlSi and Zn-based coatings dominate the press hardening industry, some alternatives exist.

Zinc-Nickel coatings were developed to avoid the liquid metal embrittlement issues of Zinc coatings while keeping galvanic protection [6,16]. Even though studies using this family of coatings showed promising results [34], ThyssenKrupp Steel, the main steel supplier producing Zn-Ni coated press hardening steel under the trademark GammaProtect, announced in 2013 that production was being discontinued.

Other metallic coating alternatives include Al-Zn and Zn-Al-Mg compositions [6]. However, none of them are currently applied in the industry.

Non-metallic or hybrid coatings have also been proposed. One of the most known examples is the Xtec coating developed by the German company Nano-X. This coating is applied as a varnish on the coil, forming a complex microstructure through a sol-gel process which includes organic and inorganic materials, notably aluminium particles to provide high temperature resistance. After curing, the coating has a thickness of approximately 7 µm. This coating has been used in both the direct and indirect methods and as an improvement to press hardening of uncoated sheet steel [35]. However, this coating imposes limitations on the component mainly in terms of paintability and weldability, and it has been mostly replaced by AlSi-coated steel.

Finally, some authors propose the use of oils on uncoated boron steel sheet. Oils would protect the sheet metal from contact with the environment and also act as a lubricant during forming, resulting in reduced friction and wear [36, 37]. However, they have not seen significant industrial application.

1.1.3 Wear in Press Hardening

Hot metal forming tools are subject to extremely harsh conditions: high mechanical loads, exposure to high temperatures and wide thermal cycles. Tools used in processes such as hot forging or extrusion suffer a wide range of damage mechanisms, including severe plastic deformation, mechanical and thermal fatigue, oxidation and thermal shock, with abrasive and adhesive wear only becoming a productivity-limiting factor only once these damages have been corrected [38, 39].

However, in press hardening tools these damage mechanisms are not the most critical in terms of process efficiency. Pressure and temperature ranges are relatively mild; instead, tool wear is the main factor defining tool life and maintenance needs [5, 40].

In the particular case of press hardening of AlSi coated material, the main active wear mechanism appears to be material transfer from the sheet metal coating to the tool. This material accumulates in irregular lumps on the tool surface (Figure 1.3) [40], which affect component quality. Accumulation of material transfer mechanism is fast and severe and results in press hardening tools requiring maintenance in the form of re-polishing in as few as 3000 production cycles [17].

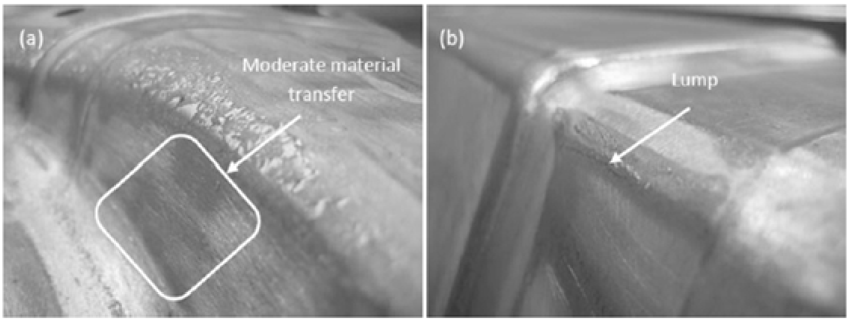


Figure 1.3: Wear on an industrial press hardening tool. a) Moderate material transfer over a surface; b) lumps of transferred material [40].

1.2 Wear and Damage in Forming Tools

1.2.1 Identification of Wear and Damage

Maintenance operations on forming tools are mostly performed based on criteria such as previous experience, intuition or observation of reject components being produced. This hampers the development of solutions for tool wear and damage: it is often impossible to discover how have the different mechanisms evolved and interacted before the tool was retired from production.

There is currently a trend to move from the current intuition-based maintenance to more complex systems, with the aim of optimising tool life and component quality. This necessity was already explored by Tsuchiya in 1999 [41], who proposed a six-step approach to the problem:

> *(...) (1) establishing a life determining standard, (2) observing damage, (3) discussing causes of damage, (4) eliminating unsuspected phenomena, (5) taking measures concerning the process and (6) steps for changing die materials and lubricants.* [41]

The author notes that the first four steps are of particular importance and key to the success of the action. If the causes leading to tool failure or need for tool maintenance are misidentified, corrective measures devised will not be efficient in improving process efficiency. Therefore, there is a need for developing techniques which can be reliably used for inspecting industrial tools and identifying damage mechanisms responsible for the end of service life.

Wear and damage studies are usually performed by analysing tools at the end of their production life. One of the first examples in the scientific literature can be seen in the work of Singh of 1973 [42], detailing the different damage mechanisms and wear morphologies observed in a failed forming tool.

A later work by Summerville et al [39] deals with the analysis of a failed hot forging tool, identifying features such as thermal fatigue damage and wear. These results are complemented by micrograph analysis, obtaining indications that damages can be related to the distribution of temperature and pressure during forming.

Pelcastre et al [43] performed destructive analysis on form fixture tools after more than 100 000 production cycles, including studies on microstructure and hardness. The authors succeeded in identifying the different wear and damage mechanisms appearing on the worn tools and their conclusions can be used in improving material selection.

One further step beyond characterization of wear mechanisms is quantification of their severity and control of their evolution. In this way, the relative importance of damage mechanisms can be better assessed and solutions designed and implemented. Smolik et al [44] studied wear in hot forging tools using a laboratory-scale setup. Different forging parameters were applied in order to evaluate the effect of tool and component temperature on system performance. Abachi et al [45] used a three dimensional measuring system to digitise tool contour in its initial state and after 678 forming cycles.

All these studies offer valuable data, but present some limitations. On the one hand, the obtained information corresponds to one single moment: there is no possibility to study the evolution in time of damage mechanisms, nor the specific nucleation and initiation steps. On the other hand, the need of retiring tools from production for their study restricts these analyses to either tool at the end of their service life or tools especially set apart for laboratory experiments.

Alternative analysis techniques have been proposed, such as integration of sensors in forming tools indirectly detecting wear [46,47]. However, these studies have been performed in tools much more simple than sheet metal forming dies, and press hardening tools in particular.

In this thesis, a non-destructive tool analysis based in surface replication has been proposed and studied. The use of surface replicas allows studying industrial tools without retiring them from production and solves some of the limitations of the studies described in this section.

1.2.2 Surface replication applied to non-destructive analysis of wear in forming tools

Surface replication consists in the application of a special polymer compound on the surface to be studied. This product flows on the surface copying its topography. After some minutes, it cures into a solid rubber and can be lifted off from the surface and studied in the laboratory [48].

A wide range exists of surface replication compounds, offering different capabilities in terms of shape conservation, attainable detail and curing time. Nilsson and Ohlsson [49] studied several different compounds, in order to determine their suitability to the inspection of machined surfaces. In general terms, differences between original and replica-obtained values were less than 10 %. Similar results were obtained by Jonsson [50].

The main application of surface replicas in materials science is the microstructural analysis in large components. ASTM standard 1351-01 (2006) “Standard Practice for Production and Evaluation of field Metallographic Replicas” describes a procedure for *in situ* polishing and etching, extraction of replicas and analysis through optical microscopy and Scanning Electron Microscopy. Even before this standard was published, replicas were already used in this field. An example can be found in a work published in 1995 by Jana [48] discussing different methodologies for analysing the microstructure of components, using different replica materials, from commercial acetate foils to liquid glue.

Replicas have also found use in analysis and quantification of corrosion appearing in components which could not be otherwise analysed, such as large structures or parts in service. Forlerer et al [51] applied surface replication to the study of a water pump in a nuclear reactor. Only by analysis though replicas, they were able to characterize the severity and cause (parasitic currents) of the observed damage.

Using replicas for the characterization of wear in various systems is also not unheard of. Eyre et al [52] used replicas to identify wear in different components in a combustion engine. The aim of this study was to later develop a laboratory setup reproducing the same wear mechanisms. Another example is the work of Cabanettes et al [53], using surface replication to quantify wear in engine cams with different surface finish. In this case, topography analysis was performed by means of optical profilometry (interferometry). The work includes a previous step characterizing the precision of the replicas, which was quantified as less of 5 % deviation between replica measurements and original surface measurement.

Finally, some authors have applied replicas to the inspection of metal working tools. Kaker et al [54] used surface replication to characterize surface finish and damage features on hot rolling rolls. The authors used Vinyl Polysiloxane impression materials, typically used in dentistry, to obtain surface replicas which were afterwards analysed by means of SEM and stored for future reference. Even though the aim of this work was not to evaluate the evolution in time of the damages, it was possible to determine the appearance of thermal shock cracks leading to spalling in the most damaged regions.

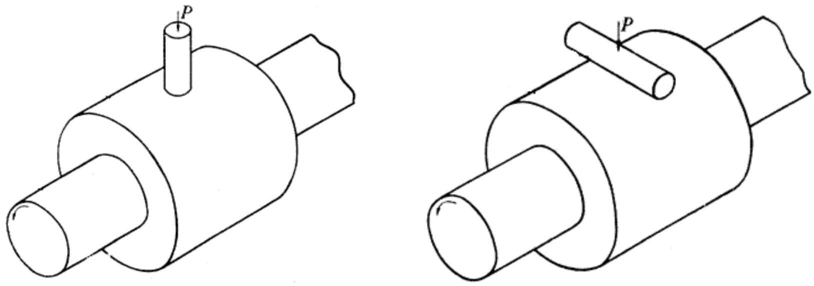
Jonsson [50] used surface replication to the inspection of sheet metal cutting dies, after discussing the applicability of different replica compositions. Ramírez et al [55] investigated wear in industrial sheet metal bending tools, by using surface replication and analysing replica cross-section. Using this methodology, it was possible to estimate the velocity of material loss processes and even evaluate the improvement in wear resistance obtained by using a tool manufactured in an alternative material.

These works showcase the applicability of surface replications to the detailed inspection of industrial metal working tools, while creating minimum disturbance to production.

1.3 Tribology

Compared to other aspects of materials science and engineering, there is a distinct lack of knowledge about wear and friction and little standardization in the methodologies associated to the measurement, characterization and even nomenclature of these phenomena [56].

The first scientific studies about tribology in the western world are popularly attributed to Leonardo da Vinci, who included in his works drawings of machines and setups which could be used to study friction. However, it is not until the 17th century that Guillaume Amonton announces his fundamental laws, later ratified by Coulomb and still used nowadays [57].



(a) Line contact (b) Point contact

Figure 1.4: Variations of the Pin-on-Ring contact used by Archard [58].

Amonton observes that, within a certain range of conditions, friction force is proportional to the normal force between two surfaces in relative movement and independent of the area of contact and sliding velocity. Amonton's laws are usually summarized in the following equation:

$$F_{Fr} = \mu N \tag{1.1}$$

Where F_{Fr} is the frictional force, tangential and opposing movement, μ is the frictional coefficient and N is the normal force between the contacting surfaces.

With the following development of engineering and machinery, interest in comprehending the phenomena arising in the contact between surfaces in relative movement became widespread, until the term tribology (from the Greek tribos, rubbing) was coined.

However, laboratory characterization of wear proved to be elusive. It was not until 1953 until Archard [58] performed one of the first methodical scientific works in the characterization of abrasive wear, its influence on contact conditions and the underlying micromechanisms. In this work, the role of asperity contact in wear and in contact mechanics is discussed, and a mathematical model for abrasive wear is proposed based on results obtained in a series of pin-on-ring tests (Figure 1.4). According to Archard, wear can be explained by the interaction of microscopic asperities in the surface, which suffer plastic deformation and fracture due to the locally high pressures appearing in asperity contact. This was in stark contrast to the leading theories in that moment, which pointed to layers of atoms being peeled from flat surfaces during their interaction.

From the works of Archard, as well as from one authored by Burwell and Strang [59], three conclusions can be drawn:

- Wear is proportional to applied load.
- Wear is independent from apparent contact area.
- Wear, normalised by sliding distance, is independent of sliding velocity.

These three conclusions were ratified in a later work by Archard [60] and finally summarized in the Archard equation, still used nowadays as a basis for modelling abrasive wear:

$$W = K\frac{P}{p_m}s \tag{1.2}$$

Where W is the volume of worn material, K is a constant reflecting the probability of a wear particle being broken off in each asperity contact, P is the applied pressure, p_m is the yield strength of the softest material of the pair and $s[m]$ is the total sliding distance.

It is worth noticing that this equation is similar to the Amonton's laws: both show proportionality to the applied load and are not affected by apparent area and sliding velocity.

Nowadays, many works use a constant K derived from Archard's equation as a "Wear constant", used to compare the performance of materials when tested under the same conditions [57]. While this constant is an entirely empirical value with no physical meaning, it is useful for the comparison of materials and tribosystems. These constants have a form similar to the following:

$$K = \frac{W}{sP} \tag{1.3}$$

Where K, in $[mm^3N^{-1}m^{-1}]$ is the wear constant or wear ratio, $W[mm^{-3}]$ is the worn volume, $s[m]$ is the total sliding distance and $P[N]$ is the normal load.

The main consequence of Archard's equation (equation 1.2) applied to the simulation of tribological systems is that the mechanical properties of the hardest element of the contact are not relevant in terms of wear: wear rate is inversely proportional to the hardness of the softest of the materials [59, 60]. Therefore, wear should occur in the softest material of the pair, at the same speed regardless of the used counterpart.

This is, however, not true: modifications in any of the elements of the contact pair results in highly altered tribological behaviour and even in the appearance of completely new wear mechanisms [57].

1.3.1 Studies of tribology at the laboratory scale

Tribological behaviour of systems is studied using a wide range of equipment commonly called tribometers. These test setups are not standardised, and are often developed for a particular system or application. Ferreiro et al [56] estimated in 100 the approximate amount of different tribotests being described in the literature in 2010, although less restrictive criteria could increase this number to more than 400. This is in stark contrast to normalised tests developed to study other material properties.

Most of these setups share common core characteristics. Tribometers include a minimum of two different specimens, which are put into contact in relative motion to each other and under a given load. One relevant factor in tribological tests is the geometry of the generated contact, as it affects contact conditions:

Point contact Surfaces contact in a single point (Figure 1.5 a and c). This setup is very robust, as alignment is not a concern. It is usually accomplished with ball against a flat surface (ball on disc or ball on flat configurations), contact between balls (as in the 4 ball test used in lubricant testing [57]) or between crossed cylinders [60, 61]. The main disadvantage of this wear geometry is that contact morphology quickly evolves into an area contact, as the softest component is worn or deformed.

Line contact Samples contact along a single line. This method requires alignment in at least one axis, to ensure that load is distributed equally along the contact line. Usual implementations are parallel cylinders [18] or cylinder-on-flat.

Flat contact Contact takes place on a whole area. This contact morphology is the most stable during the test, but requires very fine alignment between the two specimens to ensure that load is evenly distributed. Some examples are pin on disk [62, 63] (Figure 1.5 b) or ring on flat configurations.

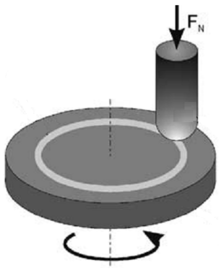
Other geometries Sometimes, contact morphology is not easily described. In some cases, a complex-shaped body will indent a softer counterpart, resulting in a complex contact area. In other cases, such as in third-body abrasion, contact between the worn surface and abrasive particles varies between the different morphologies. Finally, in some especial cases contact does not take place between solid surfaces, such as in tests dealing with erosion or cavitation [57].

These tests offer useful data: friction coefficients to be used in Finite Element simulation, understanding wear behaviour in closed systems, such as bearings, or identification of arising wear mechanisms in material pairs.

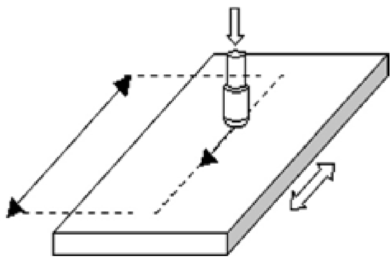
However, there is discussion [64, 66, 67] about whether this approach is valid in the case of simulating open systems such as forming tools, where new surfaces are constantly being put in contact. In these conditions, laboratory tests where the same wear track is slid on over and over lead to the formation of wear particles, complex tribolayers or protective oxides which result in wear mechanisms entirely different from the actual application.

Other authors, such as Hardell [18] or Santner [68], reason that valid conclusions can still be obtained provided that test configuration and conditions are carefully selected. A mid-term solution would be developing a simplified laboratory test and verifying that arising wear mechanisms are accurate by inspecting the actual system [52, 69]. In this way, simple, robust tests can be used instead of complex simulators, while still ensuring that relevant data is obtained.

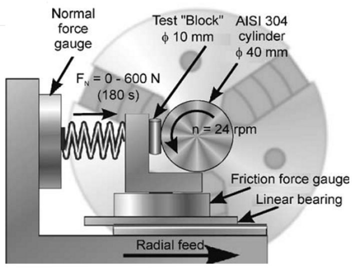
All these procedures require a certain degree of abstraction, through which a complex system is simplified into a laboratory test. According to Santner [68], certain distrust exists towards tribological results in the various disciplines of engineering: friction and wear values are obtained using simplified tests, often not representing the studied system. Results (designs, FE simulation) obtained using these data as inputs may be misleading, or entirely erroneous. Therefore, in some cases it may be reasonable to develop variations of existing tests, or even setups designed from scratch in order to ensure that the conditions of a particular system



(a) Ball on disk [64]



(b) Pin on flat [64]



(c) Crossed cylinders, point contact with rotation movement [65]

Figure 1.5: Examples of tribometer configurations.

are accurately reproduced. The term tribosimulators is often used to refer to tests custom-built to reproduce a particular tribological system.

1.3.2 Tribosimulation of press hardening

As elaborated in section 1.1.3, tool wear is the main reason for tool maintenance in press hardening. This, together with the rising interest in press hardening, has resulted in a large number of contributions in the open literature presenting press hardening tribosimulators of various levels of complexity.

A comprehensive laboratory study of galling in press hardening was performed by Hardell [18]. In his work, [62, 63], an Optimol SRV tribometer is used to reproduce contact during forming (Figure 1.6). The tool specimen is mounted in a reciprocating holder, oscillating at amplitude of 2 mm and 50 Hz frequency against a fixed, heated boron steel sheet metal specimen. The same setup was later used by Pelcastre [19, 40].

System behaviour is studied for temperatures up to 800 ºC and the performance of different tool materials and coatings compared. With this setup is possible to perform very high number of cycles in a very short time. On the other hand, this configuration results in repeated contact in a single wear track: formation of stable tribolayers and oxide glazes was observed.

Marzouki et al [66] attempted to avoid sliding over the same wear track. This work describes a simple press hardening tribosimulator based in a pin on disc configuration. In this setup, a flat-top manufactured in tool steel slid against a

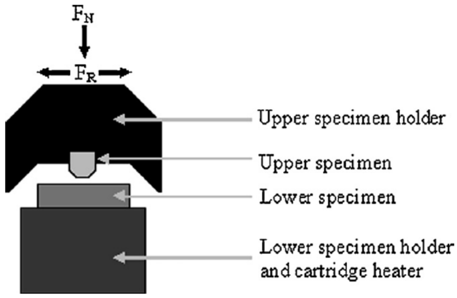


Figure 1.6: Test setup using an Optimol SRV system as described by Hardell [62].

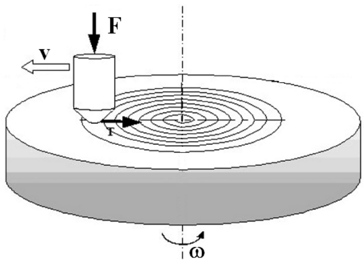


Figure 1.7: Press hardening simulator developed by Marzouki et al. [66].

boron steel sheet disk, describing a spiral wear path (Figure 1.7). This way, the pin constantly slid against fresh material, simulating the sliding against new blanks every production cycle.

Ghiotti et al developed a tribological test using a modified multifunction tribometer, that included thermal cycles in discontinuous sliding pin on disk tests [17, 30, 70]. This test was used to evaluate the coefficient of friction against tool steel of boron steels with AlSi coatings and Zn-based coatings, as well as the appearing wear mechanisms.

Merklein and Wieland [8] followed an approach based in a scratch test configuration, where flat-to-flat contact takes place between a flat-topped tool steel pin and a sheet metal sample (Figure 1.8). In this case, a total of 25 tracks, 10 mm each in length are generated. This test shows particular care in reproducing the thermal cycle suffered by press hardening tools, by cooling the pin between slide cycles. The test ensures that the tool specimen constantly slides against fresh strip material while keeping a non-complex setup different from industrial equipment. This setup is used to study wear mechanisms, but it is limited in the amount of sliding cycles that can be performed.

Both test setups generate flat-on-flat sliding and thermal cycles similar to the industrial operation conditions, while keeping a simplified test environment. However, this high degree of abstraction means that contact is localised on a very small area, which can be even smaller due to the difficulty in aligning surfaces in flat on flat contact. As a response to these possible limitations, test setups have been designed where tool-sheet metal interaction takes place in a manner more similar

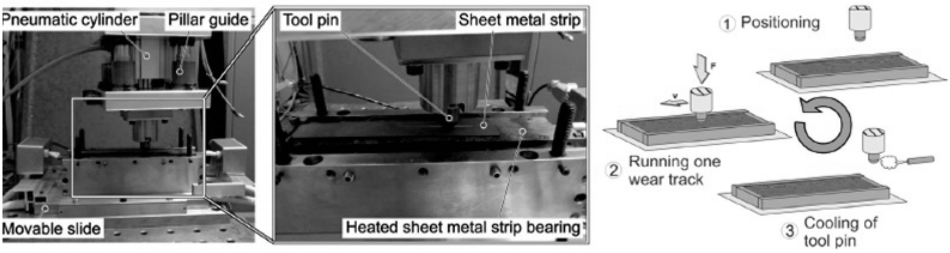


Figure 1.8: Test setup described by Merklein and Wieland [8].

to the industrial application. These tests are less abstract and require complex equipment, but reproduce even more closely the actual environment.

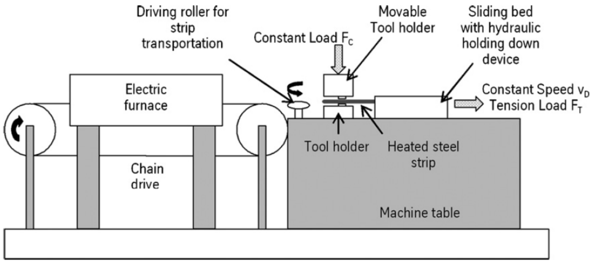
A popular test configuration is strip drawing-based equipment (Figure 1.9). In this setup, a strip of boron steel is heated up, clamped between two flat tool steel inserts using a controlled force actuator, and pulled at a constant velocity. Frictional force is measured during sliding. Tools and strip can be inspected afterwards. Variations of this setup have been reported by various authors in the scientific literature [30, 37, 71–76].

This test setup accurately reproduces sliding in press hardening, and has proved useful in obtaining friction coefficients to be used in Finite Element modelling. It is also useful to compare the effect on friction and sliding of different forming parameters [37], alternative materials [71], surface engineering [75] and even lubricants [76]. Finally, even though it is not designed to study long term wear in the industrial system (the total amount of sliding distance is very low), it is possible to study wear mechanisms on samples worn in this way.

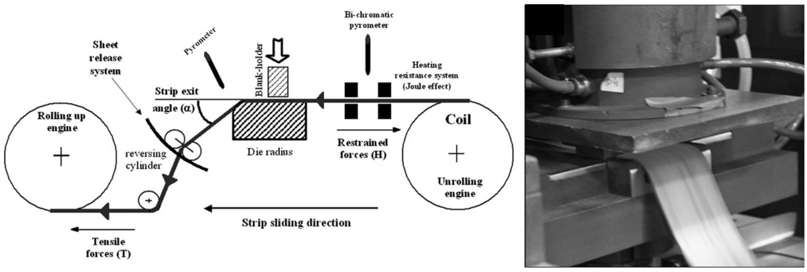
Similar equipment involves drawing the heated sheet metal strip over a radius. An example is the Deep Drawing Process Simulator used by Dessain et al. [72] and by Boher et al. [5] (Figure 1.9 b). This setup generates sliding on a significant contact area, as in strip drawing simulators, and can easily be automated in order to generate a high total amount of sliding. However, its main difference with the setups described until this point is that it introduces strip deformation during drawing.

Finally, other research groups have used complex, production-like setups to study in detail various aspects of press hardening in laboratory conditions, tribology among them. The production of U-shaped components of various geometries (also referred to as “hat shape” or “omega shape”) is a common configuration, as it allows reproducing most of the relevant factors in press hardening while keeping a simplified geometry [13, 24, 34, 77].

Another alternative are cup drawing-based devices. Geiger et al. [78] used a cup deep drawing device to characterize friction in press hardening, in order to obtain data for FE simulation. Kondratiuk and Kuhn [16] used a similar setup to evaluate the effect of different forming parameters, sheet metal coatings and tool surface engineering techniques on material transfer. This same device was used by Sobiek et al. [79] to investigate the effect of Physical Vapour Deposition (PVD) coatings on the tribological behaviour of the tooling.



(a) Linear strip drawing tribosimulator, as described by Kondratiuk [16].



(b) Deep-Drawing Process Simulator and detail of the hot strip sliding over the radius [5]

Figure 1.9: Different press hardening tribosimulators based in the strip drawing configuration.

These tribosimulators are not usually applied to study wear in press hardening, as acquisition of wear data would require cycle numbers in the same order of magnitude as in industrial tools (it is not an accelerated test). However, data obtained with such a setup would be almost as representative as in the direct study of an industrial system.

1.4 Surface Engineering in Forming Tools

Tool life has a direct impact on process efficiency and on the cost of produced components. In Die Casting, for instance, up to 20 % of the cost can be attributed to tooling [69]. Forming tools are expected to work for a high number of cycles, often in the hundreds of thousands. Therefore, surface modifications resulting in increased tool life or in improved performance are a valid investment. Two such factors will be studied in this thesis: surface finish and mechanical effect of surface engineering techniques, namely nitriding and PVD coatings.

1.4.1 Effect of surface finish on tribological performance

It is known that roughness of contacting surfaces has an effect on the wear and friction characteristics of a system. For instance, the effect of surface finish on wear is well studied for machine elements [32].

In the case of forming tools, surface finish can affect frictional forces and thus process efficiency. This was reported by Kang et al [80] in a study on can manufacturing dies. Additionally, surface finish may also have an effect on the arising wear mechanisms. Menezes et al [81] explored the effect of surface finish in the aluminium-steel contact in the laboratory and concluded that sliding parallel or perpendicular to the lay resulted in different friction coefficient, as well as a transition from chemical-based adhesion to ploughing-based wear mechanisms. Similar results were also observed by Heinrichs and Jacobsson [82].

1.4.2 Nitriding and coating as wear-reducing strategies

Surface engineering technologies are a wide range of techniques which can be used to modify the properties of a material without affecting its bulk. Some common procedures on forming tools include nitriding treatments or application of hard coatings.

There is evidence in the open literature that surface engineering techniques can improve tool life in applications such as hot forging [44,45,83,84], or High Pressure Die Casting [85,86]. According to some authors, surface engineering (particularly nitriding and PVD coatings) could also potentially offer solutions to the problems of wear in press hardening.

Hardell et al. [18] and Pelcastre et al. [40] observed that nitrided and post-oxidised tool steel samples suffered less adhesive wear than untreated tool steel, in a series of tests using a pin on flat laboratory setup. However, Pelcastre [40] also observed increased galling when the tool specimens had been treated with AlCrN and TiAlN PVD coatings.

On the other hand, Kondratiuk and Kuhn [16] observed contradictory behaviour of a commercial AlCrN coating, resulting in increased or decreased wear depending on forming temperature; trials had been performed on a semi-industrial installation. Sobiek et al. [79] introduced a new coating concept, designed to minimize wear in press hardening tools. Tests on this coating concept were run using a complex cup deep drawing setup, with clearly positive results.

Due to the contradictory observations found in the literature, the applicability of coatings on press hardening tools remains open for discussion. Moreover, further understanding on the mechanisms governing wear could lead to the development of surface modification techniques tailored for the application. Any of these developments will need to be verified in laboratory conditions, without being tested in an industrial trial.

1.4.3 High temperature mechanical characterization of engineered surfaces

One important limitation in the development and selection of coatings for hot metal forming applications is the poor availability of data for comparison of performance in working conditions. The standard technique for characterization of PVD and CVD coatings is scratch testing [87], which offers information about room temperature performance. However, some authors [88–90] have demonstrated that different coatings can be affected differently by temperature, meaning that rankings established at room temperature may not be valid at temperatures as low as 350 ºC.

A second relevant parameter in high temperature performance of coated systems is load bearing capacity provided by the substrate. Indeed, several works [91–93] have shown that improving substrate load bearing capacity, by nitriding the substrates before coating deposition, results in higher coating performance at room temperature. As steel hardness decreases at high temperature, it is expectable that the performance of a coated system will be affected by temperature effects on its substrate.

Therefore, the development of tool-coating systems for high temperature applications would benefit from a characterization technique able to evaluate mechanical response at temperatures comparable to the final application.

Chapter 2

Motivation and Scope

2.1 Objectives of this Research

The main aims of this research are the following:

Developing and evaluating a non-destructive technique for the analysis of wear

One of the main aims of this thesis was to develop a non-destructive technique that could be realistically applied to the inspection of industrial tooling. The chosen methodology is based in surface replication, a technique described in section 1.2 of the Introduction. This work was developed in the first period of the thesis, and summarized in Paper A.

Characterising the wear mechanisms in industrial tools

As discussed in section 1.2, characterization of the wear mechanisms actually taking place is crucial to the optimization of a process. Using the developed non-destructive technique, the main wear mechanisms appearing on press hardening tools had to be identified and understood. This work was performed in parallel to the development of the analysis technique. Results are summarized in Paper A.

Developing laboratory tests to comprehend the fundamental micromechanisms and evaluate their most relevant factors

Analysis of the tools allowed identifying wear and damage features appearing on the tooling. However, full comprehension of the appearing micromechanisms, as well as their implications in terms of materials science, required analysis through laboratory tests.

The development of these tests was based on the previous state of the art about the laboratory study of tribological systems, described in section 1.3 of the introduction. Implementation of the test setups and investigations conducted using them are shown in papers B and C.

Evaluating the applicability of surface engineering as a wear-reducing strategy in press hardening

As discussed in section 1.4, surface engineering, and application of coatings in particular, is an interesting method of improving the tribological performance of hot forming tools. Its applicability to press hardening needed to be studied in the laboratory (Papers C and D) and in the industrial application (Paper A).

Developing a methodology for the high temperature mechanical characterization of coated tool steel

The most common characterization techniques applied on coating-tool steel systems do not offer data relative to high temperature behaviour. In this thesis, a characterization technique based on conventional scratch test has been developed. This technique, presented in Paper D, is simple yet robust and can be used to compare the mechanical response of coated systems under the effect of temperature.

2.2 Scope of this Research

Laboratory study of the press hardening tribosystem

As discussed in section 1.3.2 of the Introduction, a number of tribosimulators for press hardening have already been described in the scientific literature. All these setups present positive points, but also limitations.

The approach of this thesis has been to inspect wear in industrial tools and study the implications of the acting micromechanisms using simplified laboratory setups, as opposed to designing a new tribosimulator setup.

Characterization of coated boron steel

While the industry consensus is that Zinc-based coatings offer interesting properties, as of November 2015 these materials are not being applied in direct press hardening. There is no standard coating composition, and developments are kept as confidential by the steel producers and OEMs.

For this reason, studies have been focussed on AlSi-coated boron steel, the most common option in the industry, while limited studies have also been performed on tools forming non-coated boron steel. However, the information gained about wear micromechanisms can be extended to other tribological system, once this can be validated through investigation of industrial tools.

Study of solutions for the observed wear phenomena

Work in this thesis has involved evaluating solutions proposed in the industry. However, the design of new solutions to be implemented in the future requires very specific knowledge (e.g., in the design of new PVD coatings or surface treatments). The design of these new alternatives falls out of the scope of this thesis.

Chapter 3

Experimental Methodology

The experimental materials and methodologies employed in this thesis are described in detail in the appended articles. A summary is presented in the following sections.

3.1 Materials

The range of materials studied in this work includes sheet metal and tool steels subject to various surface treatments. Aluminium alloys were also used as a counterpart in tribological tests.

3.1.1 Sheet metal

Studies in this thesis have been limited to the most commonly used material in the industry: Al-Si coated boron steel as distributed by Arcelor under the trademark USIBOR. The commercial grade USIBOR 1500 P has the chemical composition described in Table 3.1.

Table 3.1: Chemical composition of the USIBOR 1500P sheet steel grade [5].

Element	C	Si	Mn	Cr	Al-Ti	B
Weight %	0.2-0.25	0.15-0.35	1.1-1.4	0.15-0.30	0.02-0.06	0.002-0.004

3.1.2 Tool steels

At the moment of beginning this investigation, the material most commonly used for the manufacturing of press hardening tools was tool steel DIN 1.2344 [77], corresponding approximately to ASTM grade H13 and Japanese standard grade SDK63. In the latest years, hot work tool steel 1.2367 has also become widespread in press hardening applications, with some authors using it as reference tool steel [8]. At the same time, High Thermal Conductivity Steels (HTCS), a family of steels with improved thermal conductivity developed by Rovalma S.A., have also gained presence in press hardening [94].

Tool steel DIN 1.2344 was used in laboratory tests in Paper B and Paper C, and as a substrate in Paper D. DIN 1.2367 and HTCS 130 have been used in laboratory tests in Paper C. Chemical composition of tool steels 1.2344 and 1.2367 is shown in Table 3.2. Chemical composition of HTCS 130 is not included here, as this is a proprietary material developed by Rovalma S.A.

Table 3.2: Chemical composition of tool steels used in this thesis. Values in weight %.

Tool Steel	C	Cr	Mo	V
DIN 1.2344	0.40	5.30	1.40	1.00
DIN 1.2367	0.38	5.00	3.00	0.50

Tool steel surface finish

Tool steel samples were investigated in different surface finish conditions in Paper B and Paper C, as summarized in Table 3.3.

Table 3.3: Tool Steel surface finish conditions studied.

Tool Steel	Finish	Notes	Ra	Article
DIN 1.2344	Ground and polished	-	<0.1 µm	Paper B
DIN 1.2344	Grit 60 sandpaper	Random texture	0.35 µm	Paper B
DIN 1.2344	Grit 60 sandpaper	Unidirectionally ground	0.4 µm	Paper B
DIN 1.2367	Ground and polished	-	<0.1 µm	Paper C
DIN 1.2367	Grit 80 sandpaper	Random texture	0.8 µm	Paper C
DIN 1.2367	Milled	Patterned surface	2.8 µm	Paper C

Tool Steel surface treatments

Table 3.4 offers a summary of the surface treatments studied in this thesis.

Industrial tools manufactured in an undisclosed hot work tool steel were studied in Paper A. One set of tools was left uncoated to be used as a reference. Surface treatments were applied on a second set of tools: the die was coated with a commercial PVD AlCrN coating, and the punch was nitrided and coated with a thick CrN PVD coating. Achieved coating thickness could not be measured, as it required destructive analysis. Instead, Table 3.4 presents target coating thickness according to the supplier.

Three different surface treatments were studied in Paper C. All samples were polished to an average roughness Ra <0.1 µm before treatment. One sample was subject to gas nitriding. Afterwards, it was re-polished to eliminate the white layer and roughness generated during treatment. A commercial AlCrN PVD coating was applied on a second sample. One last sample was sprayed with a commercial graphite-based hot forming lubricant, resulting in a homogeneous layer of solid lubricant, approximately 15 µm thick.

In Paper D, three tool steel substrates were ground and polished. One of the substrates was left non-nitrided. A second sample was subjected to a short nitriding process, and re-polished afterwards to eliminate white layer. A third sample was subjected to a long nitriding treatment; afterwards, the sample was polished to eliminate roughness, but the white layer was not completely eliminated. Commercial AlCrN PVD coating was then applied on the three samples.

Table 3.4: Summary of surface treatments investigated in this work.

Substrate	Surface treatment	Thickness	Article
Tool steel	AlCrN PVD Coating	3-4 μm (target)	Paper A
Tool steel	Plasma Nitriding CrN PVD Coating	Not measured 12-13 μm (target)	Paper A
DIN 1.2367	Gas Nitriding	<100 μm Diffusion layer	Paper C
DIN 1.2367	AlCrN PVD Coating	3 ±0.2 μm	Paper C
DIN 1.2367	Graphite Lubricant Layer	15 μm	Paper C
DIN 1.2344	AlCrN PVD Coating	3±0.2 μm	Paper D
DIN 1.2344	Gas Nitriding AlCrN PVD Coating	82±2 μm Diffusion layer 3 ±0.2 μm Coating	Paper D
DIN 1.2344	Gas Nitriding AlCrN Coating	274±1 μm Diffusion layer 15±0.1 μm White layer 3±0.2 μm Coating	Paper D

3.1.3 Wear Counterparts

Aluminium alloy balls were used as counterparts in Paper B and Paper C. Their main characteristics are indicated in Table 3.5.

Table 3.5: Aluminium alloy balls used as counterparts.

Alloy	Diameter	Hardness	Article
AA2017	9.5 mm	167 ±3 HV1	Paper B
99 % Al	4 mm	51 ±1 HV1	Paper C

3.2 Analysis Techniques

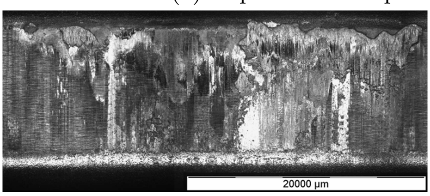
3.2.1 Surface replication

Surface replication has been proposed as a non-destructive technique allowing analysis of industrial forming tools without taking them out of production. Using surface replication, the morphology of a die surface can be transferred to a polymer replica with high topographical accuracy which is flexible and stable. This allows surface analysis of forming tools while removing the complications associated to either taking the industrial tool to the lab, or taking dedicated analysis equipment to the industrial environment. Additionally, surface replicas are stable, and can be stored for future reference.

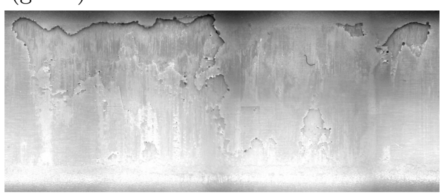
Replica material used in this thesis is a Vinyl Polysiloxane impression material (VPS). VPS resins are commonly used in dentistry to obtain moulds from the buccal cavity, to be used in the design of implants [95]. The composition used is supplied as a two-component resin applied using a manual dispensing gun. The two compounds are mixed during application in a static mixing nozzle. As applied, the compound has the properties of a highly viscous fluid. After a setting time of 2-5 minutes, it cures into a flexible rubber compound. This commercial mixture offers a resolution better than 0.1 µm. Study of replicas in the laboratory was done mainly through confocal microscopy.

In this thesis, surface replication has been applied to the study of wear mechanisms of press hardening tools. Paper A presents a study of the applicability of this technique, which was further elaborated in subsequent work [96]. An example of surface replication is presented in Figure 3.1.

(a) Replication compound (green) on a steel insert



(b) Overview of the insert surface (10x Stereo Microscopy)



(c) Overview of the surface replica (10x Stereo Microscopy)

Figure 3.1: Example of surface replication. Note that surface features are mirrored in the replica.

3.2.2 Optical profilometry / Confocal microscopy

Optical profilometry comprises a family of techniques which can be used to obtain topographical measurements from a surface using optical means. The main advantage of this technique is that it does not require contact with the measured samples, making it possible to study samples which are brittle, soft or dirty.

In this thesis, a SensoFar Plµ2300 confocal microscope was used for topography measurements. This equipment can acquire images at 100x, 200x, 500x and 1500x magnifications, and incorporates a stitching feature in which several images corresponding to neighbouring fields can be combined into a single topography spanning larger area.

Confocal microscopy was used to evaluate the precision of the replicas in Paper A, by taking measurements of the same surface features on a sample surface and in the corresponding replica, and for the analysis of replicas obtained from various industrial press hardening tools. This technique was also applied in the tribological tests performed in, Paper B, Paper C and Paper D. In this case, topographical images were used for quantitative measurement and qualitative evaluation of the generated wear tracks.

3.3 Test Methodologies

In this thesis, different laboratory tests were developed.

3.3.1 High temperature reciprocating sliding test

These tests were performed on an Optimol SRV high temperature reciprocating friction and wear test machine. In this equipment, the upper specimen (aluminium ball) is mounted in a holder attached to an oscillating electro-mechanical drive, which is pressed against a stationary lower specimen (tool steel disc) mounted on a heating block. Computerized control ensures that normal load is kept constant along the test, and Coefficient of Friction (COF) is measured online. The main test parameters are summarized in table 3.6.

Table 3.6: Parameters used in the high temperature reciprocating sliding tests (Paper B).

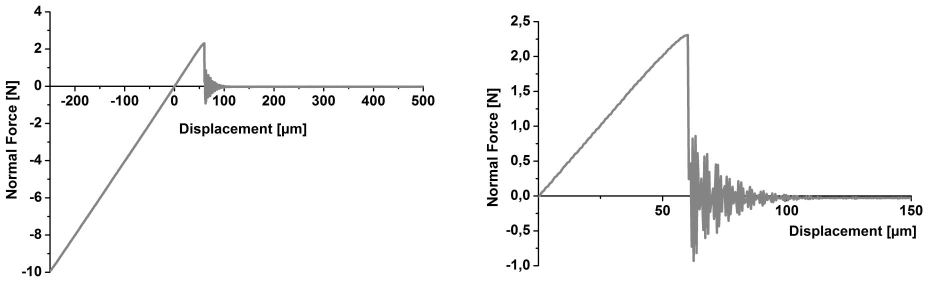
Counterpart	AA2017 9,5 mm ball
Load	20 N
Frequency	25 Hz
Stroke	2 mm
Temperature	30, 150, 250, 350, 450 ºC
Test duration	10, 300 s

Reciprocating sliding tests on a ball-on-disc configuration were used in Paper B to determine the wear mechanisms appearing in the Aluminium-tool steel system, and how these mechanisms were affected by temperature.

3.3.2 Adhesion test

The adhesion test setup developed in this thesis was designed to isolate the contribution of adhesive forces in the overall tribological interaction between two sliding counterparts.

Tests were performed using a CETR/Bruker UMT-2 multifunction tribometer in a ball on disc configuration, with relative movement perpendicular to the flat surface. A 4 mm diameter 99 % Aluminium ball was pressed against different tool materials at a temperature of 450 ºC, normal load of 10 N was maintained during 5 seconds. Afterwards, the ball specimen was vertically retracted at 50 µm/s velocity. This resulted in a linear discharge curve (corresponding to the spring mounting of the sample holder) until 0 N normal load; afterwards, a tensile force peak was generated, related to the force required to break the adhesive junction formed between the two surfaces (Figure 3.2).



(a) Complete curve obtained for steel 1.2367 (b) Detail of the tensile peak

Figure 3.2: Main parameters in the Adhesion Tests (Paper C).

Table 3.7: Main parameters in the Adhesion Tests (Paper C).

Counterpart	99% Aluminium 4 mm ball
Contact force	10 N
Holding time	5 s
Retracting velocity	50 µm/s
Temperature	450 ºC

This test setup was applied in Paper C to test the adhesive forces generated between pure aluminium and different tool materials without the influence of mechanical interaction. Test parameters are summarized in Table 3.7.

3.3.3 Unidirectional sliding tests

Unidirectional Sliding tests were performed using a CETR/Bruker UMT-2 tribometer in a conventional ball on disc configuration, where a rotating tool steel disc slid against a 99 % Aluminium ball under 3 N constant load at a temperature of 450 ºC. Tests were performed for two different durations: 50 and 500 cycles, in order to determine the evolution of transfer mechanisms as material transfer takes place. Main test parameters are summarised in Table 3.8.

Unidirectional sliding tests were used to evaluate the contribution of wear mechanisms based in mechanical interaction to overall material transfer in Paper C.

Table 3.8: Main parameters in Unidirectional Sliding tests (Paper C).

Counterpart	99% Aluminium 4 mm ball
Load	3 N
Sliding velocity	50 mm/s
Temperature	450 ºC
Test duration	50, 500 cycles

3.3.4 High temperature scratch test

As discussed in section 1.4.2 of the Introduction, one of the main limitations of standardised scratch test of hard coatings is that it does not offer information relative to high temperature behaviour. The high temperature scratch test employed in this thesis was developed to avoid this limitation, while maintaining a simple, robust test methodology.

High temperature scratch tests were performed in a CETR/Bruker UMT-2 multifunction tribometer, equipped with a high temperature chamber. Test setup was based in a modification of the scratch test [87], where the commonly used Rockwell C indenter was replaced by disposable carbide prismatic blades with nominal 400 µm radius and a 6º angle between the front and lateral faces. For each test condition three scratches were performed, and the blade tip was discarded to avoid inaccuracies due to wear or oxidation.

High temperature scratch tests were applied to the high temperature characterization of PVD AlCrN-coated tool steel with different surface treatments (Paper D). The main test parameters are summarized in Table 3.9.

Table 3.9: Main parameters used in the high temperature scratch tests (Paper D).

Indenter	CETR micro-scratch blade
Load	L_0=1 N
	dL/dx=10 N/mm
Sliding Velocity	10 mm/min
Temperature	30, 300, 500 ºC
Measurements	Coefficient of Friction
	Confocal Microscopy

3.4 Summary of Materials and Test Conditions

For better convenience, Table 3.10 has been included as a reference guide, summarizing which of the tool systems described in section 3.1.2 have been studied using the different techniques in 3.3, and in which paper can the corresponding results be found.

Table 3.10: Summary of investigations performed on tool materials.

Tool Steel	Surface condition	Test	Article
Undisclosed	-	Industrial tool	Paper A
Undisclosed	PVD AlCrN	Industrial tool	Paper A
Undisclosed	Nitrided+PVD CrN	Industrial tool	Paper A
DIN 1.2344	Ground and polished	Reciprocating sliding	Paper B
DIN 1.2344	Ground and polished	Adhesion	Paper C
DIN 1.2344	Ra 0.35 µm, Random	Reciprocating sliding	Paper B
DIN 1.2344	Ra 0.4 µm, Oriented	Recicprocating sliding	Paper B
DIN 1.2344	PVD AlCrN	High Temp. Scratch	Paper D
DIN 1.2344	Nitrided + PVD AlCrN	High Temp. Scratch	Paper D
DIN 1.2344	Nitrided (white layer) PVD AlCrN	High Temp. Scratch	Paper D
DIN 1.2367	Ground and polished	Adhesion	Paper C
DIN 1.2367	Graphite layer	Adhesion	Paper C
DIN 1.2367	Nitrided	Adhesion	Paper C
DIN 1.2367	AlCrN PVD Coating	Unidirectional sliding	Paper C
DIN 1.2367	Ra 0.8 µm, Random	Unidirectional sliding	Paper C
DIN 1.2367	Milled, Ra 2.8 µm	Unidirectional sliding	Paper C
HTCS130	Ground and polished	Adhesion	Paper C

Chapter 4

Main Results

This chapter contains a summary of the most relevant results obtained in the appended articles and the research leading to them. In order to give an integral view of the work, results in this chapter have been grouped by topic, and not by publication; reference to source publication is also provided.

4.1 Non-Destructive Wear Measurement Technique

One of the aims of Paper A was to verify the applicability of surface replication to the non-destructive measurement of wear in industrial tooling.

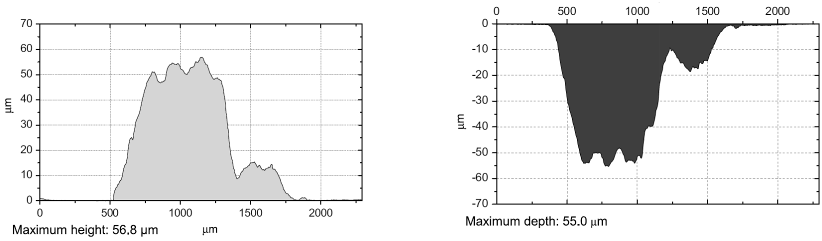
Analyses were performed on laboratory samples showing wear tracks generated by sliding against aluminium [69]. Wear track morphology of these samples presented a combination of adhesive and abrasive wear, comparable in scale to the wear morphology expected in industrial press hardening tools.

Replicas were obtained from these laboratory samples. Afterwards, wear tracks were measured through direct measurement of the sample surface (Figure 4.1 a) and measurement of the same sections on the replicas (Figure 4.1 b). For each studied zone, an area of approximately 2 mm^2 was acquired and the mean transversal profile of the wear track calculated.

Table 4.1 summarizes the obtained results, including area and volume measurements performed by direct sample analysis and the deviation obtained using replica measurements.

Results show good correspondence, with mean deviation close to 2 % and maximum deviations of nearly 3 %. These results are in good correspondence with those of Cabanettes et al. [53], reporting 5 % accuracy in replica measurements.

Further verification of the precision and robustness of surface replicas was performed in this thesis [96], in studies performed on inserts worn in press hardening tribosimulators. Results showed again low discrepancy (in this case, under 10%) between direct measurement on the tool surface and measurements on surface replicas. Moreover, it was seen that both techniques showed good correspondence with thickness of adhered material verified through cross-sectional analysis of the studied samples.



(a) Sample surface measurement (b) Replica measurement

Figure 4.1: Confocal microscopy measurements of a same wear track section, performed on the experimental sample and on the replica: Mean 2D section profile.

Table 4.1: Wear tracks measured directly on the sample and difference when measured from a replica (measurements performed in 6 points).

Point	Max. thickness [μm]	Thickness [% diff.]	Volume [$10^{-7} \mu m^3$]	Volume [% diff.]
1	56.8	3.17	3.61	-0.92
2	94.1	-2.34	4.36	1.93
3	50.8	2.17	2.58	2.30
4	37.9	-2.64	3.01	2.85
5	59.1	-0.17	2.30	-2.00
6	87.8	0.68	3.56	2.35
Mean difference		1.86		2.06

It can be concluded that topography analysis can indeed be applied to the measurement of wear in press hardening tools. Moreover, indirect topography analysis using surface replication offers minimal loss of precision when compared to direct measurements.

4.2 Measurement of Wear on Industrial Tools

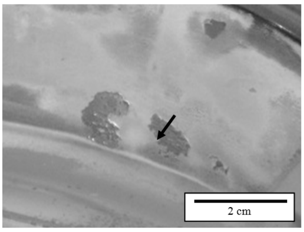
4.2.1 Description of wear on press hardening tools

Tools used in press hardening of AlSi coated boron steel mainly showed adhesive wear-related phenomena, namely transfer of material from the workpiece to the tool.

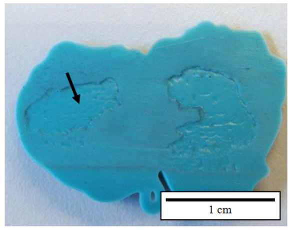
During the first production cycles (0 to 2000 cycles), tool surfaces developed a mat lustre. Topographical investigation using replicas revealed that these surfaces presented a combination of mild abrasion with irregular adhesion of material in thin layers (<5 µm).

In areas with intense tool-component interaction (mainly zones with complex tool geometry), adhered material formed macroscopic features on the tool surface which could be recognised by naked eye inspection.

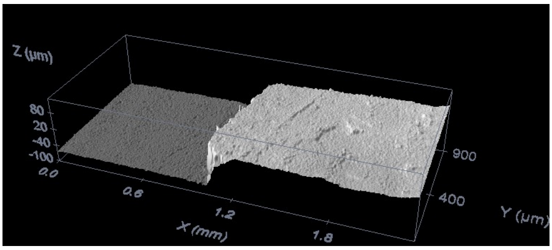
In some cases, these features consisted in lumps of adhered material, up to more than 30 µm thick and more than 2 mm wide after only 2200 production cycles (Figure 4.2). Adhesion lumps presented irregular contours, but a flat, regular top surface. Material transfer was particularly thick in zones with intense tool-component interaction, namely radius geometries. In these zones, accumulated material presented thickness over 80 µm. All these results are consistent with further observations published in [97].



(a) Lumps on the tool



(b) Surface replica



(c) Surface topography reconstruction

Figure 4.2: Material adhesion on an industrial press hardening tool. 3D reconstructions generated from 200x confocal microscopy images.

4.2.2 Analysis of the adhered material

Fragments of adhered material broken off from the tool surface were found entrapped in the replicas. These particles were hard and brittle, with texture, aspect and properties similar to compacted dust.

Semi-quantitative chemical analysis was performed on these particles by means of Energy Dispersive X-ray Spectroscopy incorporated in a Scanning Electron Microscope (SEM/EDS). Results were presented in other work from the author [97]. All particles analysed showed similar chemical composition, including primarily Aluminium, Silicon and Iron and some presence of Manganese (Table 4.2). This chemical composition roughly corresponds to the coating after heat treatment [25, 27, 73].

On the other hand, comparison of measurements in the die (situated as the upper tool in the press) and the punch (lower tool) showed that thickness of adhered material was higher in the punch. This was observed in both uncoated tools and in the coated tool set. It was also possible to observe higher amount of loose wear debris in the punch than in the die.

Table 4.2: EDX analysis of the adhered material; elements in mass %.

Sample	Al	Si	Mn	Fe
1	37.3	11.9	0.6	41.8
2	45.6	6.1	0.6	44.1
3	36	7	0.7	49.1

Based on these results, it is possible to conclude that adhesion in press hardening tools is related to the build-up of material from the coating. Moreover, this material transfer appears to take place in the form of coating wear debris compacted onto the tool by a combination of chemical and mechanical means. The same mechanism has been later elaborated by authors such as Boher [5] and Pelcastre [40]. Due to gravity, loose wear debris falls from the upper tool and accumulates in the bottom tool (in this case the punch). This results in higher overall material transfer for the tool situated at the bottom.

4.3 Characterization of the AlSi Coating

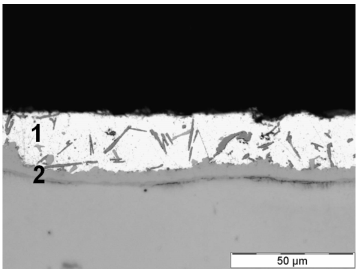
In order to better comprehend wear mechanisms appearing in press hardening of AlSi coated boron steel, it was necessary to characterize the microstructure of the coating and how it evolves during heat treatment. Samples were produced with different austenitisation times and present phases were identified based on the works of Grigorieva [27] and Suehiro [25].

In the as received status (Figure 4.3 a), most of the coating (about 20 µm) consisted in Al-Si solidification structure with τ_6 ternary intermetallic inclusions (layer 1 in Figure 4.3 a). In the interface between coating and substrate, a darker reaction layer approximately 5 µm in thickness could be observed (layer 2 in 4.3 a). This layer has been identified in reference [27] as a ternary intermetallic compound τ_5 formed on a very thin layer of Fe_2Al_5+$FeAl_3$.

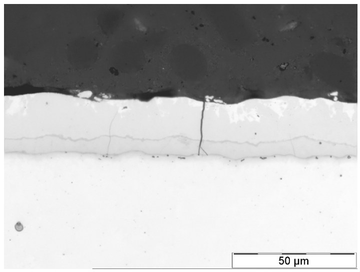
After heat treatment at 900 ºC, samples develop a microstructure consisting on five sub layers. The outermost sub-layer (sub-layer 1 in Figure 4.3 d) corresponds to Fe_2Al_5. Sub-layer 2 corresponds to the Al-Si-Fe ternary phase τ_1, sub-layer 3 corresponds again to Fe_2Al_5, and sub-layer 4 to phase τ_1. Finally, a diffusion layer (sub-layer 5) can be observed between coating and substrate consisting mainly of an iron matrix with Fe_3Al inclusions.

The alternating sub-layer structure was already observed after the reduced heat treatment time of 180 s (Figure 4.3 d). However, samples austenitised by such short time were not fully alloyed and showed remnants of metallic aluminium in the outermost part of the coating.

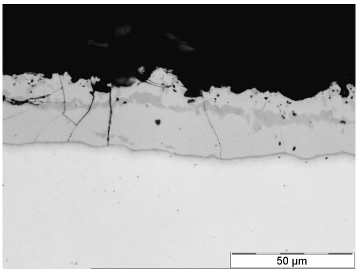
The coating was fully alloyed after 240 s austenitisation (Figure 4.3 c). As treatment time increased from 240 s to 390 s and to 1800 s (Figure 4.3 c through e), sub-layers 2 and 4 (ternary intermetallic τ_1) grew in thickness at the expense of layers 1 and 3 (Fe_2Al_5). Sub-layer 5, the Fe-based diffusion layer, grew both inwards into the substrate and outwards to the expense of the rest of the layers. As a result, total layer thickness increased as shown in Figure 4.4.



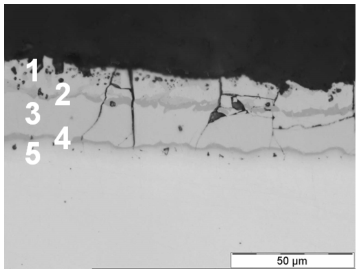
(a) As Received



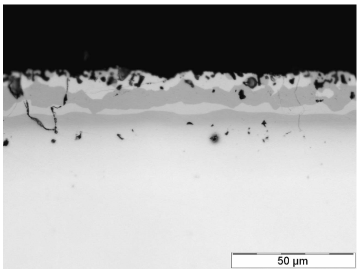
(b) 180 s at 900ºC



(c) 240 s at 900ºC



(d) 360 s at 900ºC



(e) 1800 s at 900ºC

Figure 4.3: Evolution of the AlSi coating during austenitisation at 900 ºC.

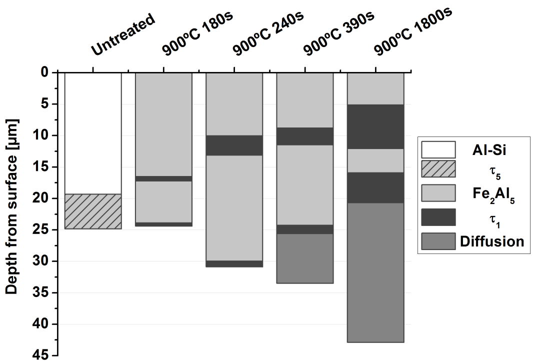


Figure 4.4: Distribution of sub-layers in the layer structure of AlSi-coated 22MnB5.

4.4 Laboratory Study of Wear Micromechanisms

Paper B and Paper C present tribological tests involving interaction of aluminium against different tool steel surfaces. The aim of these studies is identify and comprehend the wear micromechanisms generating the macroscopic wear features observed on industrial tools in Paper A while using a simplified laboratory setup.

4.4.1 The role of temperature

In Paper B, reciprocating sliding tests were performed at different temperatures (30, 150, 250, 350 and 450 ºC), as described in section 3.3. The aim was to observe how system behaviour varied as chemical/adhesive forces and mechanical properties of the counterparts were affected by temperature.

Interaction at low cycle number (10 s) was very similar in tests run at 30, 150 and 250 ºC: in all cases, abrasive wear mechanisms appeared in these first cycles, forming abrasion grooves 2-3 µm deep. Material transfer also took place in the form of large lumps of material and patches of smeared aluminium, as described by Heinrichs and Jacobsson [98]. At 350 and 450 ºC abrasion and adhesion were also identified. Additionally, wear mechanisms based in the generation and accumulation of wear debris were also active.

Greater difference was observed in tests run for 300 s (Figure 4.5). In room temperature tests abrasive wear mechanisms prevailed, generating a deep, wide wear track. Material transfer took place mainly in the form of small accumulations of wear debris, compacted inside the wear scar. At 150-250 ºC abrasion did not progress beyond the grooves generated in the first cycles. Instead, material transfer kept growing, mainly through mechanical ploughing and smearing of thin layers. Finally, at the highest temperatures of 350 ºC and 450 ºC severe abrasion was observed on the centre of the tracks, and accumulations of compacted wear debris formed at the borders. This wear debris consisted mainly in oxidized aluminium particles.

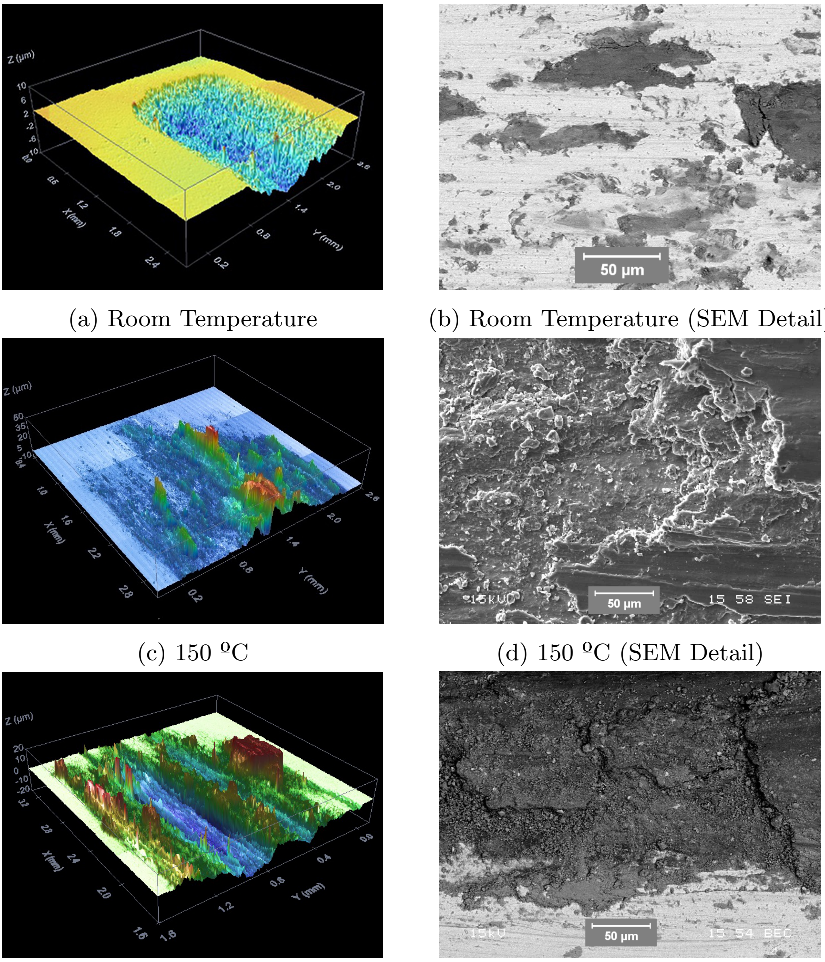
From these tests, it can be observed that different wear mechanisms arise when modifying the chemical affinity of the two surfaces and their relative mechanical properties.

The first contact between the two surfaces results in the transfer of a single, large lump of aluminium, as observed in 10 s tests. This lump generates due to chemical affinity, and has been reported in tools for aluminium processing [98,99].

Increasing temperature tends to increase chemical affinity, resulting in increased adhesive forces. Additionally, softening of the aluminium counterpart resulted in material transfer due to mechanical ploughing by the irregularities on the tool surface. On the other hand, if temperature is high enough, oxidation of the surfaces changes that mechanism into generation and compaction of wear debris similar to the formation of glaze layers [100].

4.4.2 The role of adhesive forces

In Paper C, the adhesion test configuration described in section 3.3 was used to isolate the contribution of chemical forces on adhesive wear and material surfaces.



(a) Room Temperature (b) Room Temperature (SEM Detail)

(c) 150 ºC (d) 150 ºC (SEM Detail)

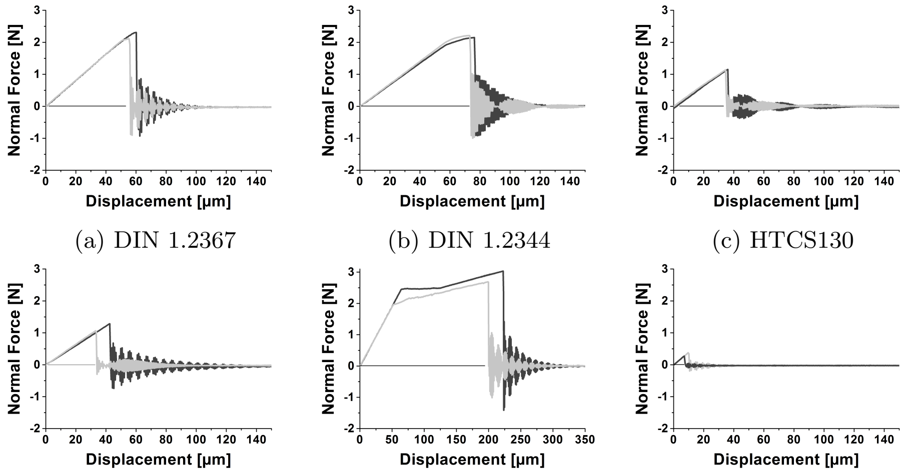
(e) 450 ºC (f) 450 ºC (SEM Detail)

Figure 4.5: Wear tracks obtained after sliding for 300 s at different temperatures. Cross-sectional topography (left column) and SEM 400x detail (right column).

Tests were performed on tool steel samples with different surface treatments (Figure 4.6).

Results were very similar for tool steel 1.2367 (Figure 4.6 a) and 1.2344 (Figure 4.6 b). Adhesion forces measured on High Thermal conductivity steel HTCS130 were lower; this result offers good correlation with laboratory tribological studies of Vilaseca et al. [69], where tool steels from the HTCS brand showed reduced aluminium adhesion compared to tool steel 1.2344.

Nitriding (Figure 4.6 d) appears to reduce adhesion forces. This could be related to the formation of an oxide layer on top of the surface: after heating to the test temperature of 450 ºC, nitrided samples developed a black colouration consistent with post-oxidation. These results can be compared to the work by Pelcastre et



(a) DIN 1.2367 (b) DIN 1.2344 (c) HTCS130

(d) Gas Nitrided 1.2367 (e) 1.2367+AlCrN (f) 1.2367+Graphite

Figure 4.6: Force-displacement curves generated during upper specimen retraction for different surfaces.

al. [19].

On the other hand, adhesive forces increased in the case of AlCrN-coated samples, resulting in higher maximum tensile force and elongation (Figure 4.6 e). Inspection of the tool steel samples after testing showed presence of aluminium, indicating that fracture had happened inside the volume of aluminium and not in the interface.

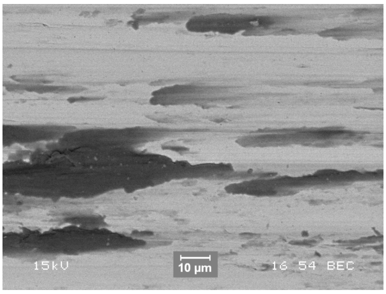
Finally, application of a solid lubricant layer (Figure 4.6 f) greatly reduced adhesion. Measured tensile forces were the lowest of the tests and analysis of the two counterparts after the test showed presence of graphite on both ball and disk. This indicates that the lubricant layer had successfully avoided direct metal to metal contact.

The results of this work show that chemical affinity has a definite effect on material transfer mechanisms. Chemical affinity can be modified by changing the materials in contact or by applying different surface treatments. However, these modifications need to be carefully selected to ensure that they will improve system performance.

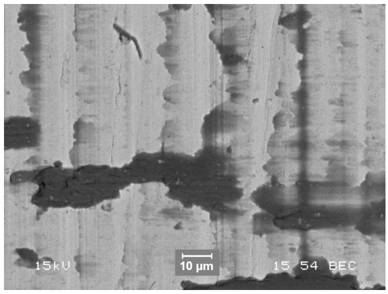
4.4.3 The role of surface finish and mechanical interaction

Studies using reciprocating sliding tests (Paper B)

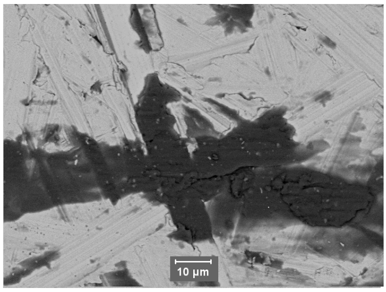
Reciprocating sliding tests were run for a low number of cycles (10 s sliding), in order to evaluate the effect of surface finish on the appearing wear mechanisms. Temperature chosen was 150 ºC, to promote a combination of abrasive wear and transfer mechanisms. Four surface conditions were studied: polished, unidirectionally ground (parallel and perpendicular to the sliding direction) and 8 ground for a random texture.



(a) Lay parallel to sliding



(b) Lay perpendicular to sliding



(c) Random surface

Figure 4.7: Micromechanisms of material transfer generated on samples with their lay oriented differently respect sliding direction; 1000x SEM/BSE wear track details.

All samples showed a central lump of adhered aluminium, representing most of the overall material transfer. However, different micromechanisms were observed through the wear, which were track strongly dependent on surface finish.

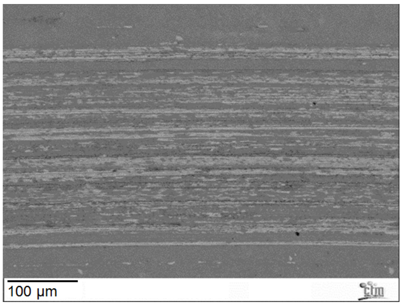
As discussed in section 4.4.1, samples with a polished surface showed complex combination of abrasive wear grooves (2-3 μm deep) with material transfer in the form of smeared layers. Samples with their lay parallel to the sliding direction (Figure 4.7 a) also showed smearing of material on the peaks of the pattern. On the other hand, no abrasive wear was observed: instead, accumulated wear debris was observed inside the valleys of the pattern. This material could have been trapped inside the grooves and compacted during interaction with the aluminium counterpart, as described by Pelcastre in [19].

Wear mechanisms changed completely with surface lay perpendicular to the sliding direction (Figure 4.7 b). In this case, tribological interaction was dominated by ploughing of the tool steel asperities on the softer aluminium ball, as seen by Heinrichs [82]. This resulted in transfer of material on the tips of the asperities and accumulation of material in front of them, until forming continuous layers. Again, no abrasive wear was observed.

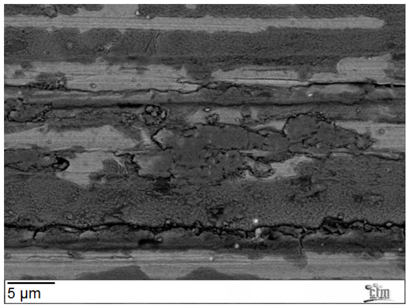
Finally, samples with random texture showed a combination of both mechanisms, depending on the angle of each feature towards the sliding direction (Figure 4.7 c).

Studies using unidirectional sliding tests (Paper C)

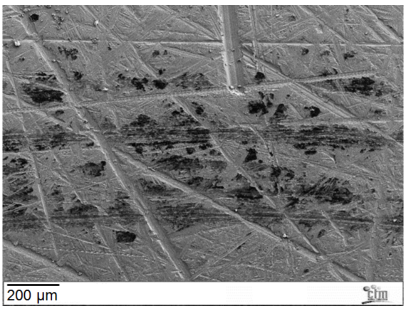
The effect of surface finish on wear mechanisms was further explored in Paper C, using unidirectional sliding pin on disc tests as described in section 3.3. Test parameters were adjusted to promote material transfer mechanisms and to limit degradation of the tool steel surface, ensuring that acting mechanisms could be correctly identified. A softer 99 % aluminium ball was used and temperature increased to 450 ºC. Sliding velocity was also lower, as well as the total amount of cycles (50 cycle short test and 500 cycle long tests compared to a minimum of 500 cycles in Paper B).



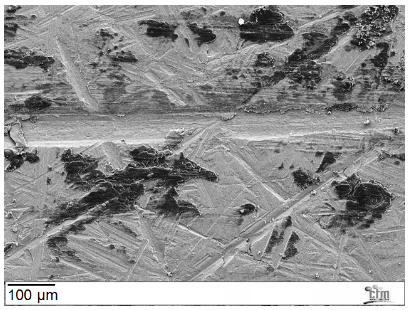
(a) Polished



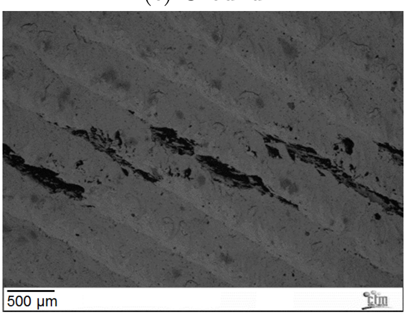
(b) Polished (detail)



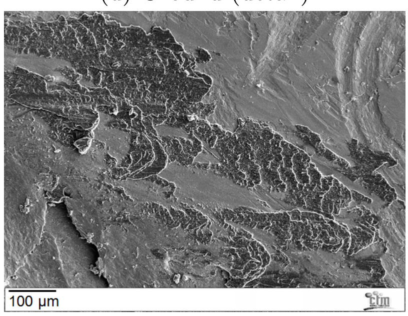
(c) Ground



(d) Ground (detail)



(e) Machined



(f) Machined (detail)

Figure 4.8: Overview (left) and detail (right) of the wear tracks generated after 50 cycles. Darker phases correspond to aluminium.

Results obtained were consistent with those of Paper B. Polished samples (Figure 4.8 a and b) showed abrasive wear in the form of grooves parallel to the sliding direction. Material transfer was observed as layers of aluminium smeared on the surface. Additionally, lumps of material transferred through mechanical means could be identified in the edges of abrasive wear grooves. In 500 cycle tests abrasive wear did not seem to have progressed much further. Instead, large patches of transferred material were observed, mainly generated through mechanical interaction with irregularities on the tool steel surface.

Samples with a random surface experienced a complex combination of wear mechanisms (Figure 4.8 c and d). Material transfer had taken place preferentially on the edges of surface grooves, which generate mechanical ploughing on the aluminium ball (Figure 4.8 d). Material was also found inside deep grooves, consisting of compacted wear debris. Finally, the flattest surface zones showed abrasive wear grooves similar to the polished sample. This suggests that wear debris generates abrasive wear damage, unless there are grooves and valleys on the tool surface where it can be trapped.

Machined samples showed mainly material transfer accumulated at the highest peaks of the pattern (Figure 4.8 e and f) and generated by ploughing. No abrasive wear was observed. After 500 cycles, wear mechanisms appeared to be the same, even though the total amount of material transfer had increased.

From this work, it can be concluded that surface finish does have an influence on the generated wear micromechanisms. It is expected that these mechanisms will gain relevance in surfaces with higher roughness, as discussed in works of Heinrichs [82] and Pelcastre [19].

In the case of polished surfaces, there are no initiation sites where mechanical interaction can take place. Instead, abrasive wear is generated by the loose wear debris until grooves deep enough are formed. In that moment, the newly created surface features will act as initiation sites.

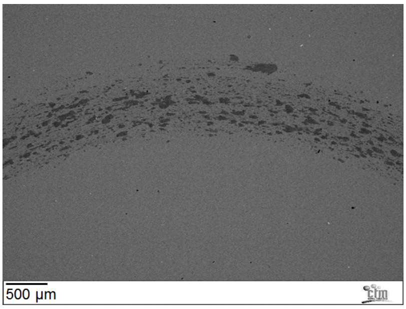
4.5 PVD Coatings on Press Hardening

4.5.1 Effect of PVD coatings on adhesion in laboratory tests

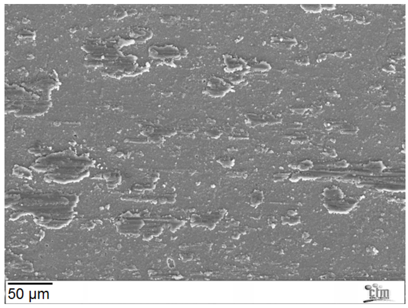
In Paper C, based on the results of adhesion force tests (4.4.2), it was decided to perform unidirectional sliding against aluminium on a sample polished and coated with AlCrN PVD coating. The aim of this test was to understand how the PVD coating affects wear micromechanisms.

The generated wear tracks (Figure 4.9) differed significantly from those on polished and uncoated samples (Figure 4.6 a and d). First of all, no abrasive wear mechanisms were observed. This can be explained by the high hardness of the AlCrN coating. On the other hand, material transfer could be identified as small lumps of adhered material homogeneously distributed along the wear track, due to the increased chemical affinity of this surface towards aluminium (4.4.2).

From this test, it can be concluded that abrasive wear can be prevented by application of hard PVD coatings, which avoid the development of wear grooves. However, these coatings also have increased affinity with the aluminium counterpart, resulting in increased material transfer. Coatings not showing this behaviour



(a) Overview of the wear track



(b) Detail

Figure 4.9: SEM/BSE images of the wear track on a polished PVD-coated sample after 50 sliding cycles; darker phases correspond to aluminium adhesion.

would display improved wear resistance, as shown by Vilaseca et al [69] for a CrN coating sliding against aluminium.

4.5.2 Effect of PVD coatings on adhesion in industrial tools

In Paper A, industrial tools coated with different PVD systems were used in production of press hardened components. These tools were compared to an uncoated set of dies after both sets had performed approximately 2000 production cycles (2200 cycles for PVD-coated tools, 1970 in the case of uncoated tools). Measurement results are presented in Figure 4.10.

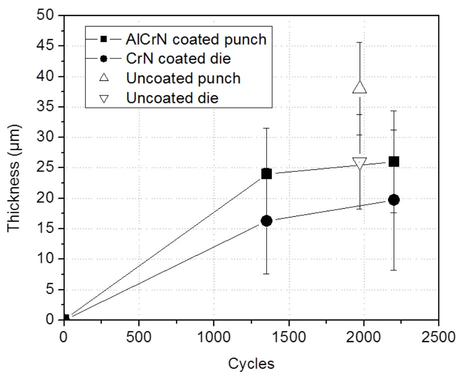


Figure 4.10: Mean adhered material thickness measured on industrial press hardening tools, uncoated and PVD-coated.

Even though PVD-coated tools had performed 230 cycles more, they presented lower overall material transfer. This result could be partially related to better tool alignment or other factors not related to the choice of material. However, the difference is significant enough to merit discussion.

These results can be compared with performance of coatings in the laboratory as discussed in the previous section (4.5.1). In those tests, it was seen that PVD coatings reduce abrasive wear, which results in less nucleation points where material can be transferred by mechanical means. As seen in section 4.2, material transfer in press hardening is, at least partially, mechanical: this component would be reduced by the application of the PVD coating.

On the other hand, it has been established that AlCrN PVD coatings show increased affinity for aluminium, when compared to bare tool steel surfaces. However, the components produced had been subject to 360 s austenitisation. In this condition, the coating should contain no metallic aluminium, only intermetallic phases [27].

Comparison of AlCrN and CrN coatings

Results of trials with industrial tools showed increased adhesion on the AlCrN-coated punch compared to CrN-coated die. However, it is not possible to obtain conclusions from this measurement. In both coated and uncoated systems, it has been observed that the lower tool (punch) suffers increased adhesion related to the accumulation of wear debris due to gravity. Therefore, it cannot be discarded that increased adhesion on the AlCrN punch is due to this phenomenon.

4.5.3 Mechanical characterization of engineered surfaces

In Paper D, the high temperature scratch test described in section 3.3 was used in the characterization of PVD coated systems at temperatures from 30 ºC to 500 ºC. Studied samples had been subject to different nitriding strategies (samples B and C only), and coated with a commercial AlCrN PVD coating (section 3.1.2).

At room temperature, a clear increase in failure load could be observed for the nitrided sample B (13 N) compared to the non-nitrided sample A (8.8 N). Sample C, with a thick composite nitride layer underneath the PVD coating, showed further increase in its performance (25 N). This is consistent with results obtained by other authors [91,93] showing increased delamination load in coatings applied on nitrided substrates.

Tests at high temperature showed a clear trend in reduction of failure load with increasing temperature (Figure 4.11). Nitrided samples B and C show higher failure load than non-nitrided sample A in the whole temperature range. This can be explained by the improved load bearing capacity provided by a harder substrate. In all cases, sample C showed the highest delamination load, with sample B providing intermediate performance between sample C and A.

The observed reduction in performance with increasing temperature can be compared with the work of Fox-Rabinovich et al. [89]. These authors reported increased failure load for AlCrN coatings at 500 ºC. The main difference is that these studies were performed using cemented carbide as a substrate, as opposed to the hot work tool steels used in this thesis. This indicates that substrate thermal softening plays an important role in the performance decrease with temperature observed in Paper D.

In order to confirm this, scratch tracks were analysed by means of confocal microscopy. Figure 4.12 shows images of selected wear tracks at failure load. Additional examples can be found in Paper D.

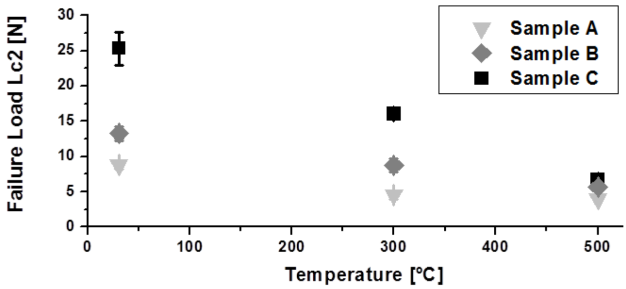
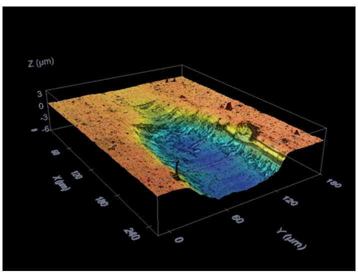


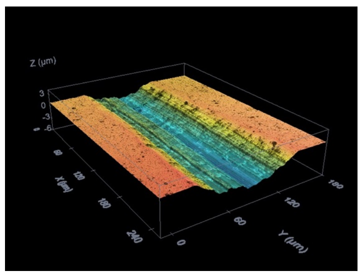
Figure 4.11: Evolution of delamination load Lc2 as a function of temperature for the three studied systems.

For all three systems, failure at room temperature was sudden, with the indenter breaking through the coating and sinking into the substrate as soon as the critical load Lc2 was reached (Figure 4.12 a). The same failure mode was observed for sample B at 300 ºC and at 500 ºC (Figure 4.12 c) and sample C at 500 ºC (Figure 4.12 d), even though these two last cases show some extent of substrate plastic deformation.

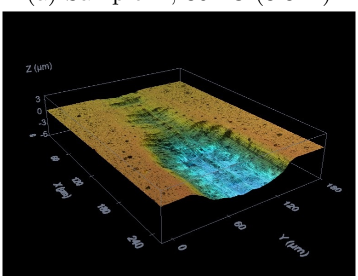
On the other hand, the 300 ºC (not shown) and 500 ºC (Figure 4.12 b) tracks on sample A showed severe plastic deformation of the coated system even before delamination, as well as sample B at 300 ºC (not shown). This clearly indicates that the system had failed due to the plastic deformation of the substrate, which causes coating delamination.



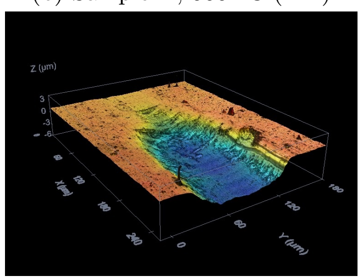
(a) Sample A, 30 ºC (8.8 N)



(b) Sample A, 500 ºC (4 N)



(c) Sample B, 300 ºC (10 N)



(d) Sample C, 500 ºC (6 N)

Figure 4.12: Confocal microscopy images of different scratch tracks at failure load.

Chapter 5

Discussion of Results

In this chapter, the wear micromechanisms responsible for wear in press hardening tools are discussed, based on the results presented in Chapter 4 and in the appended papers. This is done by correlating wear observed in industrial tools (in Paper A, and in other work performed in the context of this thesis [96, 97]) with laboratory observations from Paper B and Paper C.

The chapter has been divided into three sections. The first one discusses micromechanisms responsible for material transfer. The second section deals with micromechanisms of material removal. The third and final section describes the way these mechanisms evolve into the macroscopic features observed on industrial tools.

5.1 Material Transfer Mechanisms

5.1.1 Definition of material transfer micromechanisms

As discussed in the introduction of this thesis (section 1.1.3), it is well-known that the main effect of wear on press hardening tools used with coated sheet metal is transfer of material in the form of macroscopic features.

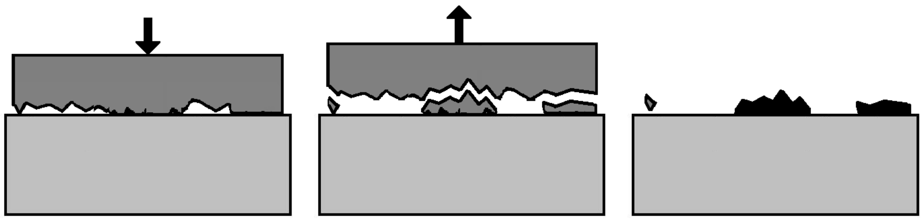
Some references describe this macroscopic phenomenon as galling [40, 75]; however, in the present discussion this use has been avoided. The term galling is usually associated with processes where material transfer is combined with severe plastic deformation and hardening of the transferred material, such as cold drawing of steel [101, 102], or forging [39, 61]. This definition fits with material transfer with intense plastic deformation in press hardening tools processing uncoated material, as described in [97].

However, material transfer in press hardening of coated material shows some differences. Based in the results of the present work, it can be concluded that the formation of compact accumulations of material transfer in press hardening of coated material is not the result of a single mechanism, but a combination of several micromechanisms acting simultaneously. Therefore, the generic description *material transfer* has been used instead of the more specific galling, and features referred to as lumps or compacts.

5.1.2 Chemical/metallurgical adhesion

In this thesis the word "adhesion" has been applied to a mechanism of material transfer involving no significant plastic deformation of the tool.

Whenever two surfaces are put in contact, adhesive forces appear which are of mainly chemical origin. In this thesis, the term "adhesion" has been used to refer to the mechanisms where interaction and material transfer appear between two surfaces when they are put in contact under load, and contact is broken with shearing taking place mainly in the soft counterpart (Figure 5.1).



(a) (b) (c)

Figure 5.1: Mechanism of material adhesion.

Adhesion in laboratory studies

Adhesion has been observed in both Paper B and Paper C, dealing with the interaction between aluminium and tool steel.

In Paper C, adhesion was responsible for the formation of a single lump in the middle of the wear track in tests at elevated temperature, as the two surfaces came into contact (Figure 4.5). In Paper D, a test setup was designed exclusively to study adhesion. Adhesive forces were observed for tool steel surfaces, even when modified by surface engineering techniques (Figure 4.6).

Adhesion in press hardening tools

In some cases, inspection of industrial press hardening tools revealed the presence of small (<5 µm) lumps of transferred material showing approximately rounded shape with no preferred orientation (Figure 5.2). Surface of these lumps did not present the typical polished surface observed in macroscopic accumulations of material transfer, as seen in Paper A and in further work in this thesis [97]. These features were observed in tool zones where no significant amount of sliding took place, resembling the conditions described in Figure 5.1. Anecdotal evidence (interview with tool maintenance teams) indicates that this form of adhesion is particularly difficult to remove from the tool, which is consistent with chemical/metallurgical soldering.

While work in Paper B and Paper C showed that metallic aluminium can indeed adhere on tool steel, there is no evidence for adhesion on steel of the intermetallic phases formed in the coating during austenitisation.

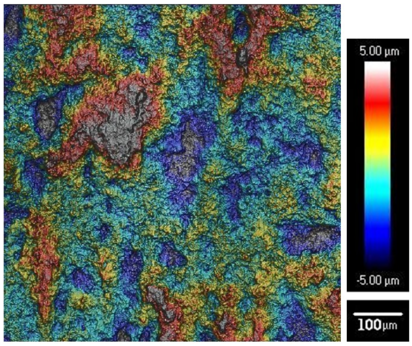
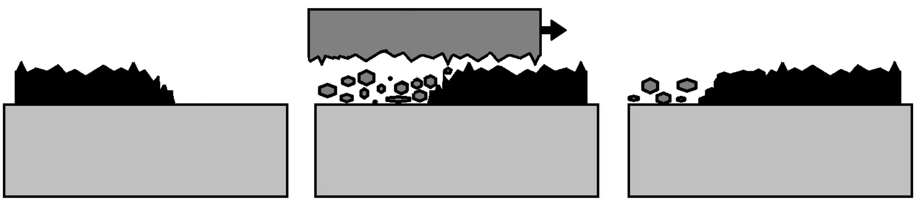


Figure 5.2: Adhesion due to normal loading in tools for press hardening of Al-Si coated B-steel. Confocal microscopy image from a surface replica.

However, there is a trend in industrial press hardening of reducing austenitisation time, often using treatments as short as 180 s [17]. Based in the transformations described by Grigorieva [27] and work in this thesis (Figure 4.3), it is possible that remnants of metallic aluminium appear in the coating for such short austenitisation times (section 4.3). This would result in a tribological system identical to that in Paper C, where adhesion has been confirmed to take place in the laboratory.

5.1.3 Compaction of wear debris

In the mechanism of debris compaction, loose wear debris gets trapped inside the tool-component contact, and is pressed against irregularities in the tool surface. Due to the high temperatures and locally high pressures involved, these particles get compacted into a glaze-like layer (Figure 5.3). This mechanism has also been proposed by authors such as Boher [5] and Pelcastre [40].



(a) (b) (c)

Figure 5.3: Mechanism of compaction of wear debris.

Compaction of wear debris in laboratory studies

Debris compaction was observed in Paper B, particularly in samples tested at 350 ºC and 450 ºC (Figure 4.5). In these test samples, wear debris (mainly oxidized aluminium and iron particles) was compacted into large layers in the edges of the wear tracks. A minor version of this mechanism was also seen in tests at room temperature and 150 ºC and in unidirectional sliding tests in Paper C (Figure 4.8).

This mechanism has also been observed by other authors in laboratory test setups reproducing the press hardening tribosystem [40], as well as in press hardening tribosimulators [5]. Moreover, in additional work done within this thesis [96], it was observed that layers of accumulated material mainly consisted of wear particles broken from the coating and compacted onto the tool surface (Figure 5.4).

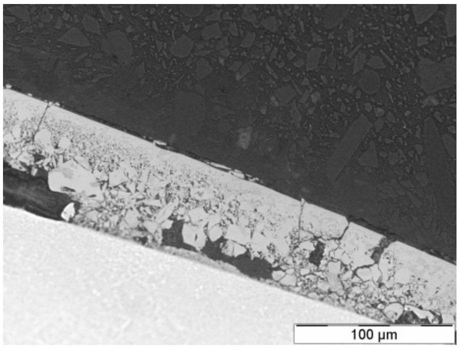


Figure 5.4: Cross-section of a tribosimulator insert. Material transfer consists mainly of compacted wear debris originating from the coating [96].

Compaction of wear debris in industrial press hardening tools

It has been not possible to directly observe this wear mechanism in industrial press hardening tools. This is a limitation imposed by the use of surface replication: loose dust is collected in the replica, altering the morphology of the feature.

However, it is possible to indirectly deduce the action of this mechanism. Paper A showed increased adhesion in the bottom tool of each set. This was related to the higher amount of wear debris present on this tool, implying that dust compaction is indeed an important material transfer mechanism.

This can be further confirmed taking into account the analysis of fragments of adhered material samples, described in section 4.2 and in [97]. These fragments showed mechanical properties similar to compacted dust, with composition consistent with particles broken off from the coating.

Finally, anecdotal evidence (conversation with tool maintenance personnel) is also consistent with this explanation, as it is reported that part of the material transfer easily breaks into small dust particles during tool maintenance.

5.1.4 Ploughing

In the mechanism of ploughing, protrusions on a hard surface interact mechanically with a softer counterpart during sliding, resulting in removal of material from the soft counterpart. Material thus removed can become loose wear debris, or remain adhered on the asperity (Figure 5.5).

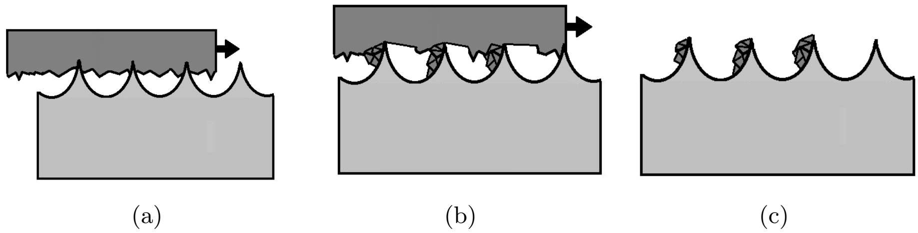


Figure 5.5: Mechanism of material transfer through ploughing.

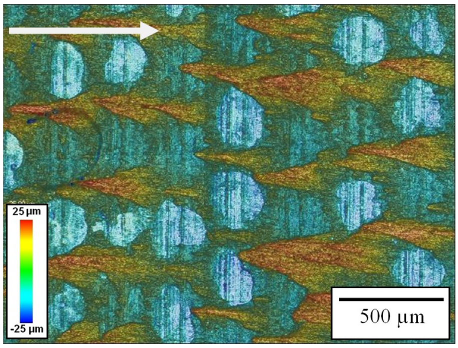


Figure 5.6: Material transfer due to ploughing observed on industrial press hardening tools; confocal microscopy image of surface replicas. Material accumulates on the highest peaks of the surface finish. White arrow indicates sliding direction.

Ploughing in laboratory studies

Ploughing has been observed in Paper B and Paper C, in studies concerning the role of surface finish on material transfer mechanisms (section 4.4.3). In both works, it has been observed that protrusions of the tool steel surfaces, generated during sample production through grinding or machining, resulted in material transfer taking place preferentially through ploughing (Figure 4.7 and Figure 4.8). Similar results have also been reported in the scientific literature [81, 82, 103].

Material transfer due to ploughing in industrial press hardening tools

Industrial press hardening tools with surface finish similar to the machined samples in Paper D and used for press hardening of AlSi coated boron steel were studied in the curse of this thesis by means of surface replication. Replicas obtained at low cycle numbers showed signs of wear consistent with material transfer through ploughing as observed in the lab (Figure 5.6).

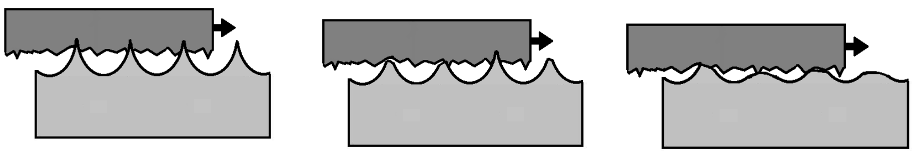
5.2 Material Removal Mechanisms

5.2.1 Abrasive wear

Two body abrasion

The mechanism of two body abrasion is described in detail by Archard et al. [58]. In this mechanism, mechanical interaction of the asperities in each surface causes asperities to be blunted, due to plastic deformation and small volumes of material being fractured and removed.

In a two-body interaction, material removal appears mainly in the softer of the two counterparts, and the speed of this removal is inversely proportional to its hardness relative to each other. According to Archard, abrasion will appear mainly on the softest counterpart (i.e., the component). However, the tool is subject to thousands of production cycles; meaning that material loss will accumulate over the cycles and macroscopic damage will eventually be observed (Figure 5.7).



(a) (b) (c)

Figure 5.7: Two body abrasion. Tool-component contact takes place mainly on the tips of the tool asperities. Due to this interaction, material is removed from the highest asperities as cycles progress.

Third body abrasion

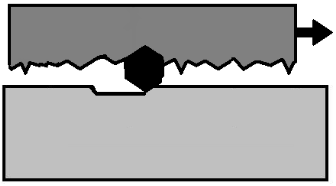
In three body abrasion, wear is generated by a particle trapped inside the contact (Figure 5.8). Depending on the properties of the two counterparts, different scenarios can occur. If one of the two counterparts is clearly softer than the other, the particle may embed in the soft counterpart; the embedded particle will then abrade the harder surface as in two body abrasion. If the two surfaces are hard enough, the particle cannot embed itself: in this case, it will remain as loose wear debris [40], generating mild wear on both surfaces.

Abrasive wear in laboratory studies

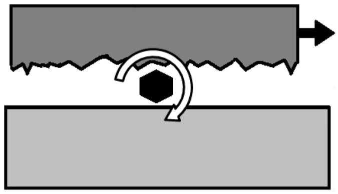
Abrasive wear mechanisms have been observed in both Paper B and Paper C, in studies of material transfer on polished surfaces. In both cases, third body abrasion took place, with oxidized wear debris generating grooves 2-3 µm deep that would later act as material transfer initiation sites.

Abrasive wear in press hardening tools

In the course of this thesis, effects of abrasive wear could be observed in tools used in press hardening of uncoated boron steel [97]. Tool surface finish consisted in



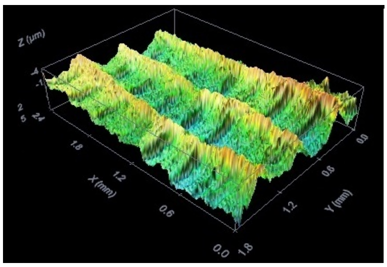
(a) Ploughing by a third body embedded in the softest counterpart



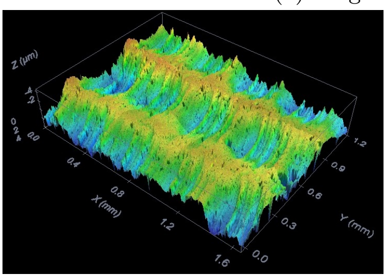
(b) Rolling particle trapped inside the contact

Figure 5.8: Third body abrasion.

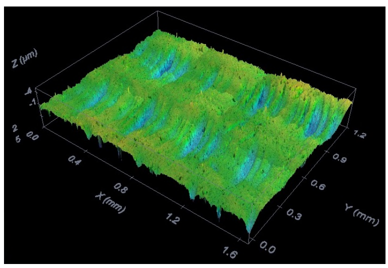
a well-defined machining pattern, consisting in a series of peaks and valleys. As cycles progressed, peaks were flattened by a combination of material removal and plastic deformation (Figure 5.9).



(a) Original surface finish



(b) Initial stage of wear



(c) Advanced stage of wear

Figure 5.9: Abrasive wear on an industrial press hardening tool. Surface finish is worn out as production cycles progress (a to c).

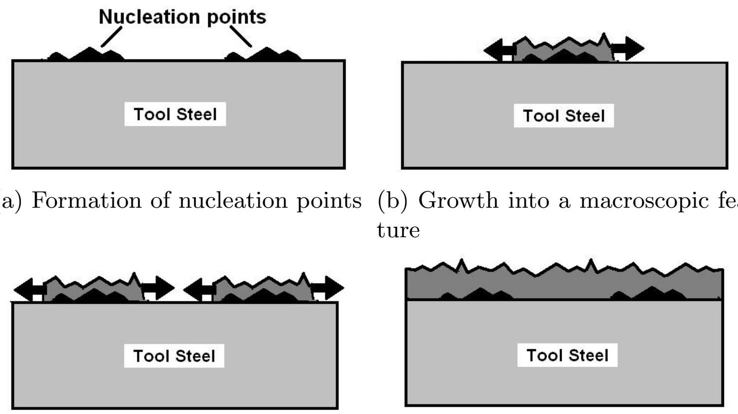
Abrasive wear can be expected to appear in press hardening of coated boron steel. Based on the existing literature [6], intermetallic phases in the AlSi coating are much harder than tool steel. Pelcastre estimated their hardness to be between 511 and 837 HV [104]. It has also been observed that scales from this coating appear on the tool, either as part of compacted layers or as loose wear debris. The latter could act as three body abrasive, just as loose oxidized aluminium particles had in laboratory tests.

However, it was not possible to identify abrasive wear on industrial tools working with coated materials, due to the original tool surface being masked by the irregular transfer layer. Nonetheless, effect of abrasive wear was recognised on the surface of thick accumulations of material, as will be discussed in section 5.3.2.

5.3 Formation of Macroscopic Features

Macroscopic features on press hardening tools working with coated material form through a complex process, which is a competitive combination of material transfer mechanisms (adhesion, dust compaction, ploughing) and material removal.

5.3.1 Material transfer to the tool



(a) Formation of nucleation points (b) Growth into a macroscopic feature

(c) Small features grow into each other (d) A large features is formed

Figure 5.10: Formation of macroscopic features.

Formation of macroscopic features begins with the establishment of nucleation points, formed by material strongly adhered to the tool surface (Figure 5.10 a). These first nucleation points could form through chemical/metallurgical adhesion (as explored in Paper C) or ploughing (in the case of rough surfaces, as seen in Figure 4.7 or Figure 5.6). In the case of smooth surfaces, ploughing sites formed by abrasive wear mechanisms could also act as nucleation points, as discussed in Paper B.

Once nucleation sites have been formed, further material transfer takes place. Both laboratory tests and analysis of industrial tools indicate that this growth takes place mainly through mechanical means: compaction of wear debris onto the existing feature, or through ploughing if the newly formed lump protrudes from the surface (Figure 5.10 b).

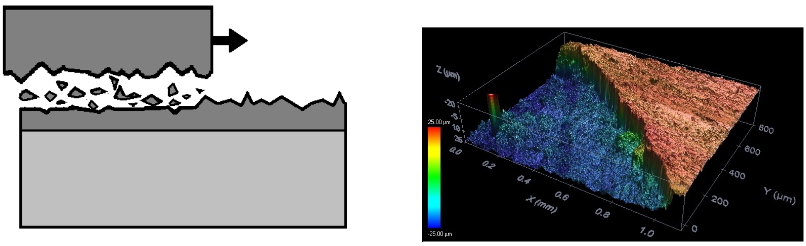
If enough of these lumps form in an area, it is possible that they grow into each other (Figure 5.10 c), as has been observed to happen in reciprocating sliding tests in Paper D (Figure c). Eventually, a continuous layer of transferred material forms (Figure 5.10 d).

5.3.2 Removal of adhered material

Once macroscopic material transfer features are formed, material removal mechanisms appear which slow their growth. This has been observed in Paper A (Figure 4.10), and also in industrial practice. Based on the presented results, two mechanisms are proposed here: *abrasion* and *fracture* of compacts.

Abrasion of compacts

The mechanism of abrasion is homologous to abrasive wear observed on the tools, and discussed in section 5.2: interaction of the newly formed contact with the hard surface of the coating or with hard wear particles results in material removal. Existence of this mechanism can be deduced by the flat, homogeneous surfaces observed in lumps and layers of transferred material in Paper A. In some cases these surfaces show texture oriented with the sliding direction (Figure 5.11 a and b).



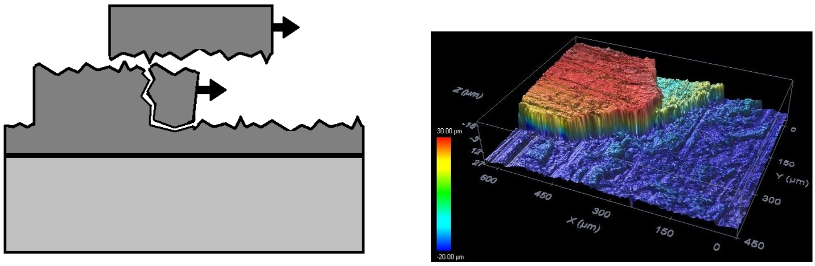
(a) (b)

Figure 5.11: Mechanisms of removal of transferred material: compact abrasion.

Fracture of compacts

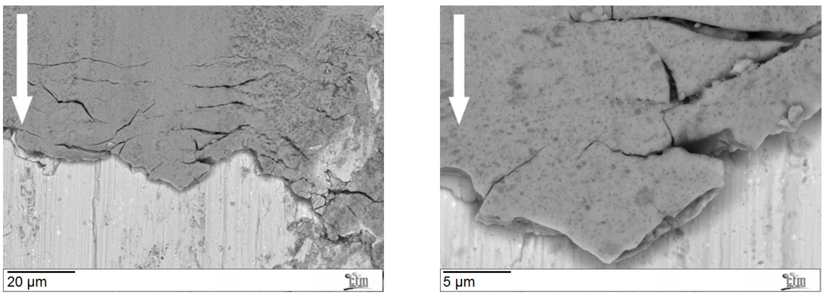
Layers of transferred material are brittle, and will break under shearing stress such as in interaction with the component (Figure 5.12 a). This can be observed on the edges of lumps or adhered layers, which usually have edges with sharp angles and clean-cut faces, (Figure 5.12 b). In some occasions, this fracture affects only part of the adhered thickness. In this case, a “step” feature is created.

Additional evidence of this mechanism has been observed in SEM studies of tribosimulator inserts (Figure 5.13). In these tools, fracture of compact layers could be observed in several points, always downstream in the material sliding direction. Inserts corresponded to a strip drawing simulator [73].



(a) (b)

Figure 5.12: Mechanisms of removal of transferred material: compact fracture.



(a) Overview (b) Detail

Figure 5.13: Fracture of compacts: SEM/BSE images obtained from tribosimulator inserts.

Chapter 6

Conclusions

In this work, wear mechanisms appearing in press hardening tools have been studied both by inspecting actual industrial tools and through laboratory tests. This study has been focussed on press hardening of AlSi coated boron steel sheet, as it is the most prevalent option in the industry. From the obtained results, a proposal has been established for the active wear micromechanisms resulting in wear in press hardening, as well as their interaction with surface finish and surface engineering techniques. The main conclusions which can be drawn from this work are the following:

- Surface replication is a valid methodology for analysing wear in industrial tools. This technique offers considerable advantages, mainly not requiring the tool to be retired from production. Its main limitation is that only topography-based information can be obtained.

- The main wear mechanism appearing in press hardening of AlSi coated boron steel is material transfer. This material transfer is not only related to chemical adhesion, but is also generated by mechanical means. The contribution of the latter is decisive in the growth of macroscopic wear features.

- Compaction of coating wear debris is one of the main micromechanisms resulting in the formation of macroscopic features on industrial tools.

- Tool surface finish affects wear micromechanisms based in mechanical interaction. High roughness and grooves perpendicular to the sliding direction promote material transfer through ploughing mechanisms.

- Even on polished surfaces, it is possible to have mechanical-based material transfer. In this case, preferential adhesion spots will be generated by abrasive wear mechanisms.

- Modification of the chemical composition of the tool surface affects chemical interaction, and therefore the prevalence of adhesive-based wear.

- Application of hard PVD coatings has mixed effects on wear. On the one hand, some coatings may promote adhesion, through increased chemical affinity with the wear counterpart. On the other hand, smooth surfaces are protected from abrasive wear mechanisms by hard coatings.

- Hard PVD coatings on press hardening tools results in a decrease in material transfer, with thinner transfer layers and lumps.
- High temperature mechanical response of hard PVD coatings is highly dependent on substrate properties. Substrate nitriding prior to coating application substantially improves system performance, and would be required in applications where locally high pressures are generated.

Chapter 7

Future Work

Results obtained in this thesis can be used as the foundation of future work with the intent of reducing wear in this same system, or in similar ones. Some possible continuations of this research are the following:

Design and evaluation of wear-preventive solutions for tooling

Once the acting wear micromechanisms have been characterised, solutions that modify the relevant factors can be designed. These can include modification of the topography, chemical composition or mechanical properties of the tools.

These solutions could be evaluated in the laboratory, using the tests employed in this thesis and comparing results to the non-modified system, as a screening before risking the cost and effort of an industrial trial.

Application of wear-reducing strategies in industrial systems

To this point, only limited experience exists with the application of PVD coatings and other wear reducing strategies on industrial tools. Extensive, long-term tests would be required to asses if these solutions offer significant advantage in the industrial practice and, if not, detect their shortcomings and design improvements.

Design and evaluation of alternative sheet metal coatings

The AlSi coating in USIBOR offers a wide range of advantages, but also shortcomings. Moreover, it has been observed that material transfer takes place mainly because of the characteristics of this coating. Careful characterization and testing of alternative materials could help identifying coatings with improved performance in particular applications.

Investigation wear mechanisms in systems different from press hardening

The replication methodology developed in this thesis can also be applied to other tribological systems. This would allow identifying the most relevant wear mechanisms and study them in the laboratory in a manner analogous to the present work.

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Appendix A

Appended Articles

Paper A

M. Vilaseca, J. Pujante, G. Ramírez, D. Casellas. *Adhesive wear analysis of PVD coated and uncoated hot stamping production tools.* Wear 308 (2013) pp. 148-154

ATTENTION ¡

Pages 70 to 78 of the thesis, containing the article ***Investigation into adhesive wear of PVD coated and uncoated hot stamping production tools,*** **http://dx.doi.org/10.1016/j.wear.2013.07.003** are available at the editor's web

http://www.sciencedirect.com/science/article/pii/S004316481300433X

Paper B

J. Pujante, L. Pelcastre, M. Vilaseca, D. Casellas, B. Prakash. *Investigations into wear and galling mechanism of aluminium alloy-tool steel tribopair at different temperatures.* Wear 308 (2013) pp. 193-198
Doi 10.1016/j.wear.2013.06.015

ATTENTION ¡

Pages 80 to 86 of the thesis, containing the article, are available at the editor's web

http://www.sciencedirect.com/science/article/pii/S0043164813004080

Paper C

J. Pujante, M. Vilaseca, D. Casellas, M.D. Riera. *The Role of Adhesive Forces and Mechanical Interaction on Material Transfer in Hot Forming of Aluminium*
Tribology Letters 59 (2015) 1-10
DOI: 10.1007/s11249-015-0542-1

ATTENTION ¡
Pages 88 to 96 of the thesis, containing the article, are available at the editor's web

http://www.sciencedirect.com/science/article/pii/S004316481300433X

Paper D

J Pujante, M. Vilaseca, D. Casellas, M.D. Riera. *High temperature scratch testing of hard PVD coatings deposited on surface treated tool steel.* Surface and Coatings Technology 254 (2014) 352-357
Doi 10.1016/j.surfcoat.2014.06.040

ATTENTION ¡

Pages 98 to 104 of the thesis, containing the article, are available at the editor's web

http://www.sciencedirect.com/science/article/pii/S0257897214005519

Appendix B

Other Appended Works

Paper I

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Wear Mechanism Identification on Hot Stamping Tools

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Abstract

One of the main damaging mechanisms in hot stamping tools is wear. Tool wear-related phenomena negatively affect the contact between tool and workpiece surfaces in hot stamping and consequently the heat transfer coefficient. Identification and understanding of these wear mechanisms acting on tool surface is crucial to predict and prevent them. Nowadays the acting wear mechanisms in tools are not well defined because direct microscopic inspection of industrial tools is difficult. Thus, tribological knowledge is quite limited and inaccurate aimed at designing new high wear resistant coatings and tool materials.

In this work a methodology based on non-destructive techniques of surface topography analysis is developed and applied for in-situ wear mechanisms identification on hot stamping tools for uncoated and Al/Si coated boron steel sheet forming. These results will enable the accurate design of tribological laboratory tests to reproduce the wear mechanisms observed on industrial tools.

1 Introduction

The recent increase in the use of hot stamping in the automobile industry has spurred related research in many fields. One of these fields is tribology, as tool wear is a limiting factor in the efficiency of the process and tool-workpiece interaction has a direct impact on component quality and mechanical properties [1]. Wear mechanisms and tribo-mechanical solicitations acting on the tools are mainly influenced by the surface condition of the boron steel sheet; coated or uncoated.

In hot stamping, the blank or preform is heated in a furnace, transferred to a press and formed and quenched in one only process step. If uncoated sheet steel is used, heating must be done in a protective atmosphere, in order to prevent oxidation and decarburisation of the blank. Even then, during the transfer of the hot, austenitised workpiece from the furnace to the press, iron oxides form on its surface due to the exposition to atmospheric oxygen. These irregularly shaped oxides (scale) have a high hardness and can act as an abrasive during forming [2], generating wear or scratches on the tool surface. As an alternative, sheet steel grades with a coating that prevents exposition of the austenitised metal to the oxidative atmosphere have been developed. The most commonly used is USIBOR 1500P, a 22MnB5 steel sheet with a hot dip Aluminium/Silicon coating developed by ArcelorMittal. In this case, the main wear mechanisms observed appear to be related to the interaction of the tool surface with the Al/Si coating [3].

Many works have been published dealing with the characterisation and understanding of the tribological pair sheet steel-tool in hot stamping. Most of them consisted in the development of laboratory tests with the aim of reproducing the behaviour of the system. Some authors, such as Marzouki et al used simple geometries and aimed to comprehend the fundamental mechanism [4]. Other groups tried to reproduce the industrial system by developing hardware simulators, as in the works of Dessain [5], Geiger [6] and Yanagida [7]. Hardell used both approaches in order to connect data from the fundamental mechanisms to the macroscopic behaviour [3]. All these results can be used to obtain information about the friction coefficient and wear behaviour of the system.

However, to our knowledge, no study has been focussed on characterising the wear mechanisms acting on actual industrial tools, as opposed to simulators. The knowledge gained from such study would be useful to design wear tests and equipments able to better reproduce the conditions in the industrial process, which could be used to test new tool steels and coatings before industrial or semi-industrial testing. Unfortunately, methodologies with the precision required to characterise fundamental wear mechanisms are largely restricted to laboratory scale, requiring specific equipment and long inspection time. Their use on industrial tools would demand complex logistics and severely affect productivity, and thus they appear unattractive from an industrial point of view.

The aim of this work is to identify and characterise the wear mechanisms acting on hot stamping production tools using an experimental methodology with a minimum impact on productivity. This will be accomplished by means of a novel surface replication technique, which will allow using laboratory equipment and methodology for the inspection of tools without removing them from production.

2 *Experimental*

The tools studied in this work were two industrial die sets producing similar components with different sheet steels. The fist tool set was used for the hot stamping of USIBOR 1500P (Al/Si coated boron steel). The second tool set was used for forming uncoated 22MnB5 steel sheet.

A novel application of the replication technique was used to study the wear mechanisms acting on the selected tools. Replication is an established methodology for microstructural characterisation [8] and inspection of corrosion [9]. This technique consists in the application on the surfaces to be inspected of a viscous compound which cures in a few minutes into a high precision thermoset polymer replica of the surface topography. In this work, a commercial two-part silicone-based rubber was used to cast replicas from selected tool spots. The polymer was applied using a dispensing gun with a static mixing nozzle. The tool surface required no specific preparation. Approximately five minutes after application, the cured replicas were carefully lifted off the tool surface and stored in polypropylene bags. Replicas were cast during natural stops of the production line, with no need to schedule additional inspection stops. The methodology used allows studying the industrial tools using precise laboratory equipment, without need to remove the tools from the shop or disturb the production process.

Replicas were inspected in the laboratory through lens imaging. Optical profilometry (using a Plμ 2300 confocal microscope) was used to obtain topographic images from selected spots, which were inverted using topography analysis software generating 3D reconstructions of the tool surface.

In order to identify the material adhered on the tools, samples of this material were obtained and analysed by means of scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX), using a ZEISS Ultra-Plus electron microscope.

3 Results and Discussion

3.1 Wear mechanisms in hot stamping of Al-Si coated boron steel

Naked eye observation of the first tooling set suggested that the main wear mechanisms present in hot stamping of coated boron steel result from adhesive wear-related phenomena, namely transfer of material from the workpiece to the tool surface. The most obvious of such features found on the tool surface was the presence of irregular lumps of adhered material, which were accurately reproduced in the replicas (figure 1). These lumps can be more than 50 µm high and several millimetres across.

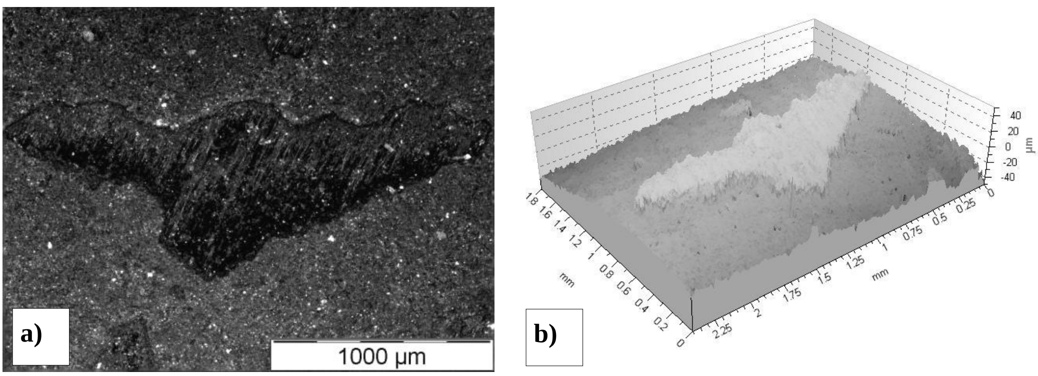

Figure 1: *Material adhered in the shape of irregular lumps on a hot stamping tool: a) 10x lens image and b) 3D reconstruction of tool surface by 200x confocal microscopy imaging.*

Another feature that can be observed is a homogeneous, continuous material layer that forms on certain parts of the die. The surface of this layer is mostly flat and regular, although lumps can also be found. The existence of such layer can only be detected by observing points where the original tool surface is exposed (figure 2). These points are either the borders of the material pileup, as shown in figure 2a, or areas where the adhered material layer breaks, as shown in figure 2c and 2d. Layers of adhered material regularly reach thicknesses in excess of 50 µm and even thicker in die radii, where it can exceed 100 µm, as shown in figure 2b.

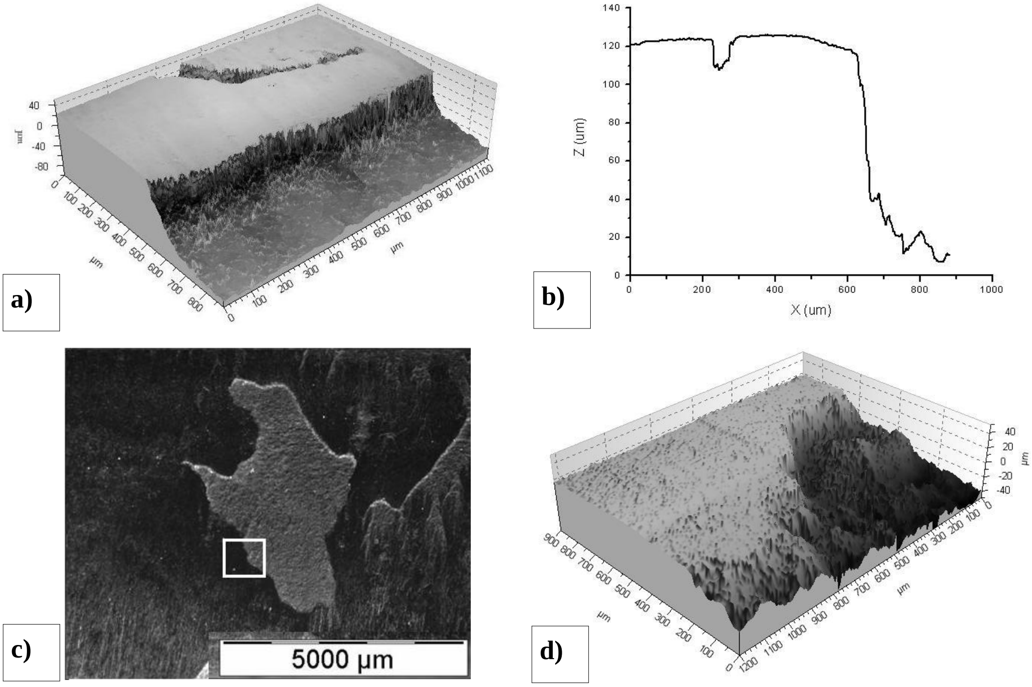

Figure 2: a) 3d tool surface reconstruction by 200x confocal microscopy imaging. The high, flat surface corresponds to the continuous adhered layer; the lower surface is the original tool surface. b) xz height profile extracted from figure 2 a. c) 10x lens image from a die replica showing breakdown of the adhered layer. d) Reconstruction of the region inside the white box in figure 2 c by 200x confocal microscopy.

EDX analysis was carried out on particles extracted from the adhered layer in order to determine the origin of the adhered material. Results of this analysis must be treated as semi-quantitative, and are displayed in table 1. It was found out that the chemical composition of the adhered material matches that of the Al/Si coating after heating.

Table 1: *Data from the EDX analysis of the adhered material. Results show mass contents in %.*

Sample	Al	Si	Mn	Fe
1	37.3	11.9	0.6	41.8
2	45.6	6.1	0.6	44.1
3	36	7	0.7	49.1

From the presented data, it can be deduced that the adhered lumps and layers consist in a buildup of material from the Al/Si coating. The formation mechanism of these features is the mechanical interlocking and solid state welding of asperities from the hard tool steel surface and the hotter, softer sheet metal coating in an adhesive wear process [10]. These interactions cause the removal through shearing of small particles from the weaker coating material, which can be eliminated as wear debris or remain attached on the tool surface. Material transferred to the tool surface is then compressed due to the forming pressure, forming a compact and well adhered layer. The continuous sliding of this surface with new workpieces causes flattening of the asperities and constant material transfer. Irregular particles are flattened and new asperities formed are worn away giving place to the flat and regular finish seen in figure 2a. This process and the breakdown of scales shown in figure 2c are the main material loss mechanisms for the adhered layer.

It is worth noticing that, even though mechanisms of adhered material removal exist, lumps and thick layers of adhered material can be found on most of the tool surface. This indicates that the material transfer mechanism is faster and more active than material removal, resulting in net adhesion growth. Avoiding buildup of adhered material is the main reason for tool maintenance, and characterisation of this mechanism will lead to optimisation of the tool maintenance cycle.

3.2 Wear mechanisms in hot stamping of uncoated 22MnB5 sheet steel

Figure 3 shows a lens image from a replica corresponding to tools for hot stamping of uncoated boron steel. The original tool steel surface can still be identified by the presence of regular machining patterns (milling marks) found in all of the studied tool zones (1). In some zones, these machining patterns can no longer be recognised (2); these areas can be identified as worn. Finally, in some areas (3) shallow but wide scratches could be seen on the tool. Surfaces of tools employed in hot stamping of uncoated 22MnB5 sheet steel showed abrasive wear-related mechanisms.

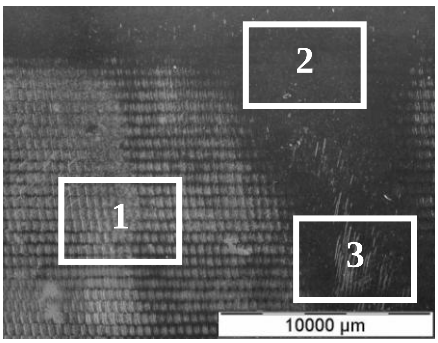

Figure 3: *10x lens images from a tool replica.*

In order to comprehend the microscopic wear mechanism causing the removal of milling marks, replicas were studied through confocal microscopy. 200x topography images were obtained, which allowed the generation of 3D reconstructions of the tool surface. Figure 4 shows three examples of such reconstructions, corresponding to three different stadiums of the wear process.

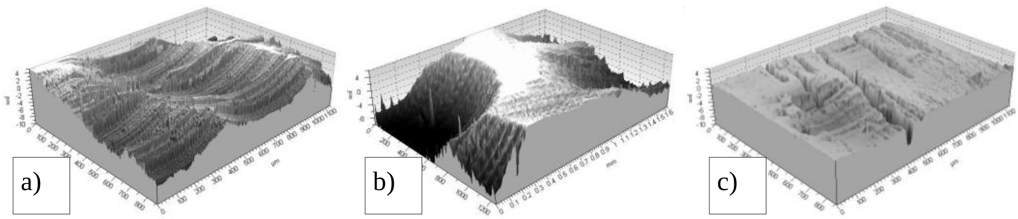

Figure 4: *3D reconstruction of the tool surface. a) original surface finish, b) initial stage of wear and c) worn surface.*

Figure 4a depicts the initial surface finish, which consists in a pattern of peaks and valleys. Figure 4b shows the first stage of wear, consisting in the flattening of the tips of the asperities. Eventually, as seen in figure 4c, only the deepest marks remain. The wear morphology observed is fully consistent with the asperity contact theory of friction model based on the work of Bowden and Tabor [11] and Archard [12]. According to this model, when two rough solid surfaces are in macroscopic contact, mechanical interaction mainly happens between the tips of the asperities. Therefore, plastic deformation and wear processes initially only happen in the highest asperities of the surface.

Based on this model, the wear mechanism can be identified as a combination of plastic deformation of the tips of the asperities and abrasive wear. The mechanism causing abrasive wear is ploughing from the asperities of the similarly hard sheet metal counterpart, aided by a third body abrasion process where oxide scale from the heated blank would act as an abradant.

In addition to this abrasive wear mechanism, a series of V-shaped lumps could also be identified in the tool (figure 5). These lumps attained sizes larger than 1000 μm in diameter and 70 μm in height within a low number of production cycles, and are surrounded by a volume of sunken material. Lumps appear in tightly packed clusters on localised areas.

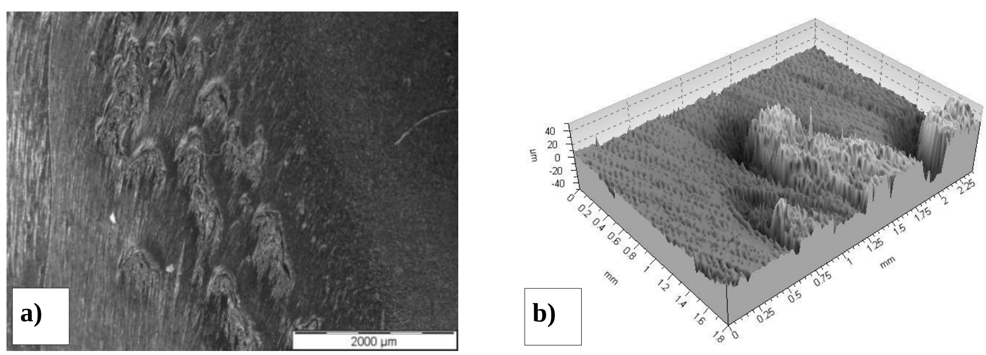

Figure 5: *a) 20x lens image of a lump cluster. b) 200x topography of a lump on a hot stamping tool.*

By their morphology and the characteristics of the industrial process, these features were identified as galling lumps. Galling is defined in the ASTM G40-05 norm as a form of surface damage arising between sliding solids, distinguished by macroscopic, usually localized, roughening and creation of protrusions above the original surface; it often includes plastic flow or material transfer or both. Even though the norm defines the identification of galling as subjective, the damage identified in the studied tools fits the description for galling, and corresponds to the observations of galling made by other authors [13].

The wear mechanism associated to this damage can be attributed to solid state welding between the asperities in the tool and the sheet metal in the spots where direct metal to metal contact exists. These junctions shear preferentially in the volume of the hotter, softer sheet steel, causing material transfer to the tool surface. The transferred particle, irregularly sized and hardened through plastic deformation, acts as a nucleation point for the growth of the galling lump.

Lumps found on the production tool have a characteristic morphology with the maximum height at the vertex. This shape causes an intense ploughing action on the sheet metal, leading to further material transfer and growth of the lump. The sunken area around the features is caused by plastic deformation of the volume under the protruding lump, which supports an increased load due to its height and ploughing effect.

4 Conclusions

Wear mechanisms on hot stamping industrial tools were successfully identified through a surface replication methodology. This technique allowed the study of the tool surface topography using laboratory equipment without disturbing the production process.

The wear mechanisms observed in hot stamping of Al-Si coated 22MnB5 were adhesive wear-related. Material from the sheet metal coating was transferred to the die surface through combined mechanical and chemical interaction. This process was found to be dynamic, as adhesion wear and breakdown happened simultaneously to adhered layer formation and growth, and could generate layers and localised lumps more than 50 µm thick on the tool surface.

In the case of uncoated 22MnB5, the main acting damage mechanisms observed were abrasive wear and galling. Abrasive wear was identified through the blurring and eventual removal of machining patterns on the tool surface, and was caused by the ploughing effect of sheet metal asperities and third body abrasion caused by oxide scale. Galling was identified as macroscopic V-shaped lumps, up to 70 µm in height and more than 1 mm in diameter and formed due to direct metal to metal contact in conditions of high load and temperature.

The results obtained in this work will improve the performance of hot stamping lines, allowing even further increase in part quality and optimisation of the maintenance cycles in press shops. The knowledge gained on wear mechanisms will be useful for the design of laboratory wear tests which reproduce the same fundamental wear mechanisms observed in the industrial process, and for the design of wear preventive solutions for hot stamping tools.

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Paper II

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