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**Evaluation of abrasive mechanisms in metallic alloys during scratch tests: a
numerical-experimental study in micro-scale**

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To my beloved mother Teresa.

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*No, our science is no illusion. But an illusion
it would be to suppose that what science
cannot give us we can get elsewhere.*
(Sigmund Freud)

ABSTRACT

Abrasion can have some approaches, such as: (i-) the manufacturing processes and the production of different surface features and finishing of workpieces and components; and (ii-) in terms of the wear and failure, which occur in tools materials. The present thesis consists in developing a numerical model by the Finite Element Method (FEM) validated by experimental tests, aiming to evaluate microstructural influences on the abrasion of metallic alloys. A simplified abrasion study was developed considering a single abrasive over a microstructure to evaluate from ploughing to cutting. Regardless of the applications, the abrasive micro-mechanism depends on mechanical and damage properties of the materials and their microstructure. Thus, scratch tests were performed in micro-scale, applying different ranges of constant normal force. Five materials were evaluated using this approach: (1) hard matrix; (2) hard matrix with hard second phase particles; (3) soft matrix; (4) soft matrix with soft particles; and (5) soft matrix with hard particles. The mechanical and damage properties of these materials were used as inputs of the numerical modelling, in which the element deletion combined with the damage models promoted the material removal. Moreover, the specimen experimental characterization was conducted using X-ray micro-tomography, electronic microscopy, optical interferometry, conventional hardness, and instrumented indentation techniques. Therefore, the objectives are related to defining rules focused on ductile material design to obtain cutting resistance, obtaining as major outputs of the tests: friction coefficient, removed volume and specific energy. By the developed numerical approach, the results were: (a-) in terms of apparent coefficient of friction (COF), the found numerical and experimental mismatches are assigned to the effects of adhesion, anisotropy, grain boundaries and twins, for instance, which were not taken into account in the modelling; (b-) the hard precipitates have a prevailing effect over the matrix regarding the apparent COF, whereas the matrix effect is dominant in soft precipitates in the COF behaviour; (c-) as a consequence of the reduced strain energy and tangential load, the local COF decreases when the abrasive scratches the hard precipitates; (d-) regarding the depth of penetration and removed volume, the numerical results are in accordance with the experimental results; (e-) there is not a remarkable micro-mechanism transition using the approach of the dimensional wear coefficient as a function of normal load, which indicates that several abrasive micro-mechanisms can occur in a single scratching; however, a predominance of a certain micro-mechanism can be observed; (f-) hard precipitates often decrease the local depth of penetration and, as a consequence, they reduce the removed volume and increase the local specific energy; (g-) considering the studied range of the normal load, soft precipitates follow the matrix mass removal behaviour, but these particles can show fluctuations of the local specific energy in the less severe abrasion; and, finally, (h) the map of the abrasive resistance $\times$ deformed hardness/attack angle is an important tool to point out the dominant ductile or brittle effect on the microstructure (mechanical properties); and it delineates frontiers for the abrasive micro-mechanisms.

Keywords: Abrasion. Metallic alloys. Finite Element Method. Microstructure.

RESUMO

A abrasão pode ser tanto tratada do ponto de vista de processos de manufatura e geração de características superficiais distintas em peças e componentes, quanto pode ser abordada em termos de desgaste e falha em diferentes ferramentas aplicadas em processos de fabricação. A presente tese remete ao desenvolvimento de um modelo numérico pelo Método dos Elementos Finitos (MEF), validado por ensaios experimentais, com o objetivo de avaliar influências de aspectos microestruturais na abrasão de ligas metálicas. A avaliação do micro-sulcamento ao micro-corte foi realizada a partir de um estudo simplificado de abrasão, considerando o riscamento de microestruturas por um único abrasivo. Independentemente da aplicação, os estudos da ocorrência dos micro-mecanismos de abrasão incorporam propriedades mecânicas e de dano dos materiais e suas fases. Dessa forma, ensaios de riscamento em micro-escala aplicando força normal constante dentro de uma faixa específica. Cinco materiais foram avaliados por meio desta abordagem: (1) matriz dura; (2) matriz dura com partículas de segunda fase duras; (3) matriz macia; (4) matriz macia com inclusões moles; e (5) matriz macia com partículas de segunda fase duras. As propriedades mecânicas e de dano desses materiais foram utilizadas como entradas da modelagem numérica, na qual a deleção de elementos combinada aos modelos de dano promoveram a remoção de material. Em adição, as caracterizações das amostras são realizadas a partir de técnicas de micro-tomografia de raios-X, microscopia eletrônica, interferometria óptica, dureza convencional e indentação instrumentada. Portanto, o objetivo desta tese é focado na construção de regras que auxiliem no projeto de materiais dúcteis para terem resistência ao corte, obtendo como saídas principais dos testes: o coeficiente de atrito, o volume removido e a energia específica. Por meio da abordagem numérica desenvolvida, os principais resultados obtidos foram: (a-) in termos de coeficiente de atrito aparente (COF), as divergências numérica e experimental encontradas foram decorrentes principalmente do efeito da adesão, anisotropia, contornos de grão e maclas que não foram levadas em conta na simulação; (b-) os precipitados duros tendem a ter efeito predominante sobre a matriz no que diz respeito ao COF aparente, ao passo que a influência da matriz é predominante sobre o comportamento dos precipitados moles; (c-) como consequência das reduções de energia de deformação e força tangencial, o COF local tende a ser diminuído quando o abrasivo passa por precipitados duros durante o riscamento; (d-) os resultados numéricos de profundidade de penetração média e volume removido total são consistentes com os resultados experimentais; (e-) não há uma transição marcante de micro-mecanismo em função de coeficiente de desgaste dimensional em função da carga, indicando uma predominância de um certo micro-mecanismo em um dado risco e, não ocorrendo transições bruscas; (f-) precipitados duros, em geral, diminuem a profundidade de penetração local e, assim, eles reduzem o volume removido e aumentam a energia específica; (g-) para a faixa de força normal avaliada, os precipitados moles seguiram o comportamento de remoção de material de sua matriz, mas podem apresentar oscilações na energia específica local nas condições menos severas de abrasão; e, por fim, (h-) o mapa de resistência à abrasão x dureza após deformação/ângulo de ataque é uma importante ferramenta para definir efeitos dominantes de propriedades mecânicas (dúctil e frágil) na microestrutura submetida à abrasão, e faz o delineamento de fronteiras de micro-mecanismos abrasivos.

Palavras-Chave: Abrasão. Ligas Metálicas. Método dos Elementos Finitos. Microestrutura.

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ABBREVIATIONS

AISI	American Iron and Steel Institute
ANOVA	Analysis of Variance
BNDES	Brazilian Development Bank
BSE	Backscattered electrons
CCDM	Centre for Development and Characterization of Materials
COF	Coefficient of friction
C3D8	8-node brick element with full Gauss integration
DF	Degrees of freedom
EBSD	Electron Backscatter Diffraction
ECM	Expanding Cavity Model
EDX	Energy-Dispersive X-Ray Spectroscopy
EP	Electrolytic Polishing
EPUSP	Escola Politécnica of University of São Paulo
FCC	Face-Centered Cubic
FEG	Field emission gun
FEM	Finite Element Method
IPT	Institute for Technological Research
LabMicro	Electronic and Atomic Force Microscopy Laboratory
LCT	Technological Characterization Laboratory
LEFM	Linear Elastic Fracture Mechanics
LFS	Surface Phenomena Laboratory
MSDM	Micro-Scale Dynamic Model
MP	Mechanical Polishing
MQ	Mean square
PEEQ	Equivalent plastic strain
PME	Department of Mechanical Engineering
PMI	Department of Mining and Petroleum Engineering
PMR	Department of Mechatronics Engineering and Mechanical Systems
PMT	Department of Metallurgical and Materials Engineering
SDEG	Scalar stiffness degradation
SE	Secondary electrons
SQ	Sum of squares

SEM	Scanning Electron Microscopy
SFE	Stacking fault energy
SPM	Scanning Probe Microscopy
UFSCar	Federal University of Sao Carlos
UTS	Ultimate tensile strength
2D	Two-dimensional
3D	Three-dimensional

NOMENCLATURE

LATIN SYMBOLS

a	Indentation radius
A	Area – circular segment
A_g	Groove area
$A_{p1}+A_{p2}$	Area of the pile-up sum
A_r	Area of material removal
d	Depth of cut
d_V	Average of the diagonals – Vickers hardness
D	Scalar damage or cumulative damage tensor
D_p	Degree of penetration
E	Elastic modulus
E_a	Absorbed energy
E_c	Cutting energy
E_d	Energy consumed in ductile-mode cutting
E_f	Frictional energy
E_i	Elastic modulus of the indenter
E_{pd}	Dissipated strain energy
E_r	Reduced modulus
E_s	Elastic modulus of the indented surface
E_{sd}	Specific cutting energy for ductile mode
E_{sp} or E_s	Specific energy
E_T	Total energy consumed during the scratching
f	Interfacial shear strength / yield stress ratio
f_{ab}	Material removal factor due to abrasion
F_c or F	Resulting cutting force
$F_{critical}$	Critical value for the ANOVA, based on a significance level (F-distribution)
F_t	Tangential force
G_f	Energy release rate
h or x	Depth of penetration, groove depth or scratch depth
h_g	Depth of the graphite
h_s	Pile-up height

H (or H_m)	Indentation hardness of the abraded material (or body hardness)
H_a	Abrasive hardness
H_{def}	Hardness of the deformed material by the abrasion effect
H_{SM}	Soft matrix hardness
HS_p	Scratch Hardness Number
I	Second-order identity tensor
k	Dimensional wear coefficient
K	Dimensionless wear coefficient
K_s	Material strength coefficient
K_{IC}	Fracture toughness (mode I)
l_p	Scratch width
m	Removed mass
<i>m</i>	Material removal rate
n	Strain-hardening exponent
N	Data number of the log (stress σ) x log (strain ϵ) curve
p	Hydrostatic pressure,
P_c	Cutting power
P-h	Load-displacement curve
q	von Mises stress
Q	Volume removed by abrasion/sliding distance ratio
R	Pin tip radius (Abrasive tip radius)
R_h	Radius of the ECM hydrostatic core
r_p	Outer radius of the ECM plastic zone
s	Sliding distance (or length of scratching)
S_a	Arithmetic Mean Height
S_{dq}	Root Mean Square Gradient (Surface Slope)
S_{ku}	Kurtosis
S_{pc}	Arithmetic Mean Peak Curvature
S_q	Root Mean Square Height
S_{sk}	Skewness
t_{critical}	Critical value for T-test
U_T	Toughness
V	Removed volume due to the abrasive action

V_c	Volume of spherical cap
V_d	Volume of removed material for ductile mode
V_{pu}	Pile-up volume
V_T	Total volume
v_s	Scratching speed
W	Normal load
Z_{gt}	Geometric transition depth

GREEK SYMBOLS

α	Semi-angle of the abrasive particle
β	Central angle
γ	Vickers indenter angle
ε_f	Strain at the beginning of the material damage
ε_{total}	Elongation
e	Specific absorbed energy
θ	Attack angle
θ_c	Critical attack angle
Λ	Dimensionless Johnson parameter regarding the strain
μ_0	Apparent COF
μ_{adh}	Interfacial friction
μ_p	Ploughing friction
η	Triaxiality
ρ	Material density
σ	Applied stress tensor
$\bar{\sigma}$	Effective stress tensor
$\bar{\sigma}_e$	Equivalent yield stress
$\bar{\sigma}_y$	Yield stress
σ_f	Tensile strength
σ_x, σ_y and σ_z	Normal stress components
τ_{cis}	Shear stress
u_c	Specific cutting energy
u_{ch}	Specific chip formation energy

u_{pl}	Specific ploughing energy
$\bar{u}^{pl}$	Effective plastic displacement at the failure point
ν	Poisson ratio
ν_i	Poisson ratio of the indenter
ν_s	Poisson ratio of the indented surface

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1 INTRODUCTION

The degree of complexity to be assigned to the studies of relations between the processing, mechanical and thermal-physical properties and material microstructure is defined based on the following evaluation: (i-) if the material can be considered homogeneous and isotropic, there is the macroscopic condition; and (ii-) if the analysis should be conducted in view of the heterogeneous and anisotropic material, in which defect and phase features strongly influence the final result, there is the microscopic condition (MACHADO, 2012).

In the literature concerning abrasive processes, several works (JOHNSON; COOK, 1983, 1985; DIETER, 1986; FANG; JAWAHIR, 2001; KOPAC; KOROSSEC; KUZMAN, 2001; ATKINS, 2003) consider the homogeneity and isotropy of materials as assumptions to evaluate stress fields, obtaining representative global results. However, these hypotheses are not applicable in the majority of cases because the material mechanical properties are directly dependent on their microstructures (SHAW, 2005; SWAMINATHAN et al., 2007; GONZÁLEZ SANTOS, 2008; LIN; HUANG, 2008; CHAWLA; MEYERS, 2009; GUO et al., 2011).

With respect to mechanical properties, thermal-mechanical evolution affects them, defining the softening and hardening of the metallic material for a given load during the manufacturing process. This history can be incorporated into the analysis in order to better define the mechanical behaviour of the material (MEYERS; VÖHRINGER; LUBARDA, 2001; MEYERS et al., 2002; ROSA et al., 2011). Another aspect for intensifying the analysis complexity of manufacturing processes consists in studying of phase transformations, which result in a dynamic approach (MACHADO, I. F.; PADILHA, 1996; MACHADO, 1998; SOUZA, 2006).

Focusing on the machining processes, components are produced by means of the material removal, attaining desired geometries, sizes and surface finishing conditions. They may be classified into: (1) processes with well-defined tool edge geometry, e.g. turning and milling, which always result in cutting and chip formation of materials; and (2) processes with undefined tool edge geometry, such as grinding, lapping, honing and polishing, which involve the different abrasive mechanisms (MACHADO et al., 2011; KALPAKJIAN; SCHMID, 2014; YOUSSEF, 2015).

Abrasion, the prevailing phenomenon throughout machining processes with undefined tool edge geometry, occurs as a consequence of the sliding movement of hard particles over the softer material surface, providing its plastic deformation, detachment or mass removal (GAHR, 1998). **Figure 1** shows the mechanisms from deformation (burnished) to cutting (removed) related to the abrasion occurrence during the grinding of a ductile material surface, according to Griffiths (2001). Also, the figure points out areas of interest for studying abrasion in view of design variables, tribosystem operating conditions, abrasive features and abraded surface properties. The present thesis focuses on the material properties submitted to abrasion, specifically alloy microstructural aspects.

A coupled study with material properties, microstructure and mechanical behaviour can be established to evaluate the effects of abrasive mechanisms resulting from machining processes. For instance, the hardness, machinability, surface finishing and dimensional stability of materials can be considered research subjects. Thus, improvements and enhanced control are claimed in view of ductility, toughness, and fatigue, corrosion and wear resistances of the manufactured component (JHAVAR; PAUL; JAIN, 2013).

Numerical simulation by the Finite Element Method (FEM) is considered as a possible resource to be applied to the abrasion studies. To use this deterministic analysis, one of the main inputs is the constitutive model, which describes the stress-strain curve based on the mechanical properties of the material. Moreover, damage nucleation and propagation criteria are necessary in the numerical modelling to predict the cutting (TKAYA et al., 2007; VALBERG, 2010).

As stated in Jeswiet et al. (2008), one of the major difficulties in modelling different steps of the manufacturing process is the microstructural evolution of their components since each step can present material changes. The authors summarize that the microstructural modifications affect the numerical study of the material, interactions occurring between variables, mechanisms and process conditions in modelling by FEM due to the thermomechanical history.

Regarding the experimental validation, tribological tests are evaluated in terms of the phenomenon, and not in terms of its frequency of occurrence (statistical), concentrating on initial conditions that determine the final mechanical behaviour of the material in view of the mass removal caused by abrasive actions. Then,

phenomenological equations in addition to the numerical model incorporated into the finite element software, become powerful tools to prescribe the ductile material response throughout manufacturing processes.

To provide a general analysis of the worked issues, this thesis was divided into the following sections: objectives (chapter 2); review of the main literature (chapter 3); materials and methods (chapter 4); results and discussions (chapter 5); conclusions and final remarks (chapter 6); and proposals for future works (chapter 7). Finally, the references and annex section are listed.

Figure 1 – Scheme of different mechanisms verified in abrasive manufacturing processes of components, in which the mechanisms derive from the effect of the attack angle (rake angle) of the abrasive particle over the piece surface. Furthermore, general classes of abrasion analyses are depicted with details about the areas of interest.

Reference: (GAHR, 1987; GRIFFITHS, 2001)

2 OBJECTIVES

The overall aim of the present thesis is to develop a numerical model using finite elements, able to determine the microstructure influence on the material removal by abrasion of metallic alloys. Hence, a deterministic model and its experimental validation are developed to study the parameters that dominate the abrasive mechanisms in different materials, which present combinations of mechanical properties of the matrix and their second phase particles.

Specific objectives of this work are outlined as follows:

- (1) to model the abrasion phenomenon by FEM, conducting scratch tests in micro-scale. The model will show a comparison between numerical and experimental results. The normal load is considered the main input variable to investigate the critical conditions, which produce the transition of micro-mechanisms, from micro-ploughing to micro-cutting. An approach based on the specific energy and the friction coefficient is applied to accomplish this purpose;
- (2) to correlate properties of the material phases to the material removal, particularly the ratio between elastic modulus and hardness (E/H);
- (3) to study, by means of experiments, aspects which may affect the scratching of materials, such as crystallographic orientation and surface finishing, to categorize the range in which the numerical model is valid (model limitations);
- (4) to present general discussions about the damage occurrence, predominantly ductile or by shear, based on the triaxial state arisen from the material microstructures after the scratch tests (numerical evaluation).

2.1 BACKGROUND AND ORIGINAL CONTRIBUTION

Difficulties regarding quantitatively evaluating the material removal by abrasion throughout machining processes, with undefined tool edge geometry, are known on account of their high complexity. In this respect, a simplified approach can be adopted to provide a better understanding in view of the mechanical behaviour of materials as well as their response when submitted to abrasive mechanisms. Thus, scratch tests can be applied to analyse the abrasion and to specify rules to design materials more susceptible to the cutting. In addition, tests in micro-scale allow investigating the microstructure effects and mechanical properties of different metallic alloys during the abrasion processes.

Numerical simulation by finite elements consists in a great resource to model ductile materials and to facilitate the design of new alloys. In turn, experiments in laboratory scale allow validating the developed models and, thus, their consolidations. Therefore, the elaboration of a computational model of scratch test in micro-scale developed here, aided by the theoretical and experimental fundaments, enriches the approach and reduces resources involved in case only one of these fields was developed. It is worth mentioning the model inputs are: real mechanical properties, constitutive equations, loadings and boundary conditions that tend to reproduce the process and laboratory conditions (abrasion tests).

3 LITERATURE REVIEW

3.1 FUNDAMENTALS OF ABRASION

Abrasion occurs when the material surface (body) is removed or deformed by the movement of hard particles (sliding or rolling), as well as by the contact between the body and the hard asperities of another surface (counterbody), which results in indentations (ASM INTERNATIONAL, 2017; HUTCHINGS; SHIPWAY, 2017).

Generally, abrasion is more pronounced when the abrasive hardness is higher than the hardness of the material evaluated. In this case, the abrasive effect takes place as penetration over the material surface, causing its plastic deformation and further cutting with final material removal (ASM INTERNATIONAL, 2017). Soft abrasion can be defined for ratios of abrasive hardness and body hardness (H_a/H_m) lower than 1.2, whereupon hard abrasion is pointed out for H_a/H_m higher than 1.2 (HUTCHINGS; SHIPWAY, 2017).

In addition, the morphology, size and fracture toughness of abrasive particles are relevant parameters in abrasion studies. Abrasive size and morphology affect the attack angle (and thus the body-counterbody contact), groove formation over the worn surface, contact pressure and transition from elastic to plastic contact. For a range of hardness, the increase of fracture toughness may intensify the volume removal due to abrasion (ASM INTERNATIONAL, 2017).

In terms of tribosystem features, the temperature, speed, normal load, humidity, physical and chemical environmental conditions can influence the abrasion behaviour (GAHR, 1998; ASM INTERNATIONAL, 2017).

One of the main approaches of the effect of normal load on removed volume on account of the abrasion was conducted by Rabinowicz; Dunn; Russell (1961), considering three-body abrasion tests. Based on **Figure 2**, the assumption of conical abrasive particle and ductile material surface to be abraded, the following equations - eq. **(1)**, **(2)** and **(3)** - were pointed out by Hutchings; Shipway (2017). These formulations complement the description in Rabinowicz; Dunn; Russell (1961) about the load distribution area – hatched area in plain view (**Figure 2a**). The contact area is in front of the particle, in view of its sliding (HUTCHINGS; SHIPWAY, 2017).

Figure 2 – Model idealized with the cono-spherical abrasive against a ductile surface, promoting its plastic deformation: (a-) tribological system simplification; (b-) elevation view; and (c-) plan view.

Reference: (HUTCHINGS; SHIPWAY, 2017)

$$W = H \cdot \underbrace{\frac{1}{2} \pi a^2}_{\text{contact area}} \quad (1)$$

Where: W is the normal load applied, H is the indentation hardness of the abraded material, and a is the indentation radius (**Figure 2c**).

Moreover, the volume removal (V) along the sliding distance (s) is given by eq. (2).

$$V = s \cdot \underbrace{a \cdot x}_{x = \frac{a}{\tan \alpha}} = \frac{a^2}{\tan \alpha} \cdot s \rightarrow a^2 = \frac{V}{s} \cdot \tan \alpha \quad (2)$$

In which: x is the depth defined in **Figure 2b**, and α is the semi-angle of the abrasive particle.

By replacing eq. (2) in eq. (1), eq. (3) can be obtained.

$$W = H \cdot \frac{1}{2} \pi \cdot \frac{V}{s} \cdot \tan \alpha \rightarrow \frac{V}{\frac{Q}{H}} = \frac{2}{\frac{\pi \tan \alpha}{K}} \cdot \frac{W}{H}$$

$$\therefore Q = K \cdot \frac{W}{H} \text{ or } Q = k \cdot W \quad (3)$$

Where: Q is the volume removed by abrasion/sliding distance ratio, K is the dimensionless wear coefficient that takes into account the particle geometry and the removed material (firstly deformed), and k is the dimensional wear coefficient, also known as the specific wear rate.

The K coefficient depicts the severity of the material removal by abrasion. **Table 1** shows some K values resulting from abrasive phenomena. As a function of particle movement, Hutchings; Shipway (2017) reported three-body abrasion as rolling abrasion, and two-body abrasion as sliding abrasion. Focusing on dry contact, Childs (1993) indicated the same K range for 3-body abrasive and severe adhesive.

Eq. (3) is similar to the Archard's equation (ARCHARD, 1953) proposed for sliding wear. In addition, the use of the eq. (3) is limited to the condition in which the applied force is strong enough to provide the abrasive particle break down (ASM INTERNATIONAL, 2017).

Table 1 – Dimensionless wear coefficient (K) ranges reported in the literature in terms of 3-body and 2-body abrasion.

Abrasive mechanism	K range	References
Three-body*	$10^{-6} \sim 10^{-2}$	(GAHR, 1998)
$5 \times 10^{-4} \sim 50 \times 10^{-4}$	(HUTCHINGS; SHIPWAY, 2017)
	$10^{-4} \sim 10^{-2}$	(CHILDS, 1993)
Two-body*	$10^{-5} \sim 10^{-1}$	(GAHR, 1998)
$5 \times 10^{-3} \sim 50 \times 10^{-3}$	(HUTCHINGS; SHIPWAY, 2017)
	$10^{-3} \sim 10^{-1}$	(CHILDS, 1993)

Reference: *(GAHR, 1998)

3.1.1 Abrasive mechanisms

In terms of mild and severe abrasion, Hokkirigawa; Kato (1988) conducted experimental analyses to study abrasive mechanisms in brass, carbon and stainless steels, using a steel pin with spherical tip. As can be seen in **Figure 3**, a transition from mild abrasion (or micro-ploughing) (c) to severe abrasion (or micro-cutting) (a) was characterized by Scanning Electron Microscope (SEM) after scratch tests with discs under rotation. The transition step provided a characteristic wedge formation (b). The increase in the material plastic strain level, caused by the abrasive sliding over its surface, can favour the mass removal (debris), groove and pile-up formations or prow with material accumulation in front of the abrasive particle. Material failure in cutting mode may happen as a function of excessive plastic deformation of the material.

Figure 3 – Characterization by SEM: (a-) micro-cutting; (b-) wedge formation; and (c-) micro-ploughing. Conditions in (a-): dry contact; material – polished brass (10 mm radius) and abrasive pin – quenched steel (27 μm radius); normal load of 0.71 N; and friction coefficient of about 1.2. Conditions in (b-): dry contact; material – polished 304 stainless steel (10 mm radius) and abrasive pin – quenched steel (27 μm radius); normal load of 0.71 N; and friction coefficient of about 2.2. Conditions in (c-): dry contact; material – polished brass (10 mm radius) and abrasive pin – quenched steel (62 μm radius); normal load of 0.35 N; and friction coefficient of about 0.4. The scheme illustration shown below involves the same mechanisms sequence.

References: (HOKKIRIGAWA; KATO; LI, 1988); (GÅHLIN; AXÉN; JACOBSON, 1998)

According to Hokkirigawa; Kato (1988) and Hokkirigawa; Kato; Li, (1988), the degree of penetration (D_p) was designated as an index that indicates the severity of the sliding contact. A mechanism map was created by the authors considering the degree of penetration and attack angle as a function of bulk hardness of the material. To satisfy that proposal and to formulate D_p , cases of the sliding contact between a diamond pin with spherical tip and polished materials (bearing steel with different heat treatments) were simplified as a hemispherical asperity against a flat surface (**Figure 4**).

Figure 4 – Simplified contact model used to represent a pin spherical tip (hemispherical region) against the disc (flat surface) – a = indentation radius, which is half of the contact width; h = groove depth; and R = radius of pin tip; and θ = attack angle.

References: (HOKKIRIGAWA; KATO, 1988; HOKKIRIGAWA; KATO; LI, 1988).

As stated in Hokkirigawa; Kato (1988), the degree of penetration (D_p) is calculated in eq. (4) based on the parameters displayed in **Figure 4**.

$$D_p = \frac{h}{a} \quad (4)$$

Where: h is the groove depth, and a is the indentation radius.

For geometry, the triangle shown in **Figure 4** had the following condition:

$$R^2 = a^2 + (R - h)^2 \rightarrow h = R - \sqrt{R^2 - a^2} \quad (5)$$

In which: R is the pin tip radius.

Replacing eq. (5) in eq.(4), eq.(6) is obtained:

$$D_p = \frac{R}{a} - \left(\frac{R^2}{a^2} - 1 \right)^{1/2} \quad (6)$$

Finally, isolating indentation radius (a) provided by eq. (1) and replacing it in eq. (6), the degree of penetration can be calculated by means of eq. (7).

$$D_p = R \left(\frac{\pi H}{2W} \right)^{1/2} - \left(\frac{\pi H}{2W} R^2 - 1 \right)^{1/2} \quad (7)$$

Where: W is the normal load applied, and H is the indentation hardness of the abraded material.

As suggested by Hokkirigawa; Kato; Li (1988), **Figure 5** reveals that there is a direct relationship between the degree of penetration - eq. (7) – and the attack angle as a function of hardness of material under abrasion. This idea agrees with the

observation of the triangle in **Figure 4**, of which eq. (8) is obtained to calculate the attack angle (θ) as a function of abrasive radius (R) and depth of penetration (h).

$$\theta = \cos^{-1} \left(\frac{R - h}{R} \right) \quad (8)$$

As listed in **Table 2**, results from eq. (8) are consistent with Machado et al., 2017 research, which provided a regression function - eq. (9) – for the attack angle, based on a cono-spherical abrasive with tip radius of 5 μm and apex angle of 60°. The geometric transition depth (Z_{gt}), from the spherical to the conical region, is equal to 2.5 μm (**Figure 6**) for this case.

$$\theta = 37.392 \cdot h^{0.5208} \quad (9)$$

In which: θ is the attack angle and h is the penetration depth.

Figure 5 – Maps of abrasive mechanisms, defined for the variation of attack angle and degree of penetration as a function of hardness of the worn material. In this case, the abrasive is spherical with a 25 μm radius.

Reference: Adapted from (HOKKIRIGAWA; KATO; LI, 1988).

Table 2 – Attack angle as a function of penetration depth calculated by equations supplied by Hokkirigawa; Kato (1988) and Machado et al. (2017), respectively.

Tip radius of abrasive particle (μm)	Depth of Penetration (μm)	θ – eq. (8)	θ – eq. (9)
5	0.5	25.8	26
	1.0	36.9	38
	2.0	53.1	54
	2.5	60	60

Figure 6 – Scheme of the variation in the attack angle with the depth of penetration, considering a cono-spherical abrasive particle with a 5 μm tip radius.

Reference: (MACHADO et al., 2017).

As can be seen in **Figure 5**, the necessary degree of penetration to promote the micro-cutting transition for the hard materials was lower than for the soft materials. Likewise, the attack angle to provide the transition from wedge formation to micro-cutting dropped for the high values of hardness (HOKKIRIGAWA; KATO; LI, 1988).

In terms of deformation models, Challen; Oxley (1979) studied the contact of a hard asperity against a soft surface by slip-line field analysis, proposing the three abrasive mechanisms (ploughing, wedge formation and cutting) and their correspondences in view of the friction coefficient and mass removal ratio. The authors examined the features of the deformation modes described by Hutchings; Shipway (2017), and displayed in **Table 3**, considering plane strain, without material flow on the normal planes to the sliding surfaces, and soft surface as isotropic, elastic-perfectly plastic.

Table 3 – Description of the modes of deformation that generate the abrasive mechanisms in ductile materials, and scheme obtained from the slip-line analysis presented by Challen; Oxley (1979) and Hutchings; Shipway (2017).

Modes of Deformation	Features	Effect of Attack Angle	Effect of Mechanical Properties
<p>Ploughing (Rubbing model)</p>	(i-) plastic deformation of soft surface occurred without its mass removal; (ii-) material flow was directed down as regards the face of the abrasive particle (groove formation); (iii-) low friction coefficients were found.	Transition from ploughing to cutting occurs when a critical attack angle (θ_c) is reached	Elastic modulus and hardness ratio (E/H) of the abraded material:
<p>Wedge Formation (Wear model)</p>	(i-) wear particle could have originated because of junction formation, deformation and fracture; (ii-) material non-steady flow hindered the friction coefficient calculation since deformation should occur before the material failure.	($\theta_c=30\sim90^\circ$ in most of metals)	$\uparrow E/H = \uparrow \theta_c$
<p>Cutting</p>	(i-) displaced material was fully removed; (ii-) material flow was directed up as regards the face of the abrasive particle – at the shear zone (chip and debris formation); (iii-) high friction coefficients and removed volume rates were found		

As a result of Challen; Oxley (1979) approach, the diagram in **Figure 7** was assembled, delimiting regions of abrasive mechanisms from the friction coefficient as a function of attack angle. Another parameter was introduced in this work to delineate the graph: ratio f between the interfacial shear strength and the yield stress. The f values vary from zero – perfect lubrication – to one – strong adhesion.

Figure 7 – Diagram of abrasive mechanisms: friction coefficient as a function of attack angle of the abrasive particle and f ratio.

Reference: (CHALLEN; OXLEY, 1979)

3.1.2 Mechanical properties effects on the abrasion

As shown in **Table 3**, the mechanical properties of the body material are important parameters that subsidize the critical attack angle definitions, in addition to the geometric characteristics. Focusing on the two-phase material, the mechanical behaviour of the phases defines its response of penetration depth and, thus, the attack angle of the abrasive. One of the possible techniques suitable to conduct the mechanical characterization of the phases consists in instrumented indentation. From the nanoindentation, load-displacement (P-h) curves can be obtained to calculate the mechanical properties of the microstructure in different regions. Nevertheless, special attention is required to prevent the interference of the property of one phase with the others in the multiphase materials. Pöhl et al. (2015) hold the view that the hardness (H) and the elastic modulus (E) of the hard precipitates of NbC on a matrix of cold work tool steel were underestimated by 28% (**Figure 8c**), when compared to pure NbC, because of its soft matrix. This effect of the soft matrix as well as the influence of the

second phase morphology (stress concentrator) were confirmed by numerical simulation using FEM, in which the plastic strain field is observed (**Figure 8**).

Figure 8 – Nanoindentation performed in a NbC particle dispersed on the matrix of the X210Cr12 tool steel: (a-) Scanning Electron Microscope (SEM) image, including the indentation mark with Berkovich indenter; (b-) plastic strain field (two-dimensional – 2D) of the phases resulting from the indentation; and (c-) load-displacement curves of the pure NbC (reference) and the NbC immersed in the soft matrix.

Reference: (PÖHL et al., 2015)

Reduced modulus and hardness values can be calculated by means of algorithms reported in the literature (OLIVER; PHARR, 1992, 2004; PULECIO, 2010). In turn, the elastic modulus of each phase is computed from the reduced modulus, as supported by Oliver; Pharr (1992). Eq. (10) associates the elastic modulus with the reduced modulus measured by the instrumented indentation:

$$\frac{1}{E_r} = \frac{(1 - \nu_i^2)}{E_i} + \frac{(1 - \nu_s^2)}{E_s} \quad (10)$$

Where: E_r is the reduced modulus; E_i and ν_i are the elastic modulus and the Poisson ratio of the indenter, respectively; E_s and ν_s are the elastic modulus and the Poisson ratio of the indented surface, respectively. Since the indenter material is diamond, E_i corresponds to 1,141 GPa and ν_i is equal to 0.07 (OLIVER; PHARR, 1992).

Some relations in terms of the mechanical properties – hardness and elastic modulus – have been reported in the literature to quantify the abrasion resistance:

- yield strain or elastic strain to failure (H/E): the criterion of the abrasion resistance is considered in the transition point from non-permanent deformations (elastic regime) to permanent deformations (plastic regime) (FINKIN, 1974);
- $(H/E)^2$: the resistance is related to the transition from elastic contact, in which the Hertz theory is valid, to plastic contact (FINKIN, 1974);
- Elastic Strain Energy Capacity ($H^2/2E$): the modulus of resilience is taken as the abrasion resistance measurement, i.e., the absorbed strain energy until plastic strain occurs by volume unit (FINKIN, 1974). The conclusions in Finkin (1974) suggested that hardness cannot be solely considered as a measurement of the abrasion resistance, since the relations mentioned above, which involve the elastic modulus, may have a great importance to characterize the material removal due to the abrasion of different ductile metallic alloys;
- H/E^2 (JOSLIN; OLIVER, 1990) and H^3/E^2 (TSUI et al., 1995): measurements of resistance to the plastic deformation during the abrasive-surface contact achieved by microindentation and nano-scratch tests. H^3/E^2 data were obtained in ball-on-disc systems, and were proportional to the transition load from the elastic contact to plastic contact (PINTAUDE, 2013). The hardness and elastic modulus were not separately determined.

3.1.3 Influence of the microstructure on the abrasive processes

Gahr (1998) identified the hardness of contact surfaces and fracture toughness of the abraded material as the major mechanical properties influencing the abrasion. Besides, concerning the abrasive, spherical and smooth particles entail a lower material removal by abrasion than sharp particles (ASM INTERNATIONAL, 2017). The effect of abrasive particles is less pronounced on the three-body abrasion than on the two-body abrasion because of the particles rotation (trend to reorient them) during the sliding and/or rolling contacts.

Focusing on the microstructural aspects, Gahr (1998) added that the abrasion resistance of the material is directly affected by the presence of second phase particles in the material matrix. Microstructural parameters that influence the resistance are: the hardness, morphology, size, volume fraction and distribution of the second phase; and properties of the matrix and the matrix-precipitate interface.

A relevant factor for studying the transition from mild abrasion (micro-ploughing) to severe abrasion (micro-cutting) is the relative hardness, which corresponds to the ratio between the abrasive hardness and the worn material hardness. As observed in **Figure 9**, the transition of abrasive regimes of single-phase materials occurs when the abrasive hardness is equal to the material hardness. On the other hand, for multiphase materials, for example, with precipitates and duplex materials, the transition occurs when the hardness of matrix and second phase are, respectively, exceeded for the abrasive hardness. Additionally, if the mass removal of the two-phase material is higher than that of the single-phase material, there is an indicative that the second phase detached of its matrix, adding abrasive (hardened particle) to the interfacial media (GAHR, 1998).

The work by Hu; Li; Llewellyn (2007) may be treated as a case study of abrasion with a microstructural approach. The objective of that study was to evaluate the effects of volume fraction, distribution and morphology of second phase particles, and the abrasive shape on the abrasion of composite materials. The main results of this work can be seen in **Figure 10**. By computational simulations and using Micro-Scale Dynamic Model (MSDM), Hu; Li; Llewellyn (2007) concluded important aspects, which are taken into account in the numerical modelling of abrasion of material microstructures; among them, the following issues can be highlighted:

Figure 9 - Effect of the relative hardness on the transition of abrasion regimes for single-phase and multiphase materials.

Reference: (GAHR, 1998).

- morphology of the second phase particles: **Figure 10(a, b, c)** shows that, considering the same abrasive geometry, the rounded second phase particles result in smaller plastic strain of the material matrix with a lower mass removal by abrasion. On the other hand, the second phase particles with larger angularity tend to reduce the abrasion resistance of the material because they result in the increase of mass loss of the material due to the stress concentrators (PHAN et al., 2017). Cases of the same angularity in **Figure 10(b, c)** were also differentiated by means of orientations of their precipitates: **Figure 10c** shows the conditions in which it seems to generate the highest mass loss (HU; LI; LLEWELLYN, 2007);
- abrasive shape: for the same precipitate morphology, **Figure 10(d, f)** indicates that the spherical abrasive was more difficult to penetrate than the other shapes, considering equal conditions of normal load applied. **Figure 10f** exemplifies the most critical case, because of the effect of the attack angles previously described, resulting in considerable crack formations in the material matrix (HU; LI; LLEWELLYN, 2007);

Figure 10 - Influence of the distribution and morphology of second phase particles on the abrasion of two-phase materials, applying MSDM.

Reference: (HU; LI; LLEWELLYN, 2007).

- volume fraction of the second phase: the increase of the volume fraction of second phase particles that have weak bonds with the matrix favours the cohesive failure, acting on the decrease of the abrasion resistance of two-phase materials. In turn, the rise regarding the volume fraction of the hard second phase can increase the abrasion resistance, with a critical value in which this condition can be reversed (HU; LI; LLEWELLYN, 2007);
- distribution of the second phase particles: the reduction of the abrasion may be obtained by a determined free path (i.e. distance between precipitates), causing a direct effect of the abrasive sizes and, mainly, of the ratio between the abrasive size and second phase particle size (HU; LI; LLEWELLYN, 2007).

Mendas; Benayoun (2013) also incorporated the microstructure effect on the evaluation of the abrasive mechanisms in cast irons, and they characterized those mechanisms by experimental scratch tests in micro-scale. The test parameters were: (i-) diamond conical abrasives, with 2 and 5 μm of tip radius; (ii-) attack angles of 10, 30 and 45°; and (iii-) constant normal load tests ranging from 1 to 15 N. In general, the authors noted that the depth of penetration compared to the graphite lamella size and its orientation in the matrix were the decisive variables for the behaviour of the materials tested. They verified that the coefficient of friction (COF) increased due to the sharper attack angles and higher normal loadings. However, COF reached a steady state for the critical attack angle. The findings of that study, in which the graphite flakes were oriented normal to the scratching direction, suggested the following effect of the relation between the depth of the graphite (h_G) and scratch depth (h_s):

- when $h_G > h_s$ (low normal load - **Figure 11a**), part of the graphite is pushed out, and the second phase particle slides in front of the abrasive. Moreover, the matrix is compressed, without debris formation, and cracks are not observed there (MENDAS; BENAYOUN, 2013);
- when $h_G < h_s$ (high normal load - **Figure 11b**), the material removal by abrasion depends on the matrix and its modes of deformation. Graphite flakes are deformed with the matrix; they can be integrated into its chip, crushed and scattered on the groove regions, or found as matrix fissures (thin spall). The tangential load presents a slight fall in these positions (MENDAS; BENAYOUN, 2013).

In the field of the numerical simulation by FEM, Ljustina; Larsson; Fagerström (2014) discussed microstructural aspects, as a nodularity of graphite presented in iron cast, during the orthogonal cutting processes. The authors performed a numerical and experimental comparison between cutting forces and microstructural evaluation of the chip formation, i.e. severe abrasion, and they validated the numerical model. From **Figure 12**, cracks tend to propagate along the graphite flakes, in case they have irregular morphologies, taking effect as notches (stress concentrators). Consequently, chip fragmentations may be intensified based on the reduction of the graphite nodularity. A higher graphite nodularity of the cast iron provides more spheroidal second phase particles, which minimize the graphite effect on the plastic deformation and abrasion of these materials (**Figure 12**), decreasing their machinability. In brief,

excluding the temperature and cutting speed influences, for great nodularity conditions, higher cutting forces were necessary to promote the machining of the cast iron.

Figure 11 – Scratch tests represented as a scheme, and the influence of the graphite size (h_G) in relation to the depth of penetration (h_S).

Reference: (MENDAS; BENAYOUN, 2013).

Figure 12 – Numerical simulation results of the orthogonal cutting of compacted graphite iron, in which different chip formations can be verified based on the nodularity corresponding to 0, 15 and 25%. Also, a field of equivalent plastic strain (PEEQ) is shown for the 0% condition, and the cutting speed was 350 m/min.

Reference: (LJUSTINA; LARSSON; FAGERSTRÖM, 2014)

3.1.4 Specific energy (E_{sp}) and material removal factor due to abrasion (f_{ab}) as indicators of the transition of the abrasive micro-mechanism

Specific energy (E_{sp}) can be taken as an indicator of the material resistance throughout the scratching (WANG et al., 2006). Usually in manufacturing processes such as machining, which can be simplified for the scratch tests, specific energy is defined as the ratio of the dissipated cutting energy to the removed volume of the material, and it can be formulated according to eq. (11).

$$E_{sp} = \frac{E_T}{V} \quad (\text{BARGE et al., 2008})$$

$$\frac{E_T}{V} = \frac{E_{pd} + E_f + E_c}{V} \quad (\text{WANG; YOON; JI, 2007})$$

Where:

$$\frac{E_{pd}}{V} = U_T = \int_0^{\varepsilon_f} \sigma d\varepsilon$$

Considering Hollomon's model (J.H. HOLLomon, 1945) to describe the stress-strain curve, the following simplification is obtained for the E_{pd} portion:

$$\frac{E_{pd}}{V} = K_s \int_0^{\varepsilon_f} \varepsilon^n d\varepsilon = \frac{K_s \varepsilon^{n+1}}{n+1} = \frac{\sigma_f \varepsilon_f}{n+1} \cong \sigma_f \varepsilon_f \quad (\text{PELLEG, 2012})$$

$$\frac{E_f}{V} = \frac{\int_0^s F_t dx}{\int_0^s A_r dx} \quad (\text{WANG et al., 2006})$$

$$\frac{E_c}{V} = \frac{P_c}{\dot{m}} = \frac{F_c \cdot v_s}{v_s \cdot A_r} = \frac{F_c}{A_r} \quad \text{or} \quad \frac{E_c}{V} = \frac{\int_0^s F_c dx}{\int_0^s A_r dx} \quad (\text{SHAW, 2005; HE; XU, 2016})$$

$$E_{sp} = \sigma_f \varepsilon_f + \frac{\int_0^s F_t dx}{\int_0^s A_r dx} + \frac{\int_0^s F_c dx}{\int_0^s A_r dx}$$

$$E_{sp} = \sigma_f \varepsilon_f + \frac{\int_0^s (F_t + F_c) dx}{\int_0^s A_r dx} \quad (11)$$

In which: E_T is the total energy consumed during the scratching (J); V is the removed volume due to the abrasive action (mm^3); E_{pd} is the dissipated strain energy (J); U_T is the toughness that corresponds to the capacity to absorb energy during the plastic regime for ductile materials (J/mm^3); σ_f is the tensile strength (GPa or J/mm^3); ε_f is the strain at the beginning of the material damage, when the tensile strength is reached (mm/mm); K_s is the material strength coefficient (GPa); n is the strain-hardening exponent; E_f is the frictional energy (J); E_c is the cutting energy (J); F_t is the tangential force (N); A_r is the area of material removal (projected area of cutting) (mm^2); s is the length of scratching (mm); P_c is the cutting power (W); $\dot{m}$ is the material removal rate (mm^3/s); v_s is the scratching speed (m/s); and F_c is the resulting cutting force (N).

Despite the specific energy spent in the abrasive processes to be formulated with complexity, **Table 4** allows noting simplified forms to calculate this outcome, which was reported in the literature for distinct applications.

Table 4 – Different treatments for calculating the specific energy described by the literature, focusing on the material removal.

Parameter	Equation		Purpose	References
Specific wear energy – energy dissipated due to material volume removal by abrasion	$e = \frac{\int_0^s F_t(s) ds}{m/\rho}$	F_t = tangential force; s = sliding distance (scratch length); m = removed mass; ρ = material density.	To study the effect of the contact of multiple abrasives (abrasive paper) on a metal surface.	(JIANG; SHENG; REN, 1998)
Specific absorbed energy (e)	$e = \frac{E_a}{m}$	E_a = absorbed energy; m = removed mass.	To evaluate the abrasion resistance of ductile cast irons by using single pass scratch test (pendulum device).	(VÉLEZ et al., 2001)
Specific cutting energy (u_c)	$u_c = u_{ch} + u_{pl}$	u_{ch} = specific chip formation energy; u_{pl} = specific ploughing energy.	To study mass removal during honing processes.	(LEE, 1993)
Specific cutting energy for ductile mode (E_{sd})	$E_{sd} = \frac{E_d}{V_d}$	E_d = Energy consumed in ductile-mode cutting, including plastic deformation and sliding friction effects; V_d = volume of removed material for ductile mode.	To analyse ductile-brittle transition in rock cutting	(HE; XU, 2016)

Sabeur et al. (2013) studied favourable conditions for cutting whilst they evaluated optimum cases regarding the superficial performance, as shown in the graph in **Figure 13**, which contains the coefficient of friction as a function of specific cutting energy. The size of the circles in the figure denotes the abrasive size, which varies from 30 to 180 μm . The authors stated that smaller abrasives have low cutting efficiency because they demand high specific energy in view of the dominant micro-ploughing. Nonetheless, abrasive sizes from 80 to 100 μm , and sliding speed of 4 $\mu\text{m/s}$, tend to provide the best cutting efficiency of the honing processes (**Figure 13**).

The increase of the rate of material removal by abrasion, which can cause the transition of micro-mechanism from mild to severe, results in the reduction of the specific energy spent during the manufacturing process (LEE, 1993), as can be seen in **Figure 14**.

Figure 13 – Friction coefficient as a function of specific energy, with indication by arrow of optimal cutting conditions of honing processes in hydrodynamic lubrication regime.

Reference: (SABEUR et al., 2013).

Figure 14 – Effect of the decline of the specific energy with the raises of the material removal rate (a) and of the depth of cut (b), which provides the abrasive micro-mechanism transition.

References: (LEE, 1993; HE; XU, 2016)

Another parameter that denotes the abrasive micro-mechanisms is the material removal factor due to abrasion (f_{ab}) based on the areas of the scratching profile, reported in Gahr (1987, 1998) and denominated by the ratio in **Figure 15**. The following analyses can be considered in the figure: (a-) when f_{ab} is 0, the resulting area of the pile-up sum ($A_{p1}+A_{p2}$) is equal to the groove area (A_g). In this case, the abrasive mechanism on the material is dominated by its plastic deformation, with volume conservation, i.e. micro-ploughing; (b-) in turn, when f_{ab} is 1, the null resulting area of the pile-up sum ($A_{p1}+A_{p2}$) is verified. In this case, there is the surface material detachment (volume removal), in which the abrasive micro-mechanism is micro-cutting. However, difficulties related to the measurement of the profile areas can be found as, for example, due to the chip formation that can be considered “false pile-up” (**Figure 16**), according to Franco; Sinatora (2017). Consequently, the reliability of f_{ab} can be low.

Figure 15 – Scratching profile in which the pile-up and groove areas can be observed. These areas are used to calculate the f_{ab} ratio.

Reference: (GOEL; CHERUKURI; TORO, 2012).

Figure 16 – Schematic scratching profile indicating the chip formation on the left, characterized by optical interferometry as being a pile-up similar to the pile-up noted on the right.

Reference: (FRANCO; SINATORA, 2017).

In this thesis, preliminary evaluations by numerical simulation were carried out focusing on the f_{ab} used as an indicative index for abrasive micro-mechanisms. Instead, because of the problems with experimental validation requiring techniques with higher costs (FRANCO; SINATORA, 2017), the specific energy application became more interesting, especially in terms of studying the local dominant micro-mechanism also pointed out by Franco; Sinatora (2017).

3.2 SIMPLIFIED ABRASION STUDIES

3.2.1 Scratch tests in micro-scale

Important variables can be measured for investigating the abrasive mechanisms by scratch tests, such as: (1) tangential force; (2) depth of penetration, or attack angle of the abrasive particle with reference to the material surface; and (3) absorbed energy during the scratching.

Wredenberg; Larsson (2007) developed a numerical model for scratch tests, with discretization by finite elements. Their outcomes were discussed in terms of the adhesion influence added to the COF analyses, and its consequence related to the association between COF and the mechanisms with abrasive predominance. Bowden; Tabor (2001) detailed that the frictional force was composed of the interfacial portion with junction growth, which consisted in the adhesion effect, and the ploughing portion (plastic deformation). In that case, the ratio between the tangential load and normal load, denominated apparent coefficient of friction or macroscopic friction, can be calculated by eq. (12):

$$\mu_0 = \frac{F_t}{W} = \mu_{adh} + \mu_p \quad (12)$$

Where: μ_0 is the apparent COF; F_t is the tangential load; W is the normal load; μ_{adh} is the adhesive or interfacial friction; and μ_p is the ploughing friction.

Briefly, the coefficient of friction can be defined as a ratio between the tangential force (motion resisting) and normal force (pressing body and counterbody). As enunciated by Coulomb (1785), the contact occurs in the asperities of the bodies (real contact area), in which the tangential force is not dependent on the apparent contact area (BLAU, 2009; HUTCHINGS; SHIPWAY, 2017).

Numerical simulations, described by Wredenberg; Larsson (2007), considered a conical abrasive positioned to provide an attack angle of 22° over the elastic-plastic material with different work-hardening conditions. The finite element mesh is displayed in **Figure 17**.

Figure 17 – Finite element mesh to model the macroscopic scratch test, with linear elements – 8 nodes. The material volume of the slab is 10 x 10 x 22.5 mm³.

Reference: (WREDENBERG; LARSSON, 2007)

One of the main results found by Wredenberg; Larsson (2007) can be observed in **Figure 18**. The ploughing friction (μ_p) stabilized at about 0.3 for all the tested materials, with n between 0 and 1. In this graph, a dimensionless Johnson parameter regarding the strain (Λ), including the influence of the E/H ratio and attack angle, is introduced at the axis of abscissas and can be distinguished at 3 levels: (i-) $\Lambda < 3$, meaningful plastic deformation of the material does not occur; thus, only elastic

analysis is taken into account; (ii-) $3 < \Lambda < 40$, plastic deformation of the material is observed and its elastic-plastic properties are considered in the modelling; (iii-) $\Lambda > 40$, the plastic deformation widely acts on the contact area, in which the elasticity does not seem to have a considerably effect in the final response of the material. Further, Wredenberg; Larsson (2007) used an approach to evaluate the work-hardening role in the apparent COF, varying the adhesive part. As shown in **Figure 19**, the apparent coefficient of friction seems to have no effect on the work-hardening for interfacial friction of up to 0.25. Afterwards, COF differences can be observed for work-hardening levels, within the range of the higher values of the adhesive part.

Figure 18 – Ploughing friction (μ_p) behaviour, considering different strain-hardening coefficients (n), as a function of Λ strain parameter ($\mu_{adh} = 0$).

Reference: (WREDENBERG; LARSSON, 2007)

Figure 19 – Effect of the adhesive part ($\mu_i = \mu_{adh}$) on the apparent coefficient of friction (μ_0) for several levels of work-hardening (n). The black line pertains to the greater values of Λ cases, in which the ploughing friction is almost constant ($\mu_p \approx 0.3$ with attack angle of 22°).

Reference: (WREDENBERG; LARSSON, 2007).

The friction can be understood as the energy consumed by the tribological system during the scratching. Vélez et al. (2001) carried out instrumented single-pass pendulum sclerometry tests of cast iron microstructures, using as abrasive the sintered WC with pyramidal geometry, and quantified the absorbed energy by removed mass. The authors reported that this parameter appears to be related to the energy necessary for removing the material due to the abrasion, thus indicating the abrasion resistance of materials. Similarly, they synthesized that the absorbed energy to promote the micro-cutting, resulting from the sclerometry test, is often the sum of the energy factors, as follows: work-hardening of the material under abrasion; creation of new surfaces; friction acting on the abrasive-substrate contact; and phase transformation as strain-induced martensite related to austenite.

For evaluating the effect of the mechanical properties of ductile materials on micro-scratch tests (diamond cono-spherical abrasive with 10 μm of tip radius), Pöhl; Harges; Theisen (2016) recommended the depth of penetration and apparent COF analyses as a function of specific work of fracture obtained by tensile tests, to classify the abrasive response of materials. The authors claim it may not be possible to acquire a straightforward correlation of the hardness and yield stress, for instance, with the test outputs. Accordingly, the influence of combined mechanical properties, e.g. the specific work of fracture, is remarkable to study the tribological behaviour of different materials.

In terms of microstructural investigation during scratch tests, for alloy design and development with improvement related to abrasion resistance, Pöhl et al. (2015) simulated scratch tests using a 3D FEM model and applying an irregular rigid abrasive against an elastic-plastic material. In addition, they described using the scalar Helmholtz free energy and damage variables in the numerical procedure; no more details were provided about their values. The authors concluded the following aspects by means of FEM numerical simulation (**Figure 20**): (i-) hard precipitates smaller than the width of the scratch track were pushed and removed with the chip formation; they did not act to enhance the material removal resistance; (ii-) hard precipitates greater than the track width, considering constant normal load, tend to reduce the local depth of penetration and width; they were not easily removed, and strongly acted on the material removal resistance.

Figure 20 – Numerical simulation by FEM of scratch tests: von Mises stress fields that allow analysing the effect of the size of second phase particles when compared to the abrasive size.

Reference: (PÖHL et al., 2015).

3.2.1.1 Damage study as a subsidy for the abrasion modelling (material removal)

Damage models allow predicting the damage evolution of materials at the time until the macrocrack onset, based on the resulting functions of the stress and strain fields. For metallic alloys, the damage particularly derives from nucleation and coalescence of microcracks and voids (LEMAITRE, 1984).

Damage calculations and predictions can follow two ways described by the interaction between constitutive equations and damage models:

(i-) uncoupled damage in the numerical simulation – the scheme of this procedure is shown in **Figure 21**. For the uncoupled damage model, the stress and strain fields initially calculated (based on known geometry, material constitutive equations and applied loadings) are considered inputs of the new simulation that can map the damage areas (≈ post-processing).

The Continuous Damage Mechanics and the evolution damage parameters determine the conditions for the crack nucleation (damage onset); the Fracture Mechanics and crack propagation factors define the crack propagation (damage growth). Finally, the material is evaluated to verify if the critical condition was reached (LEMAITRE, 1984).

(ii-) coupled damage in the numerical simulation – this approach tends to better reproduce the real behaviour of the materials, since the damage field immediately influences the stress and strain fields (simultaneously). According to Lemaitre (1984), the main property responsible for this effect is the stiffness or the elastic modulus of the materials.

Strain calculations are conducted, considering the stress redistribution. Effective stresses and equivalent strains are assessed by constitutive equations, whilst the damage criterion is introduced in the analysis. The method for calculating the coupled model is more complex than that for the uncoupled model, owing to the feedback of stress and strain fields occurring as a function of accumulated material damage along the numerical simulation (**Figure 22**).

Figure 21 – Schematic representation of the uncoupled analysis of the damage model.

Reference: (LEMAITRE, 1984).

Figure 22 – Schematic representation of the coupled analysis of the damage model.

Reference: (LEMAITRE, 1984)

In Abaqus® software, the modelling of the progressive damage and failure for ductile materials is often conducted in parallel with the plasticity models (e.g. Mises, Johnson-Cook and Hollomon), resulting in coupled analyses (SIMULIA, 2016).

3.2.1.2 Damage equating

The damage definition can be performed in view of the stress-strain curve of an elastic-plastic material, as seen in **Figure 23**. Stresses present linear relation with the strains for the region a-b, in which the elastic behaviour is assigned to the material. The yield stress is reached and the material starts its plastic regime at point b. Then, there is a substantial effect of the permanent strains after the unloading in segment b-c, overpassing the material elastic recovery; this region is called plastic with hardening. The work-hardening effect is verified in **Figure 23** when the stress increases with the strain, rendering the material more resistant mechanically (TKAYA et al., 2007). In contrast, segment c-d of the curve, noted in **Figure 23**, concerns a continuous decrease of the material capacity to support loads, with damage nucleation defined by point c. The damage evolution is verified until the null stress at point d, where the material rupture occurs. Hence, region c-d is described by a criterion of the damage evolution, which can be based on the localized and cumulative plastic deformation (TKAYA et al., 2007).

Similarly, Simulia (2016) presents a list of the parts which compose the damage modelling specifications in Abaqus® software (default settings) based on **Figure 23**:

- (i-) region a-b-c-d': definitions regarding effective material response (undamaged);
- (ii-) point c: criterion of the damage onset;
- (iii-) region c-d: definitions of the rules of damage propagation;
- (iv-) point d: element deletion can be applied to elements, causing their removal from the calculations, since the material stiffness is fully degraded. However, for many materials, there is no physical meaning involving the full stiffness degradation ($D \sim 1$), in which their failure occurs with lower damage values ($D < 1$).

Figure 23 – Stress-strain curve for an elastic-plastic material, in which the cumulative damage can be studied.

References: (TKAYA et al., 2007; SIMULIA, 2016).

Tkaya et al. (2007) reported that the damage can often be understood as a scalar variable related to the stiffness degradation of materials, and it can be associated to their active failure mechanisms. Mathematically, the authors presented an approach for modelling the scalar damage (D), considering exponential damage evolution (**Figure 24**) and using eq. (13).

$$D = 1 - \exp\left(-\int_0^{\bar{u}^{pl}} \frac{\bar{\sigma}_e}{G_f} d\bar{u}^{pl}\right) \quad (13)$$

Where: $\bar{u}^{pl}$ is the effective plastic displacement at the failure point (full degradation); $\bar{\sigma}_e$ is the equivalent yield stress (von Mises stress was assumed as a yield criterion in **Figure 23**); and G_f is the energy to be stored and required to open a unity of the crack area, which causes an energy release rate. Likewise, eq. (13) suggested that the damage evolution is based on the dissipated energy during the stiffness degradation process entailed by the plastic deformation accumulated in materials over time.

Figure 24 – Exponential damage evolution criterion based on the energy release rate (G_f): (a-) equivalent yield stress as a function of the effective plastic displacement; and (b-) damage variable as a function of the effective plastic displacement.

Reference: (SIMULIA, 2016)

The phenomenological description of the cumulative damage for materials is given by a damage scalar parameter (D), which is dependent on the stress and strain states, strain rate and temperature. When D is equal to 0, no damage is presented, and when D is of about 1, the material fracture occurs (LEMAITRE, 1984; LAPOVOK; SMIRNOV; SHVEYKIN, 2000). Moreover, Barlat (2007) depicted that the damage is a tensor variable introduced to modify the stress tensor, implying the material degradation. Eq. (14) is provided regarding tensor damage.

$$\bar{\sigma} = [I - D]^{-1} \sigma \quad (14)$$

In which: I is the second-order identity tensor; $\bar{\sigma}$ is the effective stress tensor; D is the cumulative damage tensor; and σ is the applied stress tensor.

3.2.1.3 Ductile damage criterion and tests to obtain its parameters

The ductile damage criterion, based on Rice; Tracey (1969), concerns the failure mechanisms due to nucleation, growth and coalescence of voids in the material. For the numerical modelling in a commercial finite element software, the following steps must be specified: (i-) damage nucleation – by means of the fracture strain as a function of triaxiality; and (ii-) damage evolution – designed by a displacement related to the defect propagation or the fracture energy (SIMULIA, 2016). Triaxiality is calculated according to eq. **(15)**.

$$\eta = -\frac{p}{q} \quad (15)$$

In which: η is the triaxiality; p is the hydrostatic pressure, which consists of the arithmetic mean of the three normal stress components (σ_x , σ_y and σ_z); and q is the von Mises stress, related to the second invariant of the stress deviatoric tensor.

Generally, the equivalent strain to fracture as a function of triaxiality is determined by tensile, compression and shear tests of notched specimens, which allow the occurrence of the different triaxiality states. In addition, numerical simulations tend to contribute to acquiring the damage nucleation over the applied loading history (BAO; WIERZBICKI, 2004; OH et al., 2007).

Failure modes can be estimated in view of a graph, which contains the fracture strain as a function of triaxiality, as exemplified for an aluminum alloy in **Figure 25**. When the triaxiality is negative (compression), the fracture strain tends to be reduced with the triaxiality increase; a strain minimum point is verified at about 0.2 for the pure shear condition (null triaxiality). In turn, for the low positive triaxiality range, the raise of fracture strain occurs up to 0.45, corresponding to a positive triaxiality of 0.4 obtained under tensile state. Finally, failure occurs due to the nucleation, growth and coalescence of voids (ductile failure) when the strain decrease comes from a triaxiality above 0.4 (BAO; WIERZBICKI, 2004).

Figure 25 – Behaviour of the fracture strain for different levels of triaxiality, obtained by means of experimental and numerical analyses of Al2024-T351 alloy specimens.

Reference: (BAO; WIERZBICKI, 2004).

Dzugan et al. (2012) conducted experimental and numerical studies for the ductile damage in austenitic stainless steels, applying tensile loading in cylindrical specimens with 3 different notch radii (1, 2 and 4 mm), and in butterfly specimens submitted to 6 different angles between 0 and 90° (**Figure 26**). The curve of the equivalent strain to fracture as a function of triaxiality for an austenitic stainless steel, resulting from Dzugan et al. (2012), is displayed in **Figure 27**. This graph might be assumed as part of the damage model parameters applied to the FEM modelling of abrasion of these materials, since it covers a considerable range of triaxiality states (compressive, shear and tensile responses). Conversely, the fitting undertaken by the authors, drafted to gather the results derived from their different tests, should be improved specially at the singularity point.

Figure 26 – Specimens used to study the ductile damage nucleation of austenitic stainless steels: (a-) geometry discretized by finite element, considering one-quarter of tensioned specimen in the numerical model – 1 mm of the notch radius; and (b-) butterfly specimen with tension under different angles.

Reference: (DZUGAN et al., 2012)

Figure 27 – Curve indicating the ductile damage nucleation for an austenitic stainless steel, reported in the literature.

Reference: (DZUGAN et al., 2012)

3.2.1.4 Mechanisms of damage evolution

Focusing on damage evolution, it is important to treat the mechanisms from which such an evolution proceeds in the material. Thus, the importance of the study regarding the material microstructure is considered because the microstructural defects determine its mechanisms as well as the more significant features of the progressive

damage. Consequently, the consideration of material heterogeneities supports the understanding about damage development in multi-phase materials.

With respect to micro-localized and cumulative plastic deformation, **Figure 28** shows some imperfections in the microstructure that can contribute to cause the damage, such as: dislocations, growth and coalescence of voids, micro-crack nucleation and impurities dispersed in solid solution filling punctual defects (e.g. substitutional) of the crystal lattice structures of materials (BARLAT, 2007).

Figure 28 – Schematic representation of the microstructural features, which can affect the damage evolution due to the cumulative plastic deformation.

Reference: (BARLAT, 2007)

The following ranges of the scalar damage (D) are prescribed as representative of damage evolution mechanisms (LAPOVOK; SMIRNOV; SHVEYKIN, 2000):

- $0 < D < 0.2...0.3 \rightarrow$ dislocation pattern is created;
- $0.2...0.3 < D < 0.5...0.6 \rightarrow$ micro-voids are nucleated on the grain boundaries;
- $0.5...0.6 < D < 0.8...0.85 \rightarrow$ micro-voids coalesce and tend to form micro-cracks;
- $0.8...0.85 < D < 1 \rightarrow$ macro-cracks are nucleated and start moving.

In the Linear Elastic Fracture Mechanics (LEFM) field, the employment of eq. (16), considering the plane strain state, provides the energy of failure propagation per area unit. The damage evolution can then be calculated in terms of the fracture energy (G_f), which depends on Mode I fracture toughness (K_{IC}), elastic modulus (E) and Poisson ratio (ν) (ANDERSON, 2017).

$$G_f = (1 - \nu^2) \frac{K_{IC}^2}{E} \quad (16)$$

In order to exemplify the importance of the damage criterion, a graphic comparison between numerical and experimental results of scratch tests without and with damage model is observed in **Figure 29**. The figure was divided into abrasive mechanisms regions (ploughing, transition and cutting) to enable understanding the subject (MEZLINI et al., 2005; TKAYA et al., 2007). The findings of this work are summarized as follows:

- For the apparent COF equal to 0.1 (**Figure 29**), in which the adhesion and elastic deformation of the aluminum alloy are dominant, numerical model results without and with damage model are in good agreement with the experimental results (MEZLINI et al., 2005; TKAYA et al., 2007);
- Once the plastic deformation and the ploughing effects are intensified because of the attack angle increase, numerical values differ from the experimental results. However, as observed in **Figure 29**, numerical results with damage model tend to better reproduce the experimental results than the numerical results without the damage model (MEZLINI et al., 2005; TKAYA et al., 2007);
- For the apparent COF equal to 1.3 – severe cutting – the material suffers severe plastic deformation with chip formation, and the abrasion effects exceed the adhesion influence on the surface material. Under the circumstances, **Figure 29** allows verifying that numerical results with damage coincide with the experimental point. Yet the numerical value without damage model is far from the other results for the same attack angle (MEZLINI et al., 2005; TKAYA et al., 2007). Therefore, the damage model has to be applied to obtain an enhanced analysis of the material mechanical behaviour.

Figure 29 – Numerical and experimental results of scratch test, in which the relevance of the damage criterion in the modelling can be observed, mainly for the severe abrasion (cutting) conditions.

References: (MEZLINI et al., 2005; TKAYA et al., 2007)

3.3 MATERIAL REMOVAL – CALCULATION METHOD

During scratch tests, the total removed volume is simplified in terms of the projected area of the abrasive tip (BROITMAN; FLORES-RUIZ, 2015) or the abrasive-slab contact area, which can be estimated as a circular segment for which the circular arc region is limited by a chord (**Figure 30**). The calculation method is applied to all the depths of penetration.

Figure 30 – Contact area between hard abrasive and soft material based on the circular segment (in yellow).

Reference: Adapted from (WEISSTEIN, 2002).

The calculation method for the circular segment in eq. **(18)**, shown in **Figure 30**, can be divided into the following parameters (WEISSTEIN, 2002):

- Central angle (β):

$$\beta = 2\cos^{-1}\left(\frac{R-h}{R}\right) \quad (17)$$

- Contact Area (A):

$$A = \frac{R^2}{2}(\beta - \sin \beta) \quad (18)$$

Where: R is the abrasive tip radius, h is the depth of penetration and β is the central angle defined by eq. **(17)**.

In turn, the material removal given in terms of the total volume (V_T) is predicted by eq. **(19)**.

$$V_T = \int A \, ds \quad (19)$$

In which: A is the circular segment; and s is the sliding distance.

The removed volume may be calculated for extreme conditions of abrasive mechanisms in accordance with their predominance:

(i-) Eq. **(20)** is used for prevailing micro-ploughing ($f_{ab} = 0$), i.e. the removed volume (V) corresponds to the difference the total volume (V_T) and the pile-up volume (V_{pu}).

$$V = V_T - V_{pu} \quad (20)$$

(ii-) Eq. **(21)** is employed for dominant micro-cutting ($f_{ab} = 1$), i.e. the removed volume (V) is equal to the total volume (V_T).

$$V = V_T \quad (21)$$

For calculating the pile-up volume (V_{pu}), Nikas (2015) presented an analytical model based on the expanding cavity model (ECM). The author studied rolling elastohydrodynamic contacts, but his methodology can be applied to evaluate the spherical indentation of an elastic-plastic surface, with work-hardening considerations. The indenter is considered rigid. **Figure 31** indicates that the subsurface areas affected by the indentation comprise: a hydrostatic part (in green), a plastic zone (in red) and an elastic region (in blue). Based on the geometrical parameters pointed out in **Figure 31**, the following relations are delineated to calculate the pile-up volume via analytical model (GAO; JING; SUBHASH, 2006; NIKAS, 2015):

Figure 31 – Scheme of the pile-up formation due to a spherical indentation of a ductile material surface.

Reference: (NIKAS, 2015).

- Pile-up height (h_s):

$$h_s = \frac{h}{2} \left(1 + \frac{n}{2} \right)^{\frac{2}{n}-2} - h \quad (22)$$

- Volume of spherical cap (V_C):

$$V_C = \pi h^2 \left(R - \frac{h}{3} \right) \quad (23)$$

- Radius of the ECM hydrostatic core (R_h) (R in **Figure 31**):

$$R_h = \sqrt{\frac{2}{h} \left(\frac{V_C}{\pi} - \frac{h^3}{6} \right)} \quad (24)$$

- Outer radius of the ECM plastic zone (r_p):

$$\left(\frac{r_p}{R_h} \right)^3 = \frac{1}{4} \frac{E}{\bar{\sigma}_y} \frac{R_h}{R} \quad (25)$$

- Pile-up volume (V_{pu}):

$$V_{pu} = \pi \left(d R_h^2 - \frac{\bar{\sigma}_y}{E} r_p^3 \right) \quad (26)$$

Where: h is the depth of penetration (w in **Figure 31**); R is the indenter radius (r in **Figure 31**); E , n and $\bar{\sigma}_y$ are, respectively, the elastic modulus, strain-hardening coefficient and yield stress of the indented material. Therefore, for micro-ploughing, the cumulative volume of pile-up along the scratch length can be calculated in terms of eq. (26), which depends on the variables treated in eq. (22)-(25).

4 MATERIALS AND METHODS

4.1 METHODS

4.1.1 Description of the numerical modelling by finite elements of the scratch tests

The preliminary analyses developed in this doctorate (**ANNEX A**) allowed concluding that abrasive mechanisms are better physically simulated using the three-dimensional (3D) approach when compared to the 2D one, since it provides the material flow for the abrasive sidewalls, which consists in the pile-up regions. The numerical model of the scratch test in micro-scale was conducted by Abaqus v.2016® finite element commercial software with its Explicit module.

The first input of the model was the body and counterbody geometries presented in the tribosystem. In this case, to be compliant with the experimental device, the abrasive (counterbody) was cono-spherical with 5 μm of tip radius and 60° of apex angle of the cone. Also, the abrasive particle was considered rigid in the modelling by FEM. The surface materials under abrasion (body) were evaluated as a function of microstructures.

To reconstruct the three-dimensional microstructure of the materials, X-ray microtomography was performed. This technique was adopted in the literature (HUANG; LI, 2013; LI; CHAWLA; JIAO, 2014), according to the following general procedure:

- serial images are acquired by X-ray tomography to reconstruct the microstructure. A segmentation is taken based on grayscales, working with histograms that represent the real features of the microstructure;
- generation of the finite element mesh for the reconstructed 3D microstructure.

The 3D X-ray microscope used in this work was the XRadia VersaXRM-510 by Zeiss®, which is available in the Technological Characterization Laboratory (LCT) of the Department of Mining and Petroleum Engineering (PMI), located at the Escola Politécnica of University of São Paulo (EPUSP). This equipment was suitable to reproduce the morphology, size and distribution of the second phase particles presented in the alloys studied, with a maximum spatial resolution of about 1 μm . To

achieve the ideal resolution, the volume of the specimens of the soft materials (austenitic stainless steels) corresponded to $10.85 \times 1.35 \times 0.43 \text{ mm}^3$, and of the hard material (martensitic matrix + NbC) was $10 \times 1.0 \times 0.5 \text{ mm}^3$. By means of the interaction between the X-ray beam and the specimen, 1 image/5 s was generated for soft materials as well as 1 image/2 s for the hard materials; around 1,000 images were obtained in each case.

The images were treated by Avizo Fire v 8.1 software, from FEI Inc., as a function of density differences of the phases, which made a suited contrast in grayscale possible. Moreover, the computational domain was created in this software, in which the microstructures were discretized into hexahedral elements from voxels. The use of those elements for the numerical simulations of scratch tests was found to be more suitable because of: (i-) the element deletion employed with a better representation of the material removal by abrasion, which is dependent on the damage model fulfilment; and (ii-) obtaining the convergence for the slab-abrasive contact.

Elements and nodes of the slab finite element meshes, constructed considering the microstructures imported in the Abaqus® software, were separated into the following classes:

- (1) Hard materials– domain volume of $95 \times 95 \times 65 \text{ }\mu\text{m}^3$; 2,171,400 linear hexahedral elements of type C3D8 (*8-node brick element with full Gauss integration*); and 2,223,960 nodes (**Figure 32a**);
- (2) Homogeneous soft material and Heterogeneous soft material with soft particles– domain volume of $150 \times 110 \times 60 \text{ }\mu\text{m}^3$; 3,302,700 linear hexahedral elements of type C3D8; and 3,376,209 nodes (**Figure 32b**);
- (3) Heterogeneous soft material with hard particles – domain volume of $228 \times 85 \times 33 \text{ }\mu\text{m}^3$; 3,143,252 linear hexahedral elements of type C3D8; and 3,238,485 nodes (**Figure 32c**).

The sizes of the finite element mesh strongly influence the removed volume to be calculated by numerical simulation. There are two main limitations to create this mesh: (a-) the spatial resolution of the 3D X-ray microscope: the specimen thickness should have the minimum value to allow reaching the best resolution of the device (e.g. $\approx 0.5 \text{ mm}$ thickness for the materials studied $\rightarrow 1 \text{ }\mu\text{m}$ spatial resolution, which was the maximum value for the device utilized); and (b-) the computational resources to process the microstructure reconstruction: in this case, the decision criteria consist in

evaluating whether the volume characterized using X-ray micro-tomography is enough to represent the microstructure features. Thus, only one part of the volume was treated by the Avizo Fire software to provide the best use of time and computational resources.

Figure 32 – 3D meshes applied to model by FEM the scratch tests in micro-scale.

(a-) Heterogeneous hard material: martensitic matrix and niobium carbides, which are divided into a shell (in green – rich in Nb) and a core (in red – rich in Ti).

(b-) Heterogeneous soft material with soft precipitates: austenitic matrix and manganese sulphides.

(c-) Heterogeneous soft material with hard precipitate: austenitic matrix and titanium nitride.

Table 5 – Input values conferred to the numerical simulations of micro-scratch tests.

Material	Normal Loads (mN)	Scratch Length (μm)	Scratching Velocity (mm s^{-1})
Hard (Homogeneous and Heterogeneous)	50, 100, 150, 200, 400 and 500	77	6.0
Soft (Homogeneous and Heterogeneous)	40, 50, 60, 70, 80 and 100	100	4.0

Afterwards, simplified hypotheses were considered regarding the input parameters of the numerical model, and the boundary conditions to study the single-pass scratching that was susceptible to be validated by experimental tests.

- Imposed constant normal load: to analyse abrasive mechanisms at different levels, from mild to severe, normal loads were tested as specified in **Table 5**.

The range of normal loads to be applied during the scratch tests was selected based on Hokkirigawa; Kato; Li (1988)'s work. The map in **Figure 33** comprises the designed normal loads used for studying the abrasive micro-mechanisms, as a function of degree of penetration (**Table 6**). As a first characterization, the Vickers macro-hardness of the homogeneous soft material – AISI 304 steel – in the annealed condition was $198.3 \pm 15.2 \text{ HV}_{10}$, as well as for the homogeneous hard material – AISI H13 steel – with martensitic matrix was $460.8 \pm 1.5 \text{ HV}_{30}$. Nevertheless, as can be seen in **Table 5**, the final values of the normal loads were modified seeing that the preliminary results (SERIACOPI et al., 2016) pointed out a divergence with Hokkirigawa; Kato; Li (1988). This analysis is discussed together with the results herein;

- Scratch length and scratching linear velocity were utilized as numerical model inputs, and their values are presented in **Table 5**;

- To prevent slab translational movements in the normal direction to the support planes throughout the scratching, roller supports were provided to the laterals and the base of the slab;

- The matrix and the second phase particles were assumed to have perfectly bonded interfaces (perfect interface);

- Regarding the abrasive-slab contact definitions, the “node-to-surface” was selected with null initial Coulomb friction coefficient. That approach is related to the “surface-to-surface” selection (contact pairs), which is restricted to the contact between two surfaces, requiring the definition of the nodes and/or elements of each interacting pair (interface) (SIMULIA, 2016). The contact occurs by penalty and a certain penetration of nodes can be verified to promote the convergence;

Figure 33 - Predicted conditions in terms of abrasive micro-mechanisms from the literature of the evaluated homogeneous materials.

Reference: Adapted from (HOKKIRIGAWA; KATO; LI, 1988).

Table 6 - Degree of penetration – eq. (7) – calculated for the normal loadings, for which the abrasive mechanisms transitions were estimated. The tip radius of the abrasive particle was 5 μm .

Material	Normal Load (mN)	D _p
Soft	5	0.13
	10	0.19
	30	0.35
	50	0.51
Hard	10	0.12
	20	0.17
	50	0.29
	100	0.45

- Concerning mechanical properties, numerical models were fed with data attained by means of experimental tests and the literature, as shown in **Table 7**. The details about these values used in numerical simulations are in **item 4.2.2 Mechanical properties of the steels and their phases**;

- The properties of the ductile damage, with element deletion, was applied to the material removal modelling for matrices – hard (martensitic) and soft (austenitic) - **Table 7**. As previously described, the damage nucleation of this model is designated from fracture strain as a function of stress triaxiality, for distinct strain rate levels. Likewise, the damage evolution is specified in terms of the fracture energy or energy release rate. On the other hand, brittle damage models were assigned to the second phase particles with element deletion (**Table 7**). In the latter case, the fracture

toughness, and the failure stress and strain are the main input parameters of the simulations. The MnS precipitates taken as soft particles, when compared to the TiN hard particles presented in austenitic stainless steels, were predicted to be plastically deformed along with the soft matrix, in accordance with preliminary experimental analyses (SERIACOPI et al., 2016). Consequently, there are no damage definitions prescribed for the MnS. In item **4.2.3 Damage properties of the steels and their phases**, the method for obtaining the results of the properties of damage onset and damage evolution of the phases are discussed.

Table 7 – General grades of the materials employed in the numerical modelling and their allocation in terms of mechanical and damage properties.

Material	Mechanical Behaviour	Elastic Properties	Plastic Properties	Damage Model
Hard and Homogeneous	Elastic-Plastic	Tensile Test	Tensile Test (von Mises)	Ductile (Tensile Test with notch)
Hard and Heterogeneous with hard precipitates	Matrix - Elastic-Plastic Precipitate - Elastic	Matrix - Tensile Test Precipitate - Nanoindentation	Matrix - Tensile Test (von Mises)	Matrix - Ductile (Tensile Test with notch) Precipitate – Brittle (Literature)
Soft and Homogeneous	Elastic-Plastic	Nanoindentation	Literature (Hollomon)	Ductile (Literature)
Soft and Heterogeneous with soft precipitates	Matrix - Elastic-Plastic Precipitate – Elastic-Perfectly Plastic	Matrix and precipitate - Nanoindentation	Matrix - Literature (Hollomon) Precipitate - Nanoindentation	Matrix - Ductile (Literature)
Soft and Heterogeneous with hard precipitates	Matrix - Elastic-Plastic Precipitate - Elastic	Matrix and precipitate - Nanoindentation	Matrix - Literature (Hollomon)	Matrix - Ductile (Literature) Precipitate – Brittle (Literature)

As noted in **Figure 34**, a scheme with the inputs and outputs of the micro-scratch test numerical model is proposed. Concerning the outputs, the major data are: apparent COF, depth of penetration, removed volume (material removal), specific cutting energy and characterization of the abrasive mechanisms from micro-ploughing to micro-cutting.

Figure 34 – Framework containing the steps related to the study of the single abrasive effect over metallic alloys in micro-scale.

4.1.2 Validation of the numerical model: experimental conditions designed for the scratch tests

Experimental scratch tests suitable to reproduce the single abrasive event were important to validate the numerical simulations. TI-950 triboindenter from Hysitron Inc. (**Figure 35a**) was applied to conduct the tests, based on the constant normal load setup, in which a cono-spherical diamond abrasive was used with $\sim 5\ \mu\text{m}$ of tip radius and $\sim 60^\circ$ of apex angle (**Figure 35b**). The high load module of the device, which reaches a maximum normal load of 2 N, allowed the signal acquisition of the forces and displacements in the 3D Cartesian system.

Figure 35 – Experimental apparatus in which the scratch tests were carried out, and the mechanical property measures by nanoindentation technique – TI-950 Triboindenter (a-). The geometry of the abrasive, which was used throughout the scratch tests in micro-scale (b-).

In accordance with **Figure 36**, the scratch test program consisted of the following stages: (i-) surface profilometry; (ii-) indentation by the imposed normal load; (iii-) scratching by using this constant normal load; and (iv-) unloading by disengaging the abrasive from the material surface. Scratch tests were performed on the AISI 303, 304 and 310 austenitic stainless steels, which formed the group of soft materials; on the AISI H13 tool steel as well as an alloy with martensitic matrix and NbC precipitates, which consisted in the group of hard materials. As recommended by the ASTM Standard G171 – 03 (AMERICAN SOCIETY FOR TESTING AND MATERIALS, 2009), three replicas were carried out for each normal load condition, with spacing of 50 and 100 μm between scratches to prevent the overlap of the abrasion effects.

Figure 36 – Schematic graph of the steps of the scratch tests in micro-scale, considering a constant normal load of 100 mN during the scratching stage.

The experimental parameters applied to validate the numerical model and to cover all the abrasive mechanisms are summarized in **Table 8**.

Table 8 – Setup of the experimental tests selected to evaluate different materials and the abrasive micro-mechanisms acting on them.

Material	Surface Finishing	Normal Load (mN)	Scratch Length (μm)	Scratching Speed ($\mu\text{m s}^{-1}$)	Data Acquisition Rate (Hz)
Homogeneous and soft	Mechanical and electrolytic polishing	40, 50, 60, 70, 80 and 100	400	10	200
Heterogeneous and soft with soft precipitates			60		
Heterogeneous and soft with hard precipitates	Mechanical polishing				
Homogeneous and hard	Mechanical polishing	50, 100, 150, 200, 400 and 500	400	15	200
Heterogeneous and hard with hard precipitates					

The most relevant outputs of the scratch tests were designated as follows: (1) the tangential load, which allows determining the apparent COF by using its ratio with the constant normal load (MEZLINI et al., 2005); and (2) the depth of penetration. The

removed volume and specific energy were calculated by the formulation described in items **3.1.4 Specific energy (E_{sp}) and material removal factor due to abrasion (fab) as indicators of the transition of the abrasive micro-mechanism** and **3.3 MATERIAL REMOVAL – CALCULATION METHOD**. Experimental results are compared to the numerical results.

The characterization of the 3D surface topography of materials was conducted after the scratch tests. From that evaluation, the depth of penetration with elastic recovery and scratch width (between pile-up peaks and at the baseline) were obtained, in which they can be considered indicative parameters of the material removal due to the abrasion (KOBICK; KLAUS; STREET, 2011; FRANCO; SINATORA, 2017). The technique that fulfils this purpose consisted of the Coherence Correlation Interferometry using the CCI-MP device from Taylor Hobson®. The treatment of the surface topography was carried out by using the methodology reported in Franco (2015). The settings configured for those measurements were: lens of 50X; view field (x-y) corresponding to 0.338 x 0.338 (mm); pixel size of 0.33 μm ; and optical resolution of 0.6 μm .

4.2 MATERIALS

The numerical modelling by Finite Element Method developed in the present research can predict the tribological responses of material groups, especially in terms of the material removal arising from the abrasive mechanisms. As previously mentioned, the material and its properties are model inputs (**Figure 36**). In addition, the distinction between homogeneous and heterogeneous materials is mandatory for studying the micro-scale. To simplify the model, the materials were considered heterogeneous owing to the presence of their second phase particles. Even though the abrasive is sensitive to the grains of some studied materials due to its dimensions, the modelling of distinct crystallographic orientation was not considered. For the stainless steels, the results are discussed by analysing the major experimental findings in item **5.6.2 Crystallographic orientation**.

The materials assessed during the scratching were combined into five classes:

(i-) homogeneous hard material, which consists of the AISI H13 hot work tool steel, as-forged, with martensitic matrix and nominal chemical composition displayed in **Table 9**;

Table 9 – Chemical composition (%wt.) of the tool steel and austenitic stainless steels used in the present work.

Steel	%C	%Mn	%Si	%Cr	%Ni	%P	%S	%Mo	%V
AISI H13 ⁽¹⁾	0.32-0.45	0.20-0.50	0.8-1.2	4.75-5.50	0.30 max.	-	-	1.70-1.75	0.80-1.20
AISI 304 ⁽²⁾	0.055	1.80	0.58	18.10	8.54	0.037	0.03	-	-
AISI 303 ⁽²⁾	0.050	1.88	0.48	17.20	8.21	0.036	0.20	-	-
AISI 310 ⁽²⁾	0.069	2.00	0.62	24.2	19.6	0.036	0.019	-	-

References: ⁽¹⁾(ASM INTERNATIONAL., 1990) and ⁽²⁾(SOUZA, 2006).

(ii-) heterogeneous hard material, which is a cast alloy manufactured at the Institute for Technological Research (IPT). It is a high-speed steel with martensitic matrix, having mechanical properties similar to the AISI H13 steel, and hard second phase particles of the MC type, rich in Nb. The experimental characterizations of these specimens, available in **ANNEX B** and in Tertuliano et al. (2017), indicated a chemical composition range of the carbides of the heterogeneous material microstructure. The aspects verified were: a shell (light part) rich in NbC; and a core (dark part) rich in Ti_x-Nb_{1-x}-C. Moreover, the carbides showed different mechanical behaviours under abrasion. Based on the occurrence of these carbides in this heterogeneous material,

the effect of their mechanical properties was included in the numerical simulation of scratch tests (**Figure 32a**);

(iii-) homogeneous soft material, which consists of the AISI 304 austenitic stainless steel as-annealed;

(iv-) heterogeneous soft material 1 (with soft particles) is the AISI 303 stainless steel, which presents an austenitic matrix - mechanical behaviour similar to the AISI 304 steel - and soft second phase particles of MnS;

(v-) heterogeneous soft material 2 (with hard particles) is the AISI 310 stainless steel, whose matrix is also equivalent to the AISI 304 steel and hard precipitates of TiN can be highlighted.

The chemical compositions (%wt.) of the AISI 303, 304 and 310 are presented in **Table 9**. These measurements were taken from bars with 25 mm diameter and the analysis were displayed elsewhere (SOUZA, 2006). These materials correspond to the same material batch of the present work.

The AISI H13 steel – homogeneous hard material – is often employed to manufacture tools for hot forming, owing to its good wear and fatigue resistances and high level of red hardness (ASM INTERNATIONAL., 1990). By contrast, soft materials were chosen to expand the analysis field for the abrasive manufacturing processes. These materials have a considerable corrosion resistance due their Cr content, which creates a protective oxide film layer, and the substantial presence of Ni responsible for keeping the austenite at room temperature (PADILHA; RIOS, 2002).

Concerning the stainless steels, one of the main differences between the AISI 303 and 304 steels is the S content (**Table 9**). The high sulphur content propitiates manganese sulphides dispersed in the 303 steel austenitic matrix, which enhance its machinability. Additionally, high contents of Ni and Cr (**Table 9**), when compared to the others, ensure a better high-temperature corrosion resistance of the AISI 310 steel (PADILHA; GUEDES, 1994). The TiN volume fraction of the 310 steel is very low; Ti and N contents were not characterized by Souza (2006) for this material.

A brief description of the materials and some simplifications evaluated here can be observed in **Table 10** to facilitate further analyses about their tribological responses.

Table 10 – An overview of the materials and their features studied in this work.

Simplifying Assumption	Material	Hardness
Homogeneous Soft Matrix	AISI 304 steel	Soft Matrix Hardness = H_{SM}
Heterogeneous Soft Matrix with Soft Particles	AISI 303 steel: (Matrix: AISI 304 steel Precipitates: MnS)	Second Phase Hardness ~ H_{SM}
Heterogeneous Soft Matrix with <u>Hard</u> Particles	AISI 310 steel: (Matrix: AISI 304 steel Precipitates: TiN)	Second Phase Hardness ~ $6H_{SM}$
Homogeneous <u>Hard</u> Matrix	AISI H13 steel	Hard Matrix Hardness ~ $2H_{SM}$
Heterogeneous <u>Hard</u> Matrix with <u>Hard</u> Particles	Matrix: AISI H13 steel Precipitates: Carbides rich in Nb	Second Phase Hardness ~ $10H_{SM}$

4.2.1 Specimen preparation and microstructural characterization

Specimens of the hard materials were obtained from the bars and cast billets, not presenting considerable differences between their cross and longitudinal sections. Conversely, specimens of the soft material were withdrawn from the longitudinal section of the bar of the stainless steels. Two procedures were carried out for the AISI 303 and 304 steels analysis: (i-) samples were metallographically prepared using mechanical polishing; and (ii-) samples were metallographically prepared using electrolytic polishing. These distinct types of surface finishing were conducted to evaluate the influence of the deformed layer (surface integrity) developed during the mechanical preparation and removed due to the electrolytic polishing.

The specimens mechanically polished were cut using the Isomet-5000 Precision Cutter (Buehler); afterwards, they were mounted by SimpliMet™ 3000 Mounting Press (Buehler); and were submitted to the sequence of automatic polishing listed in **Table 11**, using the TegraPol-25 polisher from Struers®.

Table 11 – Metallographic preparation procedure by mechanically polishing the specimens.

Material	Grinding	Intermediate	Polishing		
Soft	Water: SiC, #220 mesh (4 min)	MD-Largo: Diamond, 9 μ m (5 min)	MD-Mol: Diamond, 3 μ m (10 min)	-	MD-Chem/OP-S: Colloidal silica, 0.04 μ m (4 min)
Hard	Water: SiC, #220 mesh (2 min)	MD-Largo: Diamond, 9 μ m (5 min)	MD-Mol: Diamond, 3 μ m (6 min)	MD-Nap: Diamond, 1 μ m (4 min)	MD-Chem/OP-S: Colloidal silica, 0.04 μ m (15 min)

To obtain the surface finishing by electrolytic polishing, the specimens were cut (Isomet-5000), manually ground with SiC papers of #220, #320, #440 and #600 meshes, and electropolished using Pollectrol equipment from Struers®. This device was available at the Department of Metallurgical and Materials Engineering (PMT) of EPUSP. The electric current was 2 A, and the voltage applied was 40 V for 20 s to the AISI 304 steel; whereas, 40 V was applied during 40 s to the AISI 303; with exposure area of 1 cm² in both cases. The electrolyte solution was composed of 800 mL of ethyl alcohol, 140 mL of distilled water and 60 mL of perchloric acid, according to (ASM INTERNATIONAL., 2004). This solution pertains to Group I, nomenclature I-1, for compounds of HClO₄ and alcohol with or without organic additives, and for which voltages from 35 to 65 V and exposure times from 15 to 60 s are recommended for stainless steels.

The microstructures of the heterogeneous austenitic stainless steels, AISI 303 and 310, were acquired from the specimens mechanically polished, and further prepared using electrolytic etching with a solution corresponding to 10 g of oxalic acid and 100 mL of distilled water. The voltage applied was 6.0 V for 40 s to areas of 1 cm².

The microstructural characterization of the materials was performed by the Scanning Electron Microscopy (SEM) in the pre- and post-tests conditions. Other techniques related to microscopy were also important, such as: (a-) Energy-Dispersive X-Ray Spectroscopy (EDX) for the semi-quantitative measurement of the chemical composition, particularly to investigate the precipitates (hard and soft particles); and (b-) Electron Backscatter Diffraction (EBSD) to obtain a map of the crystallographic orientations. Therefore, it made it possible to differentiate grains in the stainless steels and to identify a possible strain-induced martensite formation, which can occur due to the plastic deformation caused by the scratch tests. The pieces of equipment employed for this microstructural characterization were:

- the Jeol JSM 6010-LA SEM, with tungsten filament and coupled EDX. This device was available in the Surface Phenomena Laboratory (LFS), of the Department of Mechanical Engineering (PME) and the Department of Mechatronics Engineering and Mechanical Systems (PMR) of EPUSP;
- the FEI-Inspect F50 SEM, with field emission gun (FEG) filament and EBSD, available in the Electronic and Atomic Force Microscopy Laboratory (LabMicro) of the PMT of EPUSP.

4.2.2 Mechanical properties of the steels and their phases

Firstly, the mechanical characterization of the material phases was conducted using instrumented indentation tests by TI-950 triboindenter from Hysitron Inc. (**Figure 35a**). Load-displacement (P-h) curves are obtained in these tests. Results were analysed by specific algorithms, which provides instrumented hardness and reduced elastic modulus, for example. The area function of the indenter tip is calibrated using indentations in a fused quartz standard, whereas the calibration of the optical position is carried out in an electropolished single crystal of Al. Also, the triboindenter applies the area function previously determined by means of the algorithm described by Oliver; Pharr, (1992, 2004) to calculate the mechanical properties.

Secondly, other mechanical properties of the phases, e.g. yield stress and strain-hardening exponent, can be predicted utilizing data from the tensile tests and complementary literature.

Finally, the last part of the characterization of mechanical properties was concerned with the phase failure features. For the ductile phases, the tensile tests using notched specimens and numerical simulation (hybrid model) are important to obtain the curve of the fracture strain as a function of stress triaxiality, which indicates the damage initiation in the numerical modelling (BAO; WIERZBICKI, 2004; OH et al., 2007; PRANTL et al., 2013). Regarding the damage evolution, the fracture energy input is necessary. In turn, for the brittle phases, the fracture toughness is indicated; the literature presents well-established values for the materials studied.

4.2.2.1 Soft materials

The mechanical properties of the soft materials are inputs for the FEM simulation of the scratch test in micro-scale (**Figure 34**). Here, the following mechanical behaviours were assumed: (i-) homogeneous AISI 304 steel, with isotropic properties of the austenitic matrix; (ii-) heterogeneous AISI 303 steel (AISI 304 + MnS), which presents the matrix properties corresponding to the AISI 304 properties and second phase particles with MnS inclusions properties; and (iii-) heterogeneous AISI 310 steel (AISI 304 + TiN), with a matrix similar to that of AISI 304 steel and second phase particles of TiN properties.

The mechanical characterization of the stainless steels was conducted with procedures and objectives as follows, and the measurements were compared with the literature:

- Study of the deformed layer formation (surface integrity) due to the mechanical polishing: The Vickers micro-hardness was measured using 1, 10 and 30 kgf loads and VMT-7 durometer from Buehler. After that, Vickers micro-hardness measurements were performed using 50, 100, 500 and 2,000 gf loads, and using the MMT-3 Micro Durometer from Buehler. AISI 303 and AISI 304 were applied to develop this step because of their similar mechanical behaviours. They can be susceptible to the strain-induced martensite, though. It does not occur in the AISI 310 steel since this steel presents high austenite stability (RHODES; THOMPSON, 1977; ABREU et al., 2007);
- Characterization of the mechanical properties of the phases: as observed in **Table 12**, the experimental setup of the instrumented indentation on the stainless steels is divided as a function of load applied and testing times (dwell time). All the tests were conducted with a Berkovich diamond indenter. For the hardness measurements on the second phase particles, the Scanning Probe Microscopy (SPM) was utilized, in which a surface profilometry was performed to ensure precise positioning of the indentations. The set point of force for this profilometry was 2.0 μN , and the scan rate was 0.25 Hz.

Table 12 – Experimental conditions of the nanoindentation tests for obtaining the mechanical properties of phases of the soft materials.

Phase	Loads (mN)	Loading Time (s)	Creep Time (s)	Unloading Time (s)	Number of replications
Austenitic Matrix	1, 5 and 10	5	5	5	5x5 arrays for each condition (Total: 75)
MnS	1, 5, 7 and 10	5	5	5	4 for each condition (Total: 16)
TiN	1, 5 and 10	5	5	5	4 for each condition (Total: 12)

Based on the reduced elastic modulus of the phases, the elastic modulus was calculated using eq. (10). The MnS and TiN were considered elastic-perfectly plastic and elastic, respectively, in the numerical model. Furthermore, the results were complemented and compared with the literature. **Table 13** shows the reference data

and starting points employed coupled with the experimental results to obtain the parameters of the Hollomon constitutive model (J.H. HOLLomon, 1945) for the elastic-plastic austenitic matrix.

Table 13 – Literature data of the mechanical properties used as base for the Hollomon model combined with the experimental results.

Phase	Elongation - ϵ_{total} (%)	Yield Stress - σ_y (MPa)	Ultimate Strength – UTS (MPa)
Austenitic matrix (AISI 304 steel) - annealed bar (longitudinal)	60	235	585

Reference: (ASM INTERNATIONAL., 1990)

The additional data used as the modelling inputs were: density and Poisson ratio of the phases (see **Table 14**).

Table 14 – Densities and Poisson ratios of the soft material phases – numerical model inputs.

Phase	Density (kg m^{-3})	Poisson Ratio	Reference
Austenitic matrix (AISI 304 steel) - annealed bar (longitudinal)	8,000	0.29	(ASM INTERNATIONAL., 1990)
MnS	4,000	0.25	(HAYNES, 2014)
TiN	5,400	0.25	(PIERSON, 1996a)

4.2.2.2 Hard materials

As described earlier, the hard materials were studied to evaluate the abrasion using scratch test models in micro-scale as follows:

(a-) the AISI H13 tool steel, which has a homogeneous martensitic matrix;

(b-) a heterogeneous alloy consisting of the martensitic matrix, with mechanical properties very similar to the AISI H13, and a NbC containing other elements such as Ti, which was considered the hard second phase particle.

The two materials were selected since they were characterized in a research project denominated “Advanced system for design of alloys applied to hot forming tools”, conducted in a collaboration between the LFS/EPUSP and IPT, financially supported by the Brazilian Development Bank (BNDES) and Gerdau. In view of this background, the mechanical properties of these alloys and their phases were characterized in the scope of that project, which was developed in 2012-2017. In the present thesis, the methodological procedures for the mechanical characterization are

briefly described; also, the results used as modelling inputs are listed and referenced to register the data.

Regarding the AISI H13 steel, the mechanical properties were measured by tensile tests of specimens dimensioned in accordance with the ASTM E8 Standard (AMERICAN SOCIETY FOR TESTING AND MATERIALS, 2016). The tests with 3 replications were carried out at room temperature, with extensometers and constant speed equal to 1 mm min^{-1} , in a Universal Testing Machine of 5585H model from Instron Co. The report of the tests, conducted at the Centre for Development and Characterization of Materials (CCDM) of the Federal University of Sao Carlos (UFSCar), are available in **ANNEX C**; the engineering stress-strain curves are exemplified there. The mean of the elastic modulus (E), elongation (ϵ_{total}), yield stress (σ_y) and ultimate tensile strength (UTS) of the AISI H13 steel/martensitic matrix are set out in **Table 15**. Additionally, the density and Poisson ratio were obtained in the literature for this material: $7,800 \text{ kg m}^{-3}$ and 0.3 (AFAZOV; RATCHEV; SEGAL, 2012), respectively.

Table 15 – AISI H13 steel mechanical properties applied as inputs of the scratching model of the hard materials.

Elastic Modulus - E (GPa)	Elongation - ϵ_{total} (%)	Yield Stress - σ_y (MPa)	Ultimate Strength – UTS (MPa)
207 ± 21	6.3 ± 0.5	$1,413 \pm 15$	$1,624 \pm 21$

Reference: (CCDM – DEMA UFSCAR, 2014).

The NbC - hard second phase particles dispersed in the martensitic matrix of the H13 steel - elastic modulus and the hardness at room temperature were obtained from Tertuliano et al. (2017). The measurements in sintered NbC were conducted by instrumented indentation (TI-950 Triboindenter-Hysitron). The load applied was 5 mN, using diamond Berkovich indenter, and Oliver-Pharr algorithm (OLIVER; PHARR, 1992) was utilized to calculate the mechanical properties. Moreover, the properties related to the ranges of compositions, since the carbide was composed of a core of $\text{Ti}_x\text{-Nb}_{1-x}\text{-C}$ (inner part of the carbide) and a shell composed of NbC (outer part of the carbide) (**ANNEX B**), are pointed out in **Table 16**.

Table 16 – Precipitate mechanical properties applied as inputs of the scratching model of the hard materials in micro-scale.

Phase	Elastic Modulus - E (GPa)	Indentation Hardness (GPa)	Poisson Ratio	Density (kg m ⁻³)
NbC - Shell	372 ± 2 ⁽¹⁾ 338-580 ⁽²⁾	24 ± 1 ⁽¹⁾	0.21 ⁽²⁾	7,790 ⁽²⁾
Ti _x -Nb _{1-x} -C - Core	466 ⁽³⁾	20 ± 10 ⁽⁴⁾	0.21 ⁽³⁾	5,606

References: ⁽¹⁾(TERTULIANO et al., 2017); ⁽²⁾(PIERSON, 1996b); ⁽³⁾(CHEN; ZHAO, 2007); ⁽⁴⁾(MAERKY et al., 1996)

The major data inputs of the mechanical properties (**Figure 34**) of the hard material phases were exposed here. The NbC with composition variations was considered elastic, while the martensitic matrix was accounted as elastic-plastic with the von Mises plasticity criterion.

Part of the information provided here is re-established in a result item (**5.2.3 Comparison of the mechanical properties of the soft and hard matrices**), aiming to calculate the strain-hardening exponent (n) and the strength coefficient (K_s) using ASTM Standard E646 (AMERICAN SOCIETY FOR TESTING AND MATERIALS, 2000). Based on the true stress-strain curve, the method employed basically consisted in performing a linear regression of the log (stress σ) x log (strain ε) graph for the plastic regime, and to obtain the n-value as the slope of the fitted curve. In turn, the K_s-value was calculated as given by eq. (27):

$$K_s = \frac{\sum_{i=1}^N \log \sigma_i - n \sum_{i=1}^N \log \varepsilon_i}{N} \quad (27)$$

(AMERICAN SOCIETY FOR
TESTING AND MATERIALS,
2000)

Where: N is the data number of the log (stress σ) x log (strain ε) curve.

The estimated n- and K-values for the AISI H13 and 304 steels were necessary because such parameters were fundamental for calculating the deformed volume, accumulated as pile-up throughout the micro-scratch with the cono-spherical tip (GAO; JING; SUBHASH, 2006; NIKAS, 2015).

4.2.3 Damage properties of the steels and their phases

Damage models of the material phases were incorporated into the numerical modelling to allow studying the abrasive mechanism effects on the homogeneous and heterogeneous materials.

The failure of the materials is often monitored in the Abaqus®/Explicit finite element software by the scalar stiffness degradation (SDEG), i.e. the scalar damage, which varies between 0 and 1. The abrasion model developed here worked with element deletion and the element was thus deactivated by the system; its stiffness was annulled when the SDEG output corresponded to the unit.

As regards the austenitic stainless steels (soft materials), the damage properties considered for the austenitic matrix and TiN were acquired from the literature.

After some approaches initially developed, the Ductile Model was chosen to describe the damage nucleation of the austenitic matrix, applying a composition of the curves reported in Dzugan et al. (2012) and Kim et al. (2013), since:

(a-) negative stress triaxialities (compression) were not covered in Kim et al. (2013); only the triaxialities from 0.5 to 3.0 were evaluated;

(b-) only the stress triaxiality of -0.13 was selected from Dzugan et al. (2012) work because of the curve fit designed by the authors was based on hybrid modelling, which resulted in a singularity impossible to be physically explained.

The curve of the fracture strain as a function of triaxiality, which specifies the ductile damage initiation for the austenitic matrix, can be observed in **Figure 37a**.

Figure 37 – Fracture strain as a function of stress triaxiality: information utilized to model the ductile damage on the austenitic (a-) and martensitic (b-) matrices, presented in the studied materials. The strain rate is 0.1 s^{-1} in both cases.

References: ⁽¹⁾ Adapted from (DZUGAN et al., 2012; KIM et al., 2013).

Regarding the AISI H13 steel (martensitic matrix of the heterogeneous hard material), the damage properties of a carbon steel were considered in the preliminary tests. Conversely, the numerical modelling of its tensile test, associated with its experimental tests (**ANNEX C**), allows obtaining a stress triaxiality of 0.6 corresponding to a fracture energy of 4%. For the same triaxiality conditions, the fracture strain of that carbon steel is 40 times as high as that of H13 steel. Hence, the fixed final curve of the damage nucleation of the H13 steel was extrapolated, as can be seen in **Figure 37b**.

The energy necessary to propagate the defect per area unit, which originates the material removal by abrasion, was the parameter designated in the simulation to specify the damage evolution of the ductile matrices. Anderson (2005) pointed out the G_f variable as the critical elastic energy release rate, which consists in a fracture toughness measurement, as stated in eq.(16). Therefore, there is a limitation of G_f employment to specify the failure for the ductile phases in Abaqus® software, since this input is more indicated for applications in the LEFM fields.

For the martensitic matrix, the numerical model showed a better convergence when simplified using $G_f = 0.5 \text{ Nmm}^{-1}$, which is indicated for carbon steels (SIMULIA, 2016). The G_f range reported in the literature for the H13 steel (WEAVER et al., 2011), from 3.6 to 11.3 Nmm^{-1} , resulted in convergence problems and excessive plastic deformation of this hard material, in which the micro-cutting occurrence was not predicted. Likewise, for the austenitic matrix, this parameter was fed into the simulation with the value of $G_f = 0.375 \text{ Nmm}^{-1}$, since its damage behaviour is similar to the Al alloy evaluated in Tkaya et al. (2008). Once again, the literature for the AISI 304 steel (BOYER; GALL, 1998) informed a value of about $G_f = 2.09 \text{ Nmm}^{-1}$ obtained from the Charpy test, which results in convergence problems in the numerical modelling.

The Brittle Failure model was assigned to the hard second phase particles, and literature data were used as inputs of the numerical simulation. **Table 17** shows the values of the parameters that play an important role to provide the nucleation and propagation of the damage of precipitates.

Table 17 – Inputs of the numerical model of scratching in micro-scale: failure properties of the brittle phase included in the heterogeneous hard material.

Phase	Fracture toughness (MPa m ^{1/2})	Critical energy release rate - (Nmm ⁻¹)	Failure stress - (GPa)	Failure strain	References
NbC (shell - Figure 32a)	3.0	0.023	4.75	0.012	(ACCHAR; CAIRO, 2006; CASELLAS et al., 2007)
Ti _x -Nb _{1-x} -C (core- Figure 32a)	3.6	0.027	5.70	0.012	(MAERKY et al., 1996; CHEN; ZHAO, 2007)
TiN (Figure 32c)	2.5	0.022	5.17	0.019	(DEMIRSKYI; AGRAWAL; RAGULYA, 2013)

5 RESULTS AND DISCUSSIONS

5.1 Microstructural characterization of the materials

The microstructures of the soft materials are displayed in **Figure 38**. Although the AISI 304 steel was considered homogeneous in the numerical model, crystallographic orientations of grains were characterized by EBSD (item **5.6.2 Crystallographic orientation**), as well as the annealing twins associated with the low stacking fault energy (SFE) of this material.

The hypothesis made considering the AISI 303 steel a heterogeneous material, when compared to the homogeneous 304 steel, is presented after the electrolytic etching. MnS elongated particles in the rolling direction are observed in the 303 steel microstructure (**Figure 38b**). The volume fraction of these inclusions was calculated by means of the ImageJ® software (IMAGEJ, 2015), and it corresponded to $(3.25 \pm 0.45) \%$. Further, the characterization using SEM/EDX of the AISI 303 specimen showed the presence of the sulphur and manganese (**Figure 38b**), which can be attributed to the manganese sulphide as second phase particles dispersed in the austenitic matrix. Souza (2006) conducted X-ray diffraction measurements on the AISI 303 steel bars, which were originating from the same batch of the material used in the present work, and it corroborated the MnS occurrence based on the obtained diffractograms.

As observed in **Figure 38c**, the etching disclosures the preferential orientation (texture) along the rolling direction of the AISI 310 steel. Precipitates can be seen, among them TiN, which was not detected using X-ray diffraction elsewhere (SOUZA, 2006), but they were identified indirectly using EDX, golden color (observed using optical microscopy) and for its square morphology. The scratches included one titanium nitride at least along their length, as confirmed by EDX analyses. **Figure 38c** exemplifies the micro-ploughing and micro-cutting conditions of this material treated as heterogeneous; the residual phase is found in both cases.

The microstructure of the homogeneous hard material – AISI H13 steel – is martensitic with the lath typical morphology (**Figure 39a**), as expected (ASM INTERNATIONAL., 2004). In turn, the heterogeneous hard material (**Figure 39b**) presents the casting defects (in black), which were not considered in the numerical simulation, and a volume fraction of the precipitates of $(7.6 \pm 2.8) \%$ was calculated by ImageJ® software.

Figure 38 – Characterization of the soft materials obtained by SEM and EDX: (a-) backscattered electrons (BSE) – material considered homogeneous in the simulation due to its not having second phase particles; (b-) secondary electrons (SE) – heterogeneous material 1, with austenitic matrix and manganese sulphide particles (S and Mn detected by EDX); and (c-) secondary electrons (SE) – heterogeneous material 2, with austenitic matrix and titanium nitride (yellow arrow). In the latter case, EDX analyses were carried out in the scratch to assure the presence of the second phase particles not only after the micro-ploughing, but also after the severe micro-cutting.

(a-) AISI 304 stainless steel

(b-) AISI 303 stainless steel

(To be continued)

Figure 38 – Characterization of the soft materials obtained by SEM and EDX: (a-) backscattered electrons (BSE) – material considered homogeneous in the simulation due to its not having second phase particles; (b-) secondary electrons (SE) – heterogeneous material 1, with austenitic matrix and manganese sulphide particles (S and Mn detected by EDX); and (c-) secondary electrons (SE) – heterogeneous material 2, with austenitic matrix and titanium nitride (yellow arrow). In the latter case, EDX analyses were carried out in the scratch to assure the presence of the second phase particles not only after the micro-ploughing, but also after the severe micro-cutting.

(c-) AISI 310 stainless steel

(Continuation)

Figure 39 – Hard materials used in the numerical simulations: (a-) homogeneous material: BSE/SEM images of the AISI H13 steel with and without metallographic etching; and (b-) heterogeneous material: BSE/SEM image of the cast alloy, without metallographic etching, which presents martensitic matrix and carbides as second phase particles.

(a-)

(1,000 X: Without etching, in which scratches and Berkovich indentations were carried out to obtain the image)

(5,000 X: With etching - Nital 2%, in which the martensitic matrix is noted)

(b-)

1,000 X: Carbides were highlighted on the right

5.2 Mechanical characterization of the materials: micro- and macroscopic levels

5.2.1 Soft materials - nanoindentation

The load-displacement curves obtained by nanoindentation of the matrix of the austenitic stainless steels are provided in **Figure 40a**. The Oliver-Pharr algorithm was applied to the curves shown in **Figure 40a** to calculate the reduced elastic modulus and indentation hardness of the studied materials (**Table 18**).

Regarding the MnS and TiN precipitates of the AISI 303 and 310, respectively, the mechanical properties results can be observed in **Table 18**. Once again, these data were calculated using the Oliver and Pharr formulation (OLIVER; PHARR, 1992), in which the load-displacements curves were the inputs (**Figure 40b,c**). The indentation positioning of the second phase particles (**Figure 41**) were defined using the Scanning Probe Microscopy (SPM) stage, with a piezoelectric scanner. As seen in **Table 18**, the MnS hardness may be equated with the austenitic matrix hardness and, then, it is considered a soft precipitate. Conversely, the TiN hardness is about six times the matrix hardness value and is thus considered as a hard precipitate in the numerical model.

Figure 40 – Load-displacement curves, resulting from the instrumented indentation, for the austenitic matrix and the second phase particles of the evaluated stainless steels.

(a-) Austenitic matrix (considered the same for the AISI 303, 304 and 310 steels)

(b-) MnS – second phase particles of the AISI 303 steel

(c-) TiN – second phase particles of the AISI 310 steel

Figure 41 – Images obtained in situ by means of SPM, exemplifying the indentations performed on the MnS (soft precipitate) and TiN (hard precipitate) by utilizing the force (μN) and topography (nm) gradients.

Figure 42 – 3D image of the MnS obtained by SPM, in which the pile-up formation can be noted with the applied load of 7 mN.

The SPM 3D image indicated the pile-up presence and, therefore, the real depth of contact is underestimated by the Oliver-Pharr algorithm. A scheme of the Berkovich indentations, in which this algorithm is valid, is displayed in **Figure 43(b,c)**. According to Hay (2009), the Oliver-Pharr algorithm would present overrated values of reduced elastic modulus and indentation hardness for the pile-up cases (**Figure 43a**). Consequently, the values obtained for the second phase particles have restriction as regards their wide employment.

Figure 43 – Schematic representation of the indentations resulting from Berkovich probe: (a-) pile-up; (b-) neither pile-up nor sink-in; (c-) sink-in; and (d-) excessive sink-in. The Oliver-Pharr algorithm is often valid for the (b-) and (c-) cases.

Reference: (HAY, 2009)

The elastic moduli of the matrix and precipitates of the soft materials are shown in **Table 18**. These results were calculated using eq. (10), which includes the reduced modulus and its correction based on the diamond indenter elastic modulus to attain the elastic modulus of the phases. The outcomes are consistent with the literature in terms of their orders of magnitude, as can be seen in **Table 19**.

Table 18 – Overview of the experimental mechanical properties of the austenitic matrix, MnS and TiN featured in the evaluated stainless steels.

Phase	Force (mN)	Reduced Modulus - E_r [GPa]	Elastic Modulus - E [GPa]	Hardness - H (GPa)	E/H
Austenitic Matrix	1	188 ± 10	206 ± 9	3.68 ± 0.13	56
	5	192 ± 7	211 ± 6	2.76 ± 0.16	76
	10	193 ± 9	212 ± 8	2.47 ± 0.11	86
MnS	1	126 ± 8	133 ± 8	3.26 ± 0.27	41
	5	121 ± 6	127 ± 6	2.89 ± 0.16	44
	7	126 ± 5	133 ± 4	2.67 ± 0.16	50
	10	128 ± 5	135 ± 5	2.726 ± 0.011	50
TiN	1	232 ± 5	272 ± 5	20.23 ± 0.18	13
	5	127 ± 2	133 ± 2	17.3 ± 0.9	8
	10	103 ± 6	106 ± 6	15.5 ± 1.4	7

Table 19 – Mechanical properties reported in the literature for the phases of the analysed soft materials.

Phase	Elastic Modulus - E [GPa]	Hardness - H (GPa)	References
Matrix – AISI 304 steel	192	-	(KHUTIA; DEY; HASSAN, 2015)
MnS	120.4	4.08	(LIU et al., 2017)
TiN	251	18-21	(PIERSON, 1996a)

For the mechanical properties (E and H) listed in **Table 18**, the One-Way Analysis of Variance (ANOVA) was performed and, **Table 20** shows the mean values of the austenitic matrix hardness, which were statically different at the 0.05 level, since the F-value is higher than the F-critical. However, the mean values of the matrix elastic modulus were statically equal for all the loads applied at the same level. These differences can be justified because of the effect of the distinct crystallographic orientation of the studied stainless steels, as will be discussed in depth further on. Similar results were reported in Machado et al. (2017) for the Hadfield austenitic steel. Another aspect to be highlighted is the deformed layer influence, which is formed throughout the mechanical polishing of these materials (SERIACOPI et al., 2016).

Table 20 – One-Way ANOVA for the austenitic matrix hardness of the analysed stainless steels. SQ = sum of squares; DF = degrees of freedom; MQ = mean square; F and F_{critical} are parameters (F-distribution) that indicate in case the null hypothesis is sustained at the 5% significance level.

Group	Count	Sum	Average	Variance
1 mN	20	73.3	3.68	0.018
5 mN	20	58.2	2.76	0.025
10 mN	20	49.4	2.47	0.010

Source of Variation	SQ	DF	MS	F	F _{critical}
Between groups	14.7	2	7.35	418.9	3.2
Within groups	1.0	57	0.02		
Total	15.7	59			

The mean values of the MnS elastic modulus as well as the mean values of its hardness (**Table 18**) did not vary, considering the 5, 7 and 10 mN loads. However, by including the 1 mN load into the One-Way ANOVA, the mean of the hardness was significantly different (**Table 21**), which corroborates the deformed layer existence.

Table 21 – One-Way ANOVA for the MnS precipitates of the AISI 303 stainless steel. SQ = sum of squares; DF = degrees of freedom; MQ = mean square; F and $F_{critical}$ are parameters (F-distribution) that indicate whether the null hypothesis is sustained at the 5% significance level.

Group	Count	Sum	Average	Variance
1 mN	4	13.0	3.26	0.10
5 mN	4	11.0	2.89	0.03
7 mN	4	10.7	2.67	0.03
10 mN	4	10.9	2.726	0.00014

Source of Variation	Sum of Squares	Degree of Freedom	Mean Square	F	F critical
Between groups	0.9	3	0.30	7.67	3.5
Within groups	0.5	12	0.04		
Total	1.4	15			

The mean of the elastic modulus and hardness of the TiN particles (**Table 18**) were statically different with the applied load and, thus, the null hypothesis was rejected in both cases. The F-value was 1040.5 whilst the $F_{critical}$ corresponded to 4.2 in terms of elastic modulus. Similarly, the F-value was 21.7 whereas the $F_{critical}$ was equal to 4.2 in view of the phase hardness. These results may be assigned to the irregular (non-homogeneous) surface of the hard particle, as observed in **Figure 41**.

A selection criterion of the mechanical properties to be used as numerical model inputs was elaborated since their values vary with the load in most cases. As stated in Pintaude (2013), the lower value of the E/H results in a low cutting efficiency. Hence, this parameter was utilized to choose the mechanical properties, in particular the elastic modulus and the hardness of the phases to be scratched. The following hypotheses were adopted as assumptions based on the low values of considered normal load, and they correspond to the highlighted blue lines in **Table 18**:

(a-) the austenitic matrix and soft precipitates (MnS) facilitate the material removal; then, a higher E/H ratio should be adopted for these phases;

(b-) on the other hand, the hard precipitates (TiN) tend to reduce the material removal effect; as a result, the E/H is selected as a function of minimum value to hinder the removed volume (cutting).

5.2.2 Soft materials – conventional hardness

The average results of the Vickers hardness of the AISI 303 and 304 austenitic stainless steels are set out in **Figure 44**. From the graphs, the dispersion measure is not related to the sample standard deviation, but it is the standard error of the mean, which is the ratio between the sample standard deviation and the square root of the sample number. The mean uncertainty is the dispersion measure recommended when two or more means are graphically compared (GOODIER, 2007).

Figure 44 – Conventional hardness: mean values and their dispersions for the Vickers micro- and macro-hardness of the AISI 303 and 304 steels.

The hardness values of the annealed AISI 304 steel were compared with Barbosa (2014), in which the Vickers micro-hardness measure was carried out with 50 gf, while the Vickers macro-hardness was performed with 30 kgf load. For the same conditions, the micro-hardness result of the AISI 304 was $(188.5 \pm 5.2) \text{ HV}_{0.05}$ (**Figure 44d**), in which no significant difference was detected with Barbosa (2014) that found

(207.7 ± 16.7) $HV_{0.05}$. In terms of macro-hardness, the value measured here was (176.8 ± 3.2) HV_{30} (**Figure 44c**), which is in accordance with Barbosa (2014), which provided a macro-hardness of (179.9 ± 11.6) HV_{30} for the 304 steel.

To verify whether the Vickers hardness (conventional) measures for the AISI 303 and 304 were independent of the applied load, i.e. if their values were statically equal, the One-Way ANOVA was conducted by means of the procedure described by Box; Hunter; Hunter (2005). The ANOVA results are summarized in **Table 22**, **Table 23**, **Table 24** and **Table 25**.

The means of the AISI 303 steel macro-hardness (**Table 22**) are significantly different since $F > F_{critical}$ using the hypothesis test. This response matches **Figure 44a**. A possible explanation for the macro-hardness variation with the load is the MnS influence on this steel (**Figure 45**). When the second phase particle does not effectively affect the measurement, the macro-hardness does not present variation as a function of applied load, as can be confirmed by the results of the AISI 304 steel in **Table 23** and **Figure 44c**. In the latter case, the null hypothesis is sustained ($F < F_{critical}$).

The micro-hardness means of the AISI 303 steel, applying different loads, are equal - 0.05 level ($F < F_{critical}$). Even though the average value of the hardness for the 50 gf is different from the others, the mean uncertainty is also higher when compared to the other loads, resulting to be statically equal.

Table 22 – One-Way ANOVA applied to the AISI 303 stainless steel macro-hardness. SQ = sum of squares; DF = degrees of freedom; MQ = mean square; F and $F_{critical}$ are parameters (F-distribution) that indicate if the null hypothesis is sustained at the 5% significance level.

Group	Count	Sum	Average	Variance
1 kgf	10	1,243.60	124.36	20.46
10 kgf	10	1,679.54	167.95	8.50
30 kgf	10	1,831.75	183.17	352.76

Source of Variation	Sum of Squares	Degree of Freedom	Mean Square	F	F critical
Between groups	18,637.57	2	9,318.79	73.24	3.35
Within groups	3,435.46	27	127.24		
Total	22,073.03	29			

Table 23 – One-Way ANOVA applied to the AISI 304 stainless steel macro-hardness. SQ = sum of squares; DF = degrees of freedom; MQ = mean square; F and $F_{critical}$ are parameters (F-distribution) that indicate if the null hypothesis is sustained at the 5% significance level.

Group	Count	Sum	Average	Variance
1 kgf	4	721.75	180.44	56.66
10 kgf	9	1,606.44	178.49	270.47
30 kgf	11	1,936.30	176.03	97.06

Source of Variation	Sum of Squares	Degree of Freedom	Mean Square	F	F critical
Between groups	66.42	2	33.21	0.21	3.47
Within groups	3,304.41	21	157.35		
Total	3,370.83	23			

Table 24 – One-Way ANOVA applied to the AISI 303 stainless steel micro-hardness. SQ = sum of squares; DF = degrees of freedom; MQ = mean square; F and $F_{critical}$ are parameters (F-distribution) that indicate if the null hypothesis is sustained at the 5% significance level.

Group	Count	Sum	Average	Variance
50 gf	5	1,019.47	203.89	130.84
100 gf	5	1,029.75	205.95	6.43
500 gf	5	933.52	186.70	644.32
2.000 gf	5	1,059.10	211.82	27.79

Source of Variation	Sum of Squares	Degree of Freedom	Mean Square	F	F critical
Between groups	1,747.86	3	582.62	2.88	3.24
Within groups	3,237.54	16	202.35		
Total	4,985.30	19			

Table 25 – One-Way ANOVA applied to the AISI 304 stainless steel micro-hardness. SQ = sum of squares; DF = degrees of freedom; MQ = mean square; F and $F_{critical}$ are parameters (F-distribution) that indicate if the null hypothesis is sustained at the 5% significance level.

Group	Count	Sum	Average	Variance
50 gf	5	942.34	188.47	132.91
100 gf	5	895.90	179.18	176.59
500 gf	5	853.56	170.71	20.28
2.000 gf	5	859.13	171.83	32.44

Source of Variation	Sum of Squares	Degree of Freedom	Mean Square	F	F critical
Between groups	1,006.77	3	335.59	3.71	3.24
Within groups	1,448.86	16	90.55		
Total	2,455.63	19			

Figure 45 – SEM images (secondary electrons) of the Vickers macro-hardness marks on the AISI 303 steel obtained from different applied loads.

In contrast, for the AISI 304 steel micro-hardness case, the means of the population are significantly distinct with the load ($F > F_{critical}$) as shown in **Table 25**. Because the measurements were carried out on the mechanically polished specimens, the deformed layer can be supposed to affect the results. Two hardness regions can be distinguished in **Figure 44d**: (i-) from 50 to 100 gf; and (ii-) from 500 to 2,000 gf. A statistical analysis for comparison of means (T-test) was set to verify the occurrence of these two groups.

As noted in **Figure 44d**, the 304 steel micro-hardness means of the second group (500 – 2,000 gf) are equal. For this reason, the T-test was applied to the first group, following the methodology reported in Box; Hunter; Hunter (2005). The resulting t-value was 0.52; in turn, the two-sided critical t-value was 3.18, i.e. the means of the hardness do not present a significative statistical difference ($t < t_{critical}$). Albeit equal within each group, the micro-hardness means of AISI 304 steel differ broadly. Thus, the hypothesis of the deformed layer to be between 100 and 500 gf is reasonable. In addition, an estimated thickness of the deformed layer can be determined using eq. (28) for Vickers indenter (YOVANOVICH, 2006).

$$h = \frac{d_V}{2\sqrt{2} \cdot \tan \frac{\gamma}{2}} \quad (28)$$

In which: h is the penetration depth; d_V is the average of the diagonals; and γ is the Vickers indenter angle (136°).

The deformed layer estimated by applying a 100 gf load would provide a minimum value because the transition is between 100 and 500 gf. Also, the diagonal measures utilized for calculating the depth were performed after the specimen unloading in which the elastic recovery occurred and, then, the result would be overestimated. The combination of these conditions led to predicting the thickness based on 100 gf load. **Table 26** presents the results of the penetration depth corresponding to the five measures under this load conducted on the AISI 304 steel. To conclude, the maximum thickness value of the deformed layer is $h = (4.60 \pm 0.15) \mu\text{m}$.

Table 26 – Depth of penetration (h) calculated from the Vickers micro-hardness measurements of the AISI 304 steel.

AISI 304 - 100 gf - Measurements	d_V (μm)	h (μm)
1	33.78	4.82
2	31.85	4.55
3	32.93	4.70
4	31.92	4.56
5	30.62	4.37

5.2.3 Comparison of the mechanical properties of the soft and hard matrices

The calculation of the strength coefficient (K_s) and strain-hardening exponent (n) of the soft and hard matrices resulted in the values depicted in **Table 27**.

Table 27 – Constants of the Hollomon constitutive equation calculated for the ductile matrices.

Material	K_s	n
Soft matrix – AISI 304 steel	787	0.58
Hard matrix – AISI H13	1881	0.03

The validation of constants displayed in **Table 27** can be reached by means of complementary methods.

(1) AISI H13 steel (tensile test x modelling using Hollomon plasticity equation):
As observed in **Figure 46**, the data plotted based on the Hollomon model for the AISI H13 steel suitably reproduce the experimental results (both values and behaviour mechanical) derived from tensile tests of this hard material.

Figure 46 – Comparative graph of the engineering stress-strain curves obtained from the experimental tensile tests (**ANNEX C**) and the calculation of the Hollomon model constants added to the elastic part previously established.

(2) AISI 304 steel (hardness calculation x nanoindentation value): Another method to validate the results obtained for the plasticity of the austenitic and martensitic matrices consists in applying eq.(29) (NIKAS, 2015):

$$\sigma_y = \frac{H}{3 \cdot 10^n} \quad (29)$$

Replacing the yield stress values of the AISI 304 (**Table 13**) and AISI H13 (**Table 15**) steels and their respective strain-hardening coefficients (**Table 27**) in eq.(29), the following hardness values are attained:

- 304 stainless steel: $H = 2.68 \text{ GPa}$;
- H13 tool steel: $H = 4.54 \text{ GPa}$.

It can be concluded that the calculated hardness of the AISI 304 steel through the n-exponent of Hollomon model is coherent with the values measured using instrumented indentation, which was equal to $(2.47 \pm 0.11) \text{ GPa}$ (**Table 18**).

5.3 Damage properties obtained via scratch tests: failure evaluations by stress triaxiality studies

Since the elaborated numerical model has been validated, relevant data can be acquired to propose new research lines for the metallic alloy design submitted to abrasion.

As observed in **Figure 47**, the stress triaxialities are compressive along the scratched surface of the homogeneous hard material. On the other hand, the stress triaxialities become tensile on the heterogeneous hard material surface after the abrasive pass through the carbides, which may promote the cutting/detachment of the second phase particles depending on the normal load. Moreover, some points on the wear track present null values of the stress triaxiality. These positions can favor the shear failure of the martensitic matrix (BAO; WIERZBICKI, 2004) during the scratch test in micro-scale.

Figure 47 – Stress triaxiality along the scratch length for the hard materials: comparison between homogeneous and heterogeneous cases.

A more detailed characterization focused on the superficial failure study of the homogeneous hard material allows noting that:

- for the normal load of 50 mN, the failure mode is predicted as shear with null value of the stress triaxiality and the fracture strain of about 2.5. In this condition, although the micro-ploughing is dominant along the scratch length, some regions showed chip formation as indicated by the micrographs displayed in **Figure 48**;

- for the normal load of 100 mN, the fracture strain remains at 2.5, but the average stress triaxiality is modified to a more compressive mode, originating a combination between shear and ductile failures, with micro-voids formation (**Figure 48**);

- in the latter cases, which correspond to the normal loads of 150, 300 and 400 mN, the average stress triaxialities grow out of the vertical axis (on the right and on the left), including high strain values, e.g. ~ 3.75 for 400 mN, and resulting in prevailing ductile failures (**Figure 48**).

Albeit qualitative, the proposal of the failure approach by using the stress triaxiality allowed conducting a case study based on the evaluated hard materials, and it creates a preliminary procedure for future pursuits involving other material failure properties by abrasive processes.

Figure 48 – Plastic strain at fracture as a function of stress triaxiality for the homogeneous hard material (AISI H13 tool steel).

(To be continued)

Figure 48 – Plastic strain at fracture as a function of stress triaxiality for the homogeneous hard material (AISI H13 tool steel).

(Continuation)

5.4 General comparison between numerical and experimental results of the scratch test in micro-scale

5.4.1 Apparent coefficient of friction (Apparent COF)

5.4.1.1 Homogeneous materials

Figure 49 shows the comparison between the numerical and experimental responses of the average values of apparent COF as a function of applied normal load, considering the contact pairs: abrasive-homogeneous materials (soft and hard matrices). A considerable difference between the numerical and experimental findings occurs. The verified lack of fit with numerical data to experimental is more pronounced for 50 mN applied to the soft materials and for 100 mN applied to the hard materials. A possible explanation for this behaviour might be the transition of the dominant abrasive micro-mechanisms, which will be discussed later considering the load influence.

Figure 49 – Numerical and experimental results of the average apparent coefficient of friction (COF) as a function of applied normal load: homogeneous materials.

The adhesion effect on the friction coefficient is one of the main reasons to justify the numerical and experimental divergences, observed in **Figure 49**, for the homogeneous material. The adhesive part of eq.(12) was not considered in the numerical model of the scratch test in micro-scale. The computation of the adhesion parcel (μ_{adh}) on the COF was conducted as described by Bowden; Tabor, (2001), and using eq. (30) assuming pure shear applied to von Mises criterion (BOWDEN; TABOR, 2001; HUTCHINGS; SHIPWAY, 2017).

$$\mu_{adh} = \frac{\tau_{cis}}{H} = \frac{0.577\sigma_y}{H} \quad (30)$$

Where: τ_{cis} is the shear stress; σ_y is the yield stress (235 MPa - AISI 304 steel and 1,413 MPa - AISI H13 steel) and H is the hardness (2.47 GPa – AISI 304 and 4.54 GPa –AISII H13).

The following results of the adhesive contribution of the COF were obtained for the austenitic (soft) and martensitic (hard) matrices against diamond:

(i-) Soft Matrix $\rightarrow \mu_{adh} = 0.05$;

(ii-) Hard Matrix $\rightarrow \mu_{adh} = 0.18$.

Table 28 – Relative error (%) between numerical and experimental results of apparent COF in two conditions: (i-) numerical result without the adhesive part consideration; and (ii-) numerical result with the adhesive part calculated based on Bowden; Tabor, (2001).

Phase	Normal Load (mN)	COF: without μ_{adh} (%)	COF: with μ_{adh} (%)
		$\left(\frac{Experimental - Numerical}{Experimental} \right) \cdot 100$	$\left(\frac{Experimental - Numerical}{Experimental} \right) \cdot 100$
Soft matrix ($\mu_{adh} = 0.05$)	50	20.8	13.0
	60	36.7	31.3
	70	13.9	8.7
	80	9.3	4.4
	100	14.7	10.5
Hard Matrix ($\mu_{adh} = 0.18$)	100	34.1	13.6
	150	29.7	11.1
	200	30.9	13.1
	400	20.2	3.7
	500	22.6	5.9

The addition of the adhesive parcel to the numerical results shown in **Figure 49** for scratching of homogeneous materials provided a maximum reduction of 20.5% of the relative error for the AISI H13 steel (hard matrix), and of about 8% for the AISI 304

steel (soft matrix), as verified in **Table 28**. However, the high strain-hardening coefficient of the stainless steel acts to increase the calculated μ_{adh} for its contact with the diamond, making it difficult to quantify the total adhesive influence (HUTCHINGS; SHIPWAY, 2017). This issue is consistent with Nosar; Olsson, (2013) research, in which the authors observed the material transference from the AISI 304 steel indenter to the tool steel pieces during scratch tests, and they affirmed that austenitic stainless steels have a high trend to adhere to many surfaces. Moreover, Xu; Van Der Zwaag; Xu (2015) showed that different hardening levels of stainless steels were responsible for determining the superficial damage responses, considering the adhesion influence on abrasion, after single and multiple passes of a rigid indenter over these materials throughout scratch tests.

Regardless of the adhesion effect, the average values of the numerical apparent COF for the homogeneous materials had a minimum difference close to 15% of the experimental. Two possible factors influenced the experimental results and were not taken into account in the modelling. These factors consist in:

(a) The effect of the grain boundaries and twins (BUCKLEY, 1968; PÖHL; HARDES; THEISEN, 2016): these regions can show higher shear resistance, since they act as barriers for the dislocation movements and can thus raise the local COF. As displayed in **Figure 50a**, the apparent COF of the diamond-AISI 304 steel pair increased locally when the abrasive cut grain 1 towards grain 2. For the diamond-AISI H13 steel pair, the apparent COF varied and a route deviation of the abrasive arose, probably due to the passage through grain boundaries (**Figure 50b**).

Figure 50 – Apparent COF as a function of scratch length for the case: (a-) diamond-AISI 304 stainless steel and constant normal load of 30 mN; and (b-) diamond-AISI H13 tool steel and constant normal load of 50 mN.

(b) The effect of the crystallographic orientation on the friction coefficient (BUCKLEY, 1968; KARAMAN et al., 2000; MACHADO et al., 2017): prior studies showed that the apparent COF was reduced in close-packed directions and planes of highest atomic density. This approach is well discussed in item **5.6.2 Crystallographic orientation**, in particular for the austenitic stainless steels.

5.4.1.2 Heterogeneous materials

With respect to the microstructure influence on the average values of apparent COF, the following overall aspects were raised based on the comparison between homogeneous and heterogeneous materials:

- materials with hard second phase particles (red curves in **Figure 51**) showed the trend to decrease the average COF when compared to the homogeneous matrices, especially above 40 mN and 50 mN loads for the soft and hard materials, respectively. Contrary to expectations, the tangential load is reduced because of the carbides and nitrides presence.

Figure 51 – Numerical and experimental results of the average apparent coefficient of friction (COF) as a function of applied normal load: heterogeneous materials with **hard** second phase particles.

(a-) AISI 310 steel: Soft matrix (AISI 304 steel) + Hard second phase (TiN)

(To be continued)

Figure 51 – Numerical and experimental results of the average apparent coefficient of friction (COF) as a function of applied normal load: heterogeneous materials with **hard** second phase particles.

(b-) Hard matrix (AISI H13 steel) + Hard second phase (NbC)

(Continuation)

In terms of energy dissipation in two-phase materials with hard precipitates, one part is spent due to the micro-cracks formation on the second phase and matrix-hard phase interface, fracture and fragmentation of these particles. Several authors (GAHR, 1998; PÖHL; HARDES; THEISEN, 2016; PÖHL; MOHR; THEISEN, 2017) corroborated these energy losses by means of the characterization of the abrasive micro-mechanisms in two-phase materials with hard second phase particles. By using a qualitative map of micro-mechanisms, Murray; Mutton; Watson (1979) indicated a possible occurrence of the fragmentation of brittle phases on the abrasion, which would imply expended energy of the tribosystem. As observed in **Figure 52**, the energy spent with the fracture intensifies the frictional energy effect, resulting in an increase of the COF value (UETZ; FÖHL, 1978). Nevertheless, the strain energy decreased because the plastic deformation effects on the hard particles are minimum or non-existent. Thus, the average COF diminishes until a critical depth (attack angle) due to the influence of the hard second phase particles. The strain energy is an important parcel that contributes to the dissipated energy by friction (UETZ; FÖHL, 1978; MOORE,

1979). Results suggest the strain energy had higher importance than the fracture energy in view of the general evaluations of apparent COF.

The comparison between numerical and experimental values of apparent COF of heterogeneous materials with hard precipitates (**Figure 51**) provided relative errors from 0.5 to 16% (**Table 29**). This allows concluding that the hard precipitates reduce the adhesion influence on the friction coefficient for the contact with the diamond abrasive, although adhesive forces occur (covalent or ionic source). Moreover, there is no plastic deformation on the asperity contacts of the abrasive-hard second phase during numerical simulations. Therefore, the heterogeneous materials with hard precipitates tend to decrease the COF.

Figure 52 – Scheme extracted from the literature that indicates the energy balance in tribosystems, focused on fracture energy, which contributes with the energy dissipation due to friction.

Reference: Adapted from Uetz; Föhl (1978).

Table 29 – Relative error (%) between numerical and experimental results of apparent COF for the heterogeneous materials with hard second phase particles.

Phases	Normal Load (mN)	COF: without μ_{adh} (%) $\left(\frac{Experimental - Numerical}{Experimental} \right) \cdot 100$
Soft matrix + TiN (AISI 310 steel)	50	-15.9
	60	-6.8
	70	9.7
	80	0.5
	100	3.4
Hard Matrix + NbC	100	11.8
	150	5.9
	200	-2.2
	400	1.6
	500	9.3

- soft material with soft precipitates of MnS (green curve in **Figure 53**): significant differences were not found in the tribological behaviour of the AISI 303 (heterogeneous) and 304 (homogeneous) steels within the evaluated loading range. The sulphides followed the matrix deformation in compression, which are in agreement with Chagas (2015) for machining conditions. Also, there was not a solid lubrication effect of the MnS decreasing the apparent COF reported elsewhere (XU et al., 2013), probably due to the low volume fraction of these particles in AISI 303 steel. Durand; Jeandin (1996) strongly disagreed that MnS acts as a solid lubricant, justifying that MnS is not lamellar as graphite, for instance, and it does not originate a layer that prevents the direct contact and accomodates the sliding.

Figure 53 – Numerical and experimental results of the average apparent coefficient of friction (COF) as a function of applied normal load: heterogeneous material with **soft** second phase particles.

Summarizing, the hard particles of TiN and NbC have the prevailing effect over the matrix regarding the COF, whereas the matrix influence appears to be predominant over the behaviour of the MnS soft particles.

5.4.1.3 Comparison with the literature

Concerning the abrasive micro-mechanisms, the numerical results of the friction coefficient as a function of attack angle, calculated according eq. (8) with the abrasive radius of 5 μm (Table 30), were depicted in the simplified graph of Challen; Oxley, (1979) apud Hokkirigawa; Kato (1988). As stated in Figure 54, the abrasive micro-mechanisms predicted based on numerical data from the literature are:

- the transition of dominant micro-ploughing to micro-cutting occurs from 50 to 100 mN for both hard materials;
- the micro-mechanism transition occurrence is estimated from 40 to 50 mN for the AISI 303 and 304 steels, whilst it takes place from 50 to 60 mN for the AISI 310 steel.

The hypothesis mentioned should be evaluated and validated by means of the post-scratching microstructural characterization. In the present chapter, the calculations are in agreement with Franco (2015) that pointed out 30° as a critical attack angle. Below this angle, the COF is low and dominant micro-ploughing occurs; above this angle, the COF increases up to 60° (cone apex angle). Nonetheless, the hypothesis for the homogeneous materials is contrary to the previous map of the degree of penetration as a function of hardness (HOKKIRIGAWA; KATO; LI, 1988), which can be observed in **Figure 33**, particularly for the AISI 304 steel that is a high work-hardening material.

Table 30 – Numerical results (average) of the attack angle and apparent COF as a function of the normal load applied to the different materials evaluated.

Material	Normal Load (mN)	Attack Angle (°) - Numerical	Apparent COF - Numerical
AISI H13 steel	50	23.9	0.31
	100	40.9	0.58
	150	54.7	0.68
	200	60.0	0.70
	400	60.0	0.87
	500	60.0	0.84
AISI H13 steel+NbC	50	20.6	0.22
	100	30.6	0.45
	150	43.2	0.59
	200	52.4	0.74
	400	60.0	0.87
	500	60.0	0.84
AISI 304 steel	40	25.2	0.14
	50	32.0	0.52
	60	38.2	0.59
	70	44.5	0.82
	80	44.2	0.92
	100	51.0	1.02
AISI 304 steel + MnS (AISI 303 steel)	40	22.6	0.13
	50	31.9	0.52
	60	40.1	0.76
	70	44.6	0.88
	80	44.2	0.92
	100	51.3	1.02
AISI 304 steel + TiN (AISI 310 steel)	40	25.0	0.15
	50	27.2	0.24
	60	34.1	0.30
	70	40.3	0.43
	80	43.2	0.58
	100	54.1	0.73

Figure 54 – Results of the computational modelling superimposed on the literature map of Challen; Oxley (1979) apud Hokkirigawa; Kato (1988): apparent coefficient of friction as a function of attack angle for all studied materials.

Numerical Results - Apparent COF x Attack Angle

5.4.2 Geometrical parameters of the scratch and removed volume

Figure 55 shows a prior numerical model validation of scratch test in the micro-scale, since the numerical results of the average penetration depth are in agreement with the experimental results and the error is lower than 10%. The numerical errors of depth of penetration and removed volume may be assigned to the mesh effect. Although convergence analysis and refining of contact region have been carried out, the time and computational resources were considered in the decision-making. Moreover, the mesh discretization limit is also a function of X-rays micro-tomography resolution.

Figure 55 – Numerical and experimental results related to the average values of the depth of penetration as a function of normal load for the soft (a-) and hard materials (b-).

Figure 56 – Evaluation of the linear fitting of the penetration depth data acquired from the numerical modelling by varying the normal load: (a-) soft materials; and (b-) hard materials.

Firstly, based on numerical results, the depth of penetration can be related to the normal loads by linear functions, in which it tends to increase gradually with the load, as observed in **Figure 56**; the trend line crosses the origin, and regression curves have good correlation coefficients. Hard materials had a better fitting when compared with the soft materials, presumably because of their different work-hardening behaviour during the scratch test. The same linear relation was verified by means of the micro-scratch test using a diamond tip against AISI 1070 carbon steel and gray cast iron (SERIACOPI, 2017), and by indentation of DC04 steel used to make bearing steel balls (TRZEPIECIŃSKI et al., 2015).

Secondly, from the experimental view, **Figure 57** corroborates the good linear fitting of the depth of penetration with the normal load for two different setups: with the loaded specimen (triboindenter data - Hysitron) and with the unloaded specimen (optical interferometry data - CCI). Once again, the best fit is obtained for the hard materials. For the soft materials, only the experimental data of the penetration depth of the AISI 304 and 310 steels were inserted into the graph, since the depths resulting from AISI 303 and 304 scratch tests were very similar. Particularly, the measurements of the depth of penetration after the unloading (CCI) on the stainless steels presented considerable dispersion values (**Figure 57a**). The wear debris and chip deposits are on the wear track and, thus, characterization of the false pile-up occurs (FRANCO; SINATORA, 2017), making it difficult to establish a baseline definition, and an asymmetric pile-up formation is found due to the change of the crystallographic grain orientation along the scratch. The false pile-up is intensified in these materials due to their high work-hardening, which tends to minimize the formation of the pile-up, as observed in the scratch tests of Pöhl; Hardes; Theisen (2016).

Figure 57 – Analyses of the linear fitting of the penetration depth data acquired from the experimental test by varying the normal load: (a-) soft materials; and (b-) hard materials. In this case, the information about the depth of penetration is provided during the scratching (Hysitron data) and after the elastic recovery of the specimens (CCI data).

The maximum elastic recovery of the studied material was calculated from the experimental data shown in **Figure 57**, since the depth measured by the optical interferometer includes this influence. Therefore, the recovery elastic is predicted, according to the methodology proposed by Kataria et al. (2009), as being the ratio between the difference in the total penetration depth (Hysitron data) and residual penetration depth (CCI data), and that total penetration depth (Hysitron). In terms of homogeneous materials, the maximum elastic recovery was 0.21 for the AISI 304 steel and 0.35 for the AISI H13 steel. In turn, the heterogeneous materials presented the maximum elastic recovery corresponded to 0.24 for the AISI 310 steel and 0.58 for the AISI H13+NbC. Results indicate a consistent tendency based on plastic deformation behaviour, because the elastic recovery is lower when high levels of the material plastic deformation occurs (KATARIA et al., 2009): the 304 steel suffers more plastic deformation than the H13 steel, and each one is more deformed than its respective heterogeneous material with hard precipitates.

As displayed **Figure 58**, the scratch width characterized by optical interferometer at the baseline and between pile-ups (peak-to-peak) presents a quadratic relation to the applied normal load. This result is in accordance with the Scratch Hardness Number (HS_p) of the ASTM G171 – 03 standard (AMERICAN SOCIETY FOR TESTING AND MATERIALS, 2009) and with Kobrick; Klaus; Street (2009), in which the Al 6061-T6 alloy was scratched using a Rockwell C diamond abrasive with 200 μm tip radius. Specially for the cono-spherical geometries, eq. (31) quantifies the penetration resistance (HS_p) as a function of abrasive tip movement over the solid surface, considering the constant normal load and speed (KOBICK; KLAUS; STREET, 2009; ASM INTERNATIONAL, 2017):

$$HS_p = \frac{8W}{\pi l_p^2} \quad (31)$$

Where: W is the normal load and l_p is the scratch width.

Figure 58 – Experimental values of the scratch width as a function of applied normal load, measured using two approaches – at baseline and between pile-up (peak-to-peak) – for soft materials (a-) and hard materials (b-).

Figure 59 – Reduced scratch width on the hard second phase particles – comparison between numerical and experimental outcomes: (a-) micro-cutting (severe wear) of the heterogeneous hard material; and (b-) micro-ploughing (mild wear) of the heterogeneous soft material with TiN hard particles.

Martensitic matrix – green; NbC (shell) – white; $Ti_x-Nb_{1-x}-C$ (core) – red.

Austenitic matrix – green; TiN – blue.

Regarding the microstructural aspects, the decrease of the scratch width can be observed in **Figure 58** for the heterogeneous materials with hard precipitates when compared to their homogeneous materials. The numerical simulation by FEM allowed reproducing this effect. Numerical and experimental results show the same characteristics of the deformed microstructure with respect to the scratch width, comparing with the top views in **Figure 59**. For the heterogeneous hard material (AISI H13 steel + NbC), the cono-spherical abrasive with tip radius of 5 μm and a normal load of 150 mN were considered in the numerical model; whilst the experimental scratch test was conducted with a Berkovich tip and a normal load of 9 mN. As can be seen in **Figure 59a**, the numerical result and the SEM/SE image showed similarities in terms of the matrix and carbide damages throughout the scratching. The matrix suffers plastic deformation and mass removal with debris formation occurs. The rigid abrasive presents a high resistance to continue the path when it meets the carbide,

and the brittle failure can occur in this scratch region. Likewise, for the soft matrix with TiN particles, the track width reduction can be observed during the abrasive passage through the hard phase (**Figure 59b**) even when the dominant abrasive mechanism is the micro-ploughing (20 mN).

As previously described (item **3.3 MATERIAL REMOVAL – CALCULATION METHOD**), the removed volume was calculated based on the circular segment. Considering the abrasive micro-mechanisms estimated by using apparent COF analyses (**Figure 54**), the following approaches were used: (i-) pile-up volume subtracted from the total removed volume ($f_{ab}=0$) for the dominant micro-ploughing; and (ii-) total removed volume ($f_{ab}=1$) was considered for the dominant micro-cutting. Results of pile-up volume as well as intermediate parameters for its calculation are displayed in **Table 31**. They were estimated by applying the numerical model outputs (depth of penetration) and analytical models of the literature (GAO; JING; SUBHASH, 2006; NIKAS, 2015).

Table 31 – Pile-up volume calculated by using depth of penetration data, obtained from numerical simulation, and employing a literature analytical model (GAO; JING; SUBHASH, 2006; NIKAS, 2015).

Material	Normal Load (mN)	Depth of Penetration (μm)	Pile-up Height (μm)	Volume of spherical cap (μm^3)	Radius of the ECM hydrostatic core - R_h (μm)	Outer radius of the ECM plastic zone - r_p (μm)	Pile-up Volume - V_{pu} (μm^3)
AISI H13 steel	50	0.43	0.13	2.82	2.03	4.88	2.90
	100	1.22	0.38	21.5	3.27	9.25	23.01
AISI H13 steel+NbC	50	0.32	0.10	1.61	1.77	3.81	1.65
	100	0.70	0.22	7.28	2.55	6.19	7.59
AISI 304 steel	40	0.48	0.13	3.45	2.13	9.75	3.55
	50	0.76	0.21	8.57	2.65	13.03	8.97
AISI 303 steel	40	0.38	0.11	2.25	1.92	8.50	2.31
	50	0.76	0.21	8.54	2.64	13.01	8.94
AISI 310 steel	40	0.47	0.14	3.36	2.12	9.37	3.46
	50	0.55	0.16	4.59	2.28	10.36	4.75
	60	0.86	0.25	10.95	2.801	13.64	11.53

Figure 60 depicts the numerical and experimental results of the removed volume, normalised by the scratch length, of the homogeneous and heterogeneous soft material for the tested normal loads; whereas **Figure 61** displays the same comparison for the hard materials. Similarly to the depth of penetration response, the values of material removal are validated by laboratory tests, and errors were lower than 15%.

Figure 60 – Comparative analysis of the numerical and experimental removed volume, normalised by the scratch length, as a function of the normal load for the soft materials evaluated.

Figure 61 – Comparative analysis of the numerical and experimental removed volume, normalised by the scratch length, as a function of the normal load for the hard materials evaluated.

(a-)

Homogeneous - AISI H13 tool steel

(b-)

Heterogeneous - AISI H13 + NbC

Figure 62 – Compilation of the removed volume values, attained from the numerical model of micro-scratch tests: (a-) for the austenitic stainless steels; and (b-) for the tool steels.

The numerical result of removed volume of the AISI 303 steel with MnS inclusions is comparable to the austenitic matrix (AISI 304 steel), as can be seen in **Figure 62a**. Conversely, for most of the analyses the AISI 310 steel provided lower material removal than the AISI 304 steel (**Figure 62a**). This behaviour in reducing the removed volume based on hard second phase effect was also observed in the hard-heterogeneous materials (**Figure 62b**). The effect of the hard second phase decreasing the material removal is in agreement with several authors (GAHR, 1998; HU; LI; LLEWELLYN, 2007; PÖHL; MOHR; THEISEN, 2017). In the present thesis, the following conditions can be established:

- The mechanical behaviour of the second phase particles was decisive in the removed material reduction; conversely, the MnS (soft particle) did not play a significant role in this aspect;
- The depth (critical size) of the second phase particles is a relevant factor to be considered in the analysis. Particularly, the TiN acted as strengthening phase until reaching 80 mN. For higher loads, it tended to be damaged and to attain its limit deformation capacity (DUTRA et al., 2013); in these conditions, TiN might be pulled out applying the load of 100 mN, and increases the volume removed of the 310 steel when compared to austenitic matrix (**Figure 62a**).

To investigate the transition of the abrasive micro-mechanisms, the dimensional wear coefficient (k) was calculated by eq. (3) as stated by different authors (ARCHARD, 1953; RABINOWICZ; DUNN; RUSSELL, 1961; HUTCHINGS; SHIPWAY, 2017), and by employing average values of the removed volume from computational modelling. Eq. (3) implies the dimensional wear coefficient does not consider the material hardness, and it is expressed in $[\text{mm}^3 \text{N}^{-1} \text{m}^{-1}]$. This parameter refers to the specific material removal ratio for a defined tribological condition (DUTRA et al., 2013).

In contrast to the initial indications of the analyses using apparent COF, there is no strong transition of abrasive micro-mechanism by the approach of the dimensional wear coefficient as a function of normal load (**Table 32**). Although the change in an order of magnitude occurs in some cases (detached), it seems to be slight. Equivalent results are experimentally characterized by Franco (2015), who indicated a prevalence of one abrasive micro-mechanism in scratches by evaluating apparent COF. Concerning the order of magnitude validity of the removed volume calculated in the numerical model, the values shown in **Table 32** follow Dutra et al., (2013), which listed k-values ranging between $\sim 3 \times 10^{-2}$ and $\sim 7 \times 10^{-2} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ for scratch tests carried out using a diamond Vickers indenter (136° of angle) over high chromium cast irons (380-450 HV_{0.2}).

Table 32 – Dimensional wear coefficients calculated from the numerical simulation removed volume for the distinct materials and normal load conditions.

Material	Normal Load (mN)	k (mm ³ N ⁻¹ m ⁻¹)
AISI H13 steel	50	2.28E-02
	100	5.48E-02
	150	7.50E-02
	200	8.97E-02
	400	1.11E-01
	500	1.11E-01
AISI H13 steel+NbC	50	1.47E-02
	100	2.40E-02
	150	4.24E-02
	200	5.40E-02
	400	6.68E-02
	500	7.88E-02
AISI 304 steel	40	3.35E-02
	50	5.44E-02
	60	7.55E-02
	70	9.88E-02
	80	8.48E-02
	100	1.01E-01
AISI 304 steel + MnS (AISI 303 steel)	40	2.41E-02
	50	5.42E-02
	60	8.63E-02
	70	9.95E-02
	80	8.51E-02
	100	1.02E-01
AISI 304 steel + TiN (AISI 310 steel)	40	3.27E-02
	50	3.39E-02
	60	5.47E-02
	70	7.53E-02
	80	7.99E-02
	100	1.17E-01

5.5 Local comparison between numerical and experimental results of the scratch tests in micro-scale: homogeneous and heterogeneous materials

5.5.1 Hard second phase particles

5.5.1.1 Apparent coefficient of friction

Figure 63 allows observing the correspondence of the apparent COF, obtained via computational modelling, with the microstructure of the hard materials along the scratch length.

For the rigid abrasive against heterogeneous hard material, the COF was grouped in 3 categories according to characterizations of the dominant abrasive micro-mechanisms, utilizing the top views of the numerical results as shown in **Figure 63a**:

- Case 1 – 50 and 100 mN: micro-ploughing (plastic deformation) and wedge formation, respectively;
- Case 2 – 150 mN: less severe micro-cutting, with chip formation;
- Case 3 – 400 and 500 mN: more severe micro-cutting, with accentuated material removal and precipitate fragmentation in the heterogeneous materials.

The same abrasive micro-mechanisms resulted from the micro-scratch simulation with the rigid abrasive against the AISI H13 steel - homogeneous hard material (**Figure 63b**).

Figure 63 – Results of the numerical modelling: apparent coefficient of friction along the sliding distance for the abrasive-hard materials contact pair based on different normal load conditions. The top views of the deformed microstructure are included in the evaluation of this tribological response. The image scales are compatible with the scratch length of the graphs (abscissa).

In all the cases in **Figure 63a**, under constant normal load, the apparent COF was verified to increase before and after the carbide (hard particle) position. This can be assigned to the accumulated local plastic deformation of the martensitic matrix, i.e. the addition of the energy part absorbed due to deformation that composes the frictional work (MOORE, 1979). To meet the ductile damage criterion of the matrix, null stiffness is given to the element, which is deleted from the domain, allowing the abrasive pursuance with removal of a part that suffered excessive plastic deformation. Keeping the applied normal load along the track, the abrasive particle finds the carbides, in which the energy parts of elastic deformation and a possible fracture, with new surfaces creation, become the main contributions of the friction coefficient; thus, the apparent COF is reduced. However, the brittle second phase particles can present certain plasticity when submitted to abrasion, as pointed out by Moore (1979), contributing with a small percentage of the strain energy.

With reference to the overall evaluation of **Figure 63**, for the homogeneous and heterogeneous materials, the apparent coefficient of friction tends to rise with the applied normal load up to reaching a constant value, in which the severe micro-cutting occurs (400 and 500 mN in **Figure 63a**, and 400 mN in **Figure 63b**), with maximum dissipated cutting energy (damage) and constant attack angle equal to the cone apex angle.

Similarly to the heterogeneous hard material, the numerical results of apparent COF for the heterogeneous soft material with hard precipitates (AISI 310 steel with austenitic matrix and TiN particles) in **Figure 64** showed a local increase of their values when the abrasive precedes the hard second phase position due to the cumulative plastic deformation of the soft matrix. In the same way, there is a decrease of the deformation part of the friction coefficient when the rigid abrasive overpasses the nitride in all cases (**Figure 64**). Specifically, the 60 mN condition presented convergence problems with a scratch speed of 4,000 $\mu\text{m/s}$, which was modified to 3,000 $\mu\text{m/s}$ afterwards to allow the further movement of the abrasive up to the final sliding distance of 100 μm . For this reason, some slight oscillations in the tangential load signal (COF) can be verified in **Figure 64**, with a better definition of the COF fall when the TiN is cut.

Figure 64 – Results of the numerical modelling: apparent coefficient of friction along the sliding distance for the abrasive-AISI 310 steel contact pair based on different normal load conditions. The AISI 310 consists of a soft austenitic matrix with hard particles of TiN. The top views of the deformed microstructure are included in the evaluation of this tribological response. The image scales are compatible with the scratch length of the graphs (abscissa).

To investigate the microstructural effects of the soft materials in terms of the depth of penetration, removed volume and specific energy, the normal load cases were separated based on the prevailing abrasive micro-mechanisms; the procedure for the hard materials was also performed:

- 40 mN: dominant micro-ploughing (mild regime);
- 50 mN: transition from micro-ploughing to micro-cutting (wedge formation);
- 60 mN: less severe micro-cutting;
- 80 mN: more severe micro-cutting.

5.5.1.2 Depth of penetration, material removal and specific energy

5.5.1.2.1 Dominant micro-ploughing

Hard matrix and carbides:

From the numerical simulation results, the depth of penetration, and as a consequence of the removed volume, comes down at the carbide positions, when there is a predominance of micro-ploughing, e.g. in the 50 mN case (**Figure 65**). These hard second phase particles help to sustain the external applied normal load, and tend to minimize the rigid abrasive effect over the heterogeneous material, acting as barriers to be overtaken (GAHR, 1987). In turn, the plastic deformation of the martensitic matrix of the heterogeneous material is reduced when compared to the homogeneous material, since the carbides have a notable size to promote a better material resistance for this condition.

The following general evaluations in terms of energies were taken into account for calculating the specific energy for the heterogeneous hard material:

- strain energy: this energy parcel receives elastic contribution, and a minimum plastic contribution, at the carbide regions. Hence, a marked reduction of the strain energy would be expected locally;
- frictional energy: a decrement of the tangential load (apparent COF) is verified in **Figure 63a** at the carbide positions. Thus, the decrease of energy loss due to the friction is predicted in these regions;
- cutting (damage) energy: it is not possible to assert that considerable energy was spent with the cutting and/or the carbides fragmentation due to the dominant abrasive micro-mechanisms. In this case, the cutting energy parcel would not be significant when compared to the others.

Based on the listed considerations for micro-ploughing, it can be concluded that the specific energy only increases locally at the carbides as a consequence of the decrease of the removed volume, and not because of the sum of the energies. In the same way, for the specific condition of 50 mN (**Figure 65**), the specific energy of the heterogeneous material is higher than that of the homogeneous material owing to the removed volume effect. The numerical results of **Figure 65** for the prevailing micro-ploughing are validated by the experimental results seen in **Figure 66**. The local aspects verified in the numerical simulations were corroborated with the experimental

analyses conducted considering larger scratch lengths, which were representative of the behaviour under abrasion of the materials studied.

Figure 65 – Numerical simulation results by applying a normal load of 50 mN – local comparison between the AISI H13 steel (homogeneous hard material) and the AISI H13+carbides (heterogeneous hard material). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

**Carbides (hard particles
on the hard matrix):**

↓Depth of penetration = ↓V
and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

Figure 66 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the hard materials. In this condition, the test was carried out with constant normal load of 50 mN ($15 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-ploughing (mild wear). Also, the scratch length shown in the micrograph is compatible with the graph scales.

(a-) Heterogeneous hard material: martensitic matrix with NbC hard precipitates – SEM images (BSE)*.

(b-) Homogeneous hard material: AISI H13 hot work tool steel – SEM images (SE)*.

*Micrographs by Erika Fernanda Prados.

Soft matrix and nitrides:

By means of simulation, the nitrides role in the austenitic matrix is verified to be compatible with the carbides in the martensitic matrix: **Figure 67** shows that the depth of penetration is lower in the TiN region, resulting in reduced values of the removed volume and maximum values of local specific energy. The plastic deformation of the AISI 310 austenitic matrix is accumulated preceding its hard phase position; yet there is no excessive plastic deformation of this matrix after the abrasive event over the nitride. A possible reason for this behaviour can be the high work-hardening of the austenitic stainless steels that tends to minimize the pile-up formation, which was inferred from the literature (HERNOT et al., 2006; PÖHL; HARDES; THEISEN, 2016) by indentation tests.

The experimental validation of the 40 mN (micro-ploughing condition) for the heterogeneous soft material with hard second phase particle is available in **Figure 68**. The tribological feature responses obtained from the numerical model agree with the experimental scratch test outcomes.

Figure 67 – Numerical simulation results by applying a normal load of 40 mN – AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

(To be continued)

Figure 67 – Numerical simulation results by applying a normal load of **40 mN** – AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

**Nitrides (hard particles
on the soft matrix):**

↓ Depth of penetration = ↓ V
and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

(Continuation)

Figure 68 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with hard particles. In this condition, the test was carried out with constant normal load of **40 mN** ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-ploughing (mild wear). Also, the scratch length shown in the micrograph is compatible with the graph scales.

Heterogeneous soft material with TiN hard particles: AISI 310 austenitic stainless steel – SEM images (BSE).

5.5.1.2.2 Transition from micro-ploughing to micro-cutting

Hard matrix and carbides:

Applying a normal load of 100 mN, the local effects in terms of the removed volume, depth of penetration and specific energy are the same observed for the first case (50 mN). However, the amplitude of the specific energy is reduced to 100 mN, specially in the hard precipitate regions (**Figure 69**), indicating a possible transition from micro-ploughing to micro-cutting. Lee (1993) identified the same behaviour of the abrasive mechanism transition developed from the diminished specific energy with the increase of the material removal ratio during honing processes. Similarly, the author divided the specific energy components into ploughing, related to the deformation parcel; sliding corresponding to the frictional part; and chipping formation including the cutting contribution.

As regards the 100 mN case, which exemplifies the micro-mechanism transition, it was validated experimentally as shown in **Figure 70**. Nevertheless, oxidation points can be noted on the scratch track of the AISI H13 steel (**Figure 70b**), which are not reproduced by modelling. The simulation does not take into account the local heating that occurs as a function of plastic deformation. Moore (1979) pointed out that 90% of the energy spent with plastic deformation is often dissipated in heating form. Furthermore, **Figure 70a**, which contains the experimental results of the heterogeneous hard material – 100 mN, also corroborates that the influence of the deformation component is predominant over the fracture component with respect to the apparent COF values, which keep low at the carbides. Likewise, a specific energy peak of about 70 μm from the scratch end (from right to left) can be observed in **Figure 70a**, and it designates the fracture of the carbide shell (NbC) with its exposed core ($\text{Ti}_x\text{-Nb}_{1-x}\text{C}$). Once again, for the numerical and experimental outputs, the removed volume of the homogeneous is higher than that of the heterogeneous material, showing the effectiveness of the hard precipitates on the improvement in the resistance to the material removal by abrasion.

Figure 69 – Numerical simulation results by applying a normal load of 100 mN – local comparison between the AISI H13 steel (homogeneous hard material) and the AISI H13+carbides (heterogeneous hard material). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

**Carbides (hard particles
on the hard matrix):**

↓Depth of penetration = ↓V
and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

Figure 70 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the hard materials. In this condition, the test was carried out with constant normal load of 100 mN ($15 \mu\text{m s}^{-1}$). A transition from micro-ploughing to micro-cutting has occurred. Also, the scratch length shown in the micrograph is compatible with the graph scales.

(a-) Heterogeneous hard material: martensitic matrix with NbC hard precipitates – SEM images (BSE)*.

(b-) Homogeneous hard material: AISI H13 hot work tool steel – SEM images (SE)*.

*Micrographs by Erika Fernanda Prados

Soft matrix and nitrides:

Albeit intensified, the local numerical responses observed in micro-ploughing condition remain the same for the mechanism transition (**Figure 71**), and can be confirmed by the data arising from experimental tests (**Figure 72**): the TiN of the 310 steel tends to decrease the local material removal, increasing the specific energy values (peaks), since the hard second phase particles can be sheared with the increment of the applied normal load (GAHR, 1987), resulting in higher specific cutting energy.

Figure 71 – Numerical simulation results by applying a normal load of 50 mN - AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

(To be continued)

Figure 72 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with hard particles. In this condition, the test was carried out with constant normal load of 50 mN ($10 \mu\text{m s}^{-1}$). A transition from micro-ploughing to micro-cutting has occurred. Also, the scratch length shown in the micrograph is compatible with the graph scales.

**Nitrides (hard particles
on the soft matrix):**

↓ Depth of penetration = ↓ V

and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

(Continuation)

Figure 72 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with hard particles. In this condition, the test was carried out with constant normal load of 50 mN ($10 \mu\text{m s}^{-1}$). A transition from micro-ploughing to micro-cutting has occurred. Also, the scratch length shown in the micrograph is compatible with the graph scales.

Heterogeneous soft material with TiN hard particles: AISI 310 austenitic stainless steel – SEM images (BSE).

5.5.1.2.3 Dominant micro-cutting

Hard matrix and carbides:

The characterization of the micro-cutting of the hard materials was divided into a less severe case – 150 mN – and a more severe case – 400 mN.

According to **Figure 73** and **Figure 75**, in which micro-cutting prevails, the numerical simulation allows verifying the chip formation and carbide cutting due to the abrasive action. The micro-cutting evidences can be also supported by the evaluation of the friction coefficient along the scratch length, when a normal load of 150 mN was used over the heterogeneous hard material (**Figure 63a**). In this case, COF peaks occurred due to the localized plastic deformation of the martensitic matrix, which plays a role in reducing the deformation capacity throughout the micro-scratching to provide the micro-cutting (GAHR, 1987).

For the 150 mN condition (**Figure 73**), the depth of penetration is lower than the carbide thickness, and they may substantially act in the material removal resistance. On the other hand, for the 400 mN condition (**Figure 75**), the depth reached by the abrasive particle is comparable to the critical dimension of the second phase particles, promoting their significant shear and fragmentation (MURRAY; MUTTON; WATSON, 1979; GAHR, 1987), which tend to approximate the maximum values of the removed volume of the heterogeneous material to the homogeneous material.

As a consequence of the different severity levels of abrasion on the hard materials, the following aspects can be verified by numerical simulation:

- the specific energy for the 150 mN case (**Figure 73**): maximum points occur in the carbide positions, and the specific energy amplitude is lower for the carbide with a higher Ti core, since the $Ti_x-Nb_{1-x}C$ fracture toughness is 20% higher than the NbC, which minimizes the local cutting energy parcel (damage);
- the specific energy for the 400 mN case (**Figure 75**): as opposed to the others, in this case, the specific energy shows valleys at the precipitates positions because they were pulled out and fragmented on the wear track, increasing the local removed volume.

With a good statistical representativeness, the numerical local results in **Figure 73** and **Figure 75** are considered validated with the experimental local results provided in **Figure 74** and **Figure 76**, respectively. The experimental tests confirm that the

carbides remain on the wear track after the scratch using 150 mN load (**Figure 74**), which does not occur when 400 mN is applied (**Figure 76**) in which the track is carbide-free. During the dominant micro-cutting, a notable quantity of chips is formed in the homogeneous material (AISI H13 steel), without strong evidences of oxidation as seen in the 100 mN condition (abrasive micro-mechanism transition). In relation to the heterogeneous material, a differential fact is displayed in **Figure 76** between 150 and 250 μm : probably due to a carbide net on the track sides, with an optimum ratio of carbide size and spacing, there was a decreased in the removed volume in this region, which significantly increased the local specific energy.

Figure 73 – Numerical simulation results by applying a normal load of 150 mN – local comparison between the AISI H13 steel (homogeneous hard material) and the AISI H13+carbides (heterogeneous hard material). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

**Carbides (hard particles
on the hard matrix):**

↓ Depth of penetration = ↓ V

and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

Figure 74 –Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the hard materials. In this condition, the test was carried out with constant normal load of 150 mN ($15 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-cutting (less severe). Also, the scratch length shown in the micrograph is compatible with the graph scales.

(a-) Heterogeneous hard material: martensitic matrix with NbC hard precipitates – SEM images (BSE)*.

(b-) Homogeneous hard material: AISI H13 hot work tool steel – SEM images (SE)*.

* Micrographs by Erika Fernanda Prados.

Figure 75 – Numerical simulation results by applying a normal load of 400 mN – local comparison between the AISI H13 steel (homogeneous hard material) and the AISI H13+carbides (heterogeneous hard material). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

Carbides (hard particles on the hard matrix):

↓Depth of penetration = ↓V

and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

Figure 76 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the hard materials. In this condition, the test was carried out with constant normal load of 400 mN ($15 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-cutting (more severe). Also, the scratch length shown in the micrograph is compatible with the graph scales.

(a-) Heterogeneous hard material: martensitic matrix with NbC hard precipitates – SEM images (BSE)*.

(b-) Homogeneous hard material: AISI H13 hot work tool steel – SEM images (SE)*.

* Micrographs by Erika Fernanda Prados.

Soft matrix and nitrides:

For the less severe micro-cutting (60 mN), as well as for the other micro-mechanisms, the hard particle of the nitride act to reduce the located volume loss and, as a consequence, it raises the specific energy at the micro-region (**Figure 77**). However, the abrasive excessively penetrates the soft matrix after its passage by the hard nitride. This differential behaviour in relation to the others develops a plateau at the removed volume graph between 50 and 100 μm (**Figure 77**), in addition to the pronounced fall of the local specific energy. Despite the numerical results being validated qualitatively, the variations in the tribological outputs are not so clear in the presence of TiN during the experimental scratch tests conducted using a 60 mN load (**Figure 78**).

As regards the more severe micro-cutting (80 mN), the computational modelling results point out a large removed volume of the 310 steel with fragmentation of the TiN particle (**Figure 79**). As the less severe micro-cutting (60 mN), the specific energy maximum point is kept in the nitride region during the more severe micro-cutting; in the latter case, the peak seems to be attenuated (lower amplitude). **Figure 80** indicates the experimental validation of the more severe micro-cutting, in which a considerable chip formation can be noted.

Figure 77 – Numerical simulation results by applying a normal load of 60 mN – AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

**Nitrides (hard particles
on the soft matrix):**

↓Depth of penetration = ↓V

and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

Figure 78 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with hard particles. In this condition, the test was carried out with constant normal load of 60 mN ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-cutting (less severe). Also, the scratch length shown in the micrograph is compatible with the graph scales.

Heterogeneous soft material with TiN hard particles: AISI 310 austenitic stainless steel – SEM images (BSE and SE).

Figure 79 – Numerical simulation results by applying a normal load of 80 mN – 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

**Nitrides (hard particles
on the soft matrix):**

↓Depth of penetration = ↓V
and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

Figure 80 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with hard particles. In this condition, the test was carried out with constant normal load of 80 mN ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-cutting (more severe). Also, the scratch length shown in the micrograph is compatible with the graph scales.

Heterogeneous soft material with TiN hard particles: AISI 310 austenitic stainless steel – SEM images (BSE and SE).

5.5.2 Soft second phase particles

5.5.2.1 Apparent coefficient of friction

Due to the similarity of mechanical properties of the AISI 303 and 304 steels, it is not possible to observe in **Figure 81a,b** a representative influence of the MnS inclusions on the numerical response of apparent COF along the scratch length. The sulphides deformed with the soft matrix in **Figure 81a**, since their damage properties were not specified in modelling, and there are only small variations and distinct COF

values for the cases in which the micro-cutting seems to be dominant (60, 70 and 80 mN).

Figure 81 – Results of the numerical modelling: apparent coefficient of friction along the sliding distance, based on different normal load conditions. The contact pairs are: rigid abrasive – AISI 303 steel (a-) and rigid abrasive – AISI 304 steel (b-). The top views of the deformed microstructure are included in the evaluation of this tribological response. The image scales are compatible with the scratch length of the graphs (abscissa).

5.5.2.2 Depth of penetration, material removal and specific energy

5.5.2.2.1 Dominant micro-ploughing

Concerning the micro-ploughing condition (40 mN), the numerical modelling results of removed volume and depth of penetration (**Figure 82**) did not exhibit a significant influence of the sulphides (soft particles – AISI 303 steel) along the scratching when compared to the nitrite influences (hard particles – AISI 310 steel). Nonetheless, in view of the specific energy approach (**Figure 82**), the sulphide regions of the AISI 303 steel are prone to cause specific energy variations, which may be attributed to a gradual decline of the specific strain energy during the abrasive movement over the sulphide. This energy sharply increases after the abrasive event due to the matrix material resistance in front of the abrasive particle. The matrix material works as a barrier. Such condition was evidenced in the literature (NAKAMURA; IWABUCHI, 2012; MENDAS; BENAYOUN, 2013), in terms of the friction coefficient changes during the pass of the diamond abrasive over graphite soft particles in cast irons: the COF value presented a local drop, and the continuous sliding of the abrasive over the surface suddenly increased its value.

For the AISI 303 steel, the experimental validation of the numerical behaviour can be observed in **Figure 83**. However, the apparent COF fluctuations seem to be substantially influenced by the grain boundaries and crystallographic orientations, which were not considered in the numerical modelling by FEM.

Figure 82 – Numerical simulation results by applying a normal load of 40 mN – AISI 303 steel (austenitic matrix + MnS) x AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

Sulphides (soft particles on the soft matrix):

(no effect) Depth of penetration = (no effect) V

and

$$\uparrow \downarrow E_{sp} = \frac{(\uparrow \downarrow E_{pd}) + E_f + E_c}{V}$$

Nitrides (hard particles on the soft matrix):

$\downarrow$ Depth of penetration = $\downarrow V$

and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

Figure 83 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with soft particles. In this condition, the test was carried out with constant normal load of 40 mN ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-ploughing (mild wear). Also, the scratch length shown in the micrograph is compatible with the graph scales.

Heterogeneous soft material with MnS soft particles: AISI 303 austenitic stainless steel – SEM images (SE). (*) microstructural effect (material heterogeneity); (**) anisotropy effect of the material.

5.5.2.2.2 Transition from micro-ploughing to micro-cutting

In contrast to the micro-ploughing condition (**Figure 82**), the numerical results in **Figure 84** indicate that the material removal of the AISI 303 steel is higher than the AISI 310 steel during the abrasive micro-mechanism transition from mild to severe with 50 mN load. For the wedge formation case, the sulphides of the AISI 303 are deformed along with the austenitic matrix and pushed into the sides, creating pile-up and chips as can be seen in **Figure 84**. The influence of the MnS of the 303 steel is more pronounced in oscillations of specific energy signal, due to the small and abrupt variations of the strain energy. In experimental terms (**Figure 85**), the anisotropy of this steel affects the tangential load (and, thus, the apparent COF), probably because of the mechanical properties of different grains (MACHADO et al., 2017).

Figure 84 – Numerical simulation results by applying a normal load of 50 mN – AISI 303 steel (austenitic matrix + MnS) x AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

Sulphides (soft particles on the soft matrix):

(no effect) Depth of penetration = (no effect) V
and

$$\uparrow \downarrow E_{sp} = \frac{(\uparrow \downarrow E_{pd}) + E_f + E_c}{V}$$

Nitrides (hard particles on the soft matrix):

$\downarrow$ Depth of penetration = $\downarrow V$
and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

Figure 85 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with soft particles. In this condition, the test was carried out with constant normal load of **50 mN** ($10 \mu\text{m s}^{-1}$). A transition from micro-ploughing to micro-cutting has occurred. Also, the scratch length shown in the micrograph is compatible with the graph scales.

Heterogeneous soft material with MnS soft particles: AISI 303 austenitic stainless steel – SEM images (SE). (*) axisymmetric pile-up creation (crystallographic orientation effect); (**) chip formation.

5.5.2.2.3 Dominant micro-cutting

For the less severe micro-cutting (60 mN), the numerical results indicated, as well as for the micro-mechanism transition, the AISI 303 steel had a higher material removal than the AISI 310 steel (**Figure 86**). Conversely, the micro-cutting smoothed the sulphide effects with respect to the specific energy, since the energy spent with the chip formation tends to surpass the other effects that became secondary. The experimental results observed in **Figure 87** corroborate this local aspect, in particular the apparent COF and specific energy along the scratch length.

As stated in **Figure 86**, the MnS inclusions did not perform as stress concentrator regions, intensifying the localized removed volume for the micro-cutting load, in contrast to what was verified throughout machining conditions (LJUSTINA; LARSSON;

FAGERSTRÖM, 2014). The factors that can provide this distinction and the inhibition of the inclusion role consist in: (a-) simplified mechanical loading of the scratch tests in micro-scale when compared to the machining stresses; (b-) the scratch setup being orthogonal to the rolling direction, where the sulphides are shown as elongated particles, because the distribution, morphology and size of these soft particles influence the contact stress fields (FUKUMASU et al., 2005); and (c-) no consideration about the thermal loadings into the modeling of the micro-scratch tests.

Figure 86 – Numerical simulation results by applying a normal load of 60 mN – AISI 303 steel (austenitic matrix + MnS) x AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

Sulphides (soft particles on the soft matrix):

(no effect) Depth of penetration = (no effect) V

Nitrides (hard particles on the soft matrix):

↓Depth of penetration = ↓V
and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

(To be continued)

Figure 86 – Numerical simulation results by applying a normal load of 60 mN – AISI 303 steel (austenitic matrix + MnS) x AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

(Continuation)

Figure 87 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with soft particles. In this condition, the test was carried out with constant normal load of 60 mN ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-cutting (less severe). Also, the scratch length shown in the micrograph is compatible with the graph scales.

Heterogeneous soft material with MnS soft particles: AISI 303 austenitic stainless steel – SEM images (SE).

Concerning the more severe micro-cutting (80 mN), the numerical results provided once again a reversal tribological behaviour between the AISI 303 and AISI 310 steels (**Figure 88**). As the less severe micro-cutting case (60 mN), the MnS does not affect representatively the specific energy response from the applied normal load of 80 mN (**Figure 88**). Moreover, a lower microstructural influence is also proved experimentally for the AISI 303 steel (soft particles) (**Figure 89**): the oscillations that appear for this material can be mainly related to the matrix anisotropy, and the sulphide role is secondary.

Figure 88 – Numerical simulation results by applying a normal load of 80 mN – AISI 303 steel (austenitic matrix + MnS) x AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

Sulphides (soft particles on the soft matrix):

(no effect) Depth of penetration = (no effect) V

Nitrides (hard particles on the soft matrix):

↓Depth of penetration = ↓V

and

$$\uparrow E_{sp} = \frac{E_{pd} + E_f + E_c}{\downarrow V}$$

(To be continued)

Figure 88 – Numerical simulation results by applying a normal load of 80 mN – AISI 303 steel (austenitic matrix + MnS) x AISI 310 steel (austenitic matrix + TiN). The modelling outputs are provided in terms of removed volume and depth of penetration x scratch length; and specific energy x scratch length.

(Continuation)

Figure 89 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the heterogeneous soft materials with soft particles. In this condition, the test was carried out with constant normal load of 80 mN ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-cutting (more severe). Also, the scratch length shown in the micrograph is compatible with the graph scales.

Heterogeneous soft material with MnS soft particles: AISI 303 austenitic stainless steel – SEM images (SE).

5.5.3 Soft matrix (homogeneous material)

The numerical simulations of the scratch tests in micro-scale of the homogeneous soft material, AISI 304 steel, provided expected findings in terms of geometrical and tribological parameters. Results in **Figure 90** show that:

- the depth of penetration rises with the applied normal load, which results in increases of the attack angle in accordance with eq. (9) and the abrasive micro-mechanism transition map (**Figure 54**). For each case of normal load, the scratch depth is almost constant along the sliding distance. Additionally, the ploughing part that composes the apparent COF increases for the higher attack angles until attaining the critical angle of the abrasive with the surface, whose condition is above 80 mN according the previous map;

- the removed volume also grows up with the normal load, as a consequence of the penetration depth trend, and there are no significant modifications along the wear track, except when 40 mN are considered. In this case, the dominant mechanism is micro-ploughing, in which the work-hardening influence seems to be less representative, amplifying the removed volume and dropping the specific energy along the scratch length;

- as opposed to the removed volume result, the specific energy tends to be decreased with the incremental normal load, reinforcing the idea that an abrasive micro-mechanism transition occurs in this homogeneous material (LEE, 1993; SABEUR et al., 2013; HE; XU, 2016).

Finally, the numerical results for the AISI 304 steel micro-scratch are in agreement with their experimental results displayed from **Figure 91** to **Figure 94**. The prevailing micro-ploughing is characterized for 40 mN as well as the other austenitic stainless steels; the wedge formation (transition) for 50 mN; the less severe micro-cutting for 60 mN; and more severe micro-cutting for 80 mN. Nevertheless, the annealing twins, anisotropy and material transference by adhesion tend to distance the modelling results from those obtained by experimental test. This outcome does not reduce the importance of the numerical model, and its role in providing valuable local information according to the descriptions carried out in the present topic.

Figure 90 – Results of the numerical modelling for the abrasive-AISI 304 steel contact pair: apparent coefficient of friction and depth of penetration as a function of sliding distance; removed volume and specific energy as a function of scratch length.

Figure 91 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the homogeneous soft material (AISI 304 steel). In this condition, the test was carried out with constant normal load of 40 mN ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-ploughing. Also, the scratch length shown in the micrograph (SEM/SE) is compatible with the graph scales.

Figure 92 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the homogeneous soft material (AISI 304 steel). In this condition, the test was carried out with constant normal load of 50 mN ($10 \mu\text{m s}^{-1}$). A transition from micro-ploughing to micro-cutting has occurred. Also, the scratch length shown in the micrograph (SEM/SE) is compatible with the graph scales.

Figure 93 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the homogeneous soft material (AISI 304 steel). In this condition, the test was carried out with constant normal load of 60 mN ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-cutting (less severe). Also, the scratch length shown in the micrograph (SEM/SE) is compatible with the graph scales.

Figure 94 – Tribological responses of the experimental scratch tests in micro-scale (local apparent COF, removed volume and specific energy) for the homogeneous soft material (AISI 304 steel). In this condition, the test was carried out with constant normal load of 80 mN ($10 \mu\text{m s}^{-1}$). The dominant abrasive micro-mechanism was micro-cutting (more severe). Also, the scratch length shown in the micrograph (SEM/SE) is compatible with the graph scales.

5.6 Experimental analyses of factors that influence the cutting material responses

5.6.1 Surface finishing

Scratch tests and 2D numerical simulations were conducted to evaluate the mechanical behaviour of AISI 303 stainless steel under abrasive action, considering two types of surface finishing: mechanical and electrolytic polishing. The results were published in Seriacopi et al. (2016).

In terms of Vickers micro-hardness, mechanical polishing resulted in (206.0 ± 1.1) HV_{0.1}, whereas this property was (194.4 ± 4.6) HV_{0.1} for the electrolytic polished sample, which confirms the strain-hardened layer in the first one.

The results of the surface topography characterization are shown in **Figure 95**. Based on this analysis, topography parameters were calculated and are displayed in **Table 33**. These values indicate that the electropolished surface is rougher, has a higher density of peaks, and sharper asperities than the mechanical polished surface. Bhuyan et al. (2005) reported that a more efficient control of the electrolytic polishing parameters, such as electrolyte temperature, current density, polishing time and pulsed current application, is necessary to adjust the flatness and to avoid the majority of pitting formation on these surfaces.

Predicted results from the literature (HOKKIRIGAWA; KATO; LI, 1988) indicate that the transition from ploughing to cutting occurs at 10 mN. In Hokkirigawa; Kato; Li (1988), the authors developed experimental tests with several materials, among them the AISI 304 steel. **Figure 96** indicates this transition has occurred from 50 to 60 mN for the AISI 303 steel electropolished specimen (EP) and from 40 to 50 mN in the AISI 303 steel mechanically polished specimen (MP). The cutting has prevailed at 60 mN for EP and 50 mN for MP. As shown in **Figure 97**, SEM micrographs confirmed the predominance of ploughing for 50 mN in EP and 40 mN in MP. The cutting has started prevailing at 60 mN for EP and 50 mN for MP.

Figure 95 – Topography results obtained by optical interferometry for AISI 303 stainless steel with different surface finishing.

Table 33 – AISI 303 specimens surface topography parameters before scratch tests.

Parameter	Description	Mechanical Polishing	Electrolytic Polishing
$S_a(\mu\text{m})$	Arithmetic Mean Height	0.014 ± 0.001	0.117 ± 0.042
$S_q(\mu\text{m})$	Root Mean Square Height	0.021 ± 0.001	0.187 ± 0.051
S_{sk}	Skewness	0.196 ± 0.282	0.996 ± 0.452
S_{ku}	Kurtosis	8.12 ± 0.85	14.7 ± 3.5
S_{dq}	Root Mean Square Gradient (Surface Slope)	0.0174 ± 0.0008	0.156 ± 0.002
S_{pc}	Arithmetic Mean Peak Curvature (μm^{-1})	0.080 ± 0.003	1.02 ± 1.43

Figure 96 – Apparent Coefficient of Friction in function of normal force in the mechanical polishing (MP) and electrolytic polishing (EP) of the AISI 303 steel specimens.

Numerical results in **Figure 98** allowed confirming two main behaviours:

(i-) electropolished (EP) surfaces have higher mass removal than mechanical polished (MP), which is justified by the presence of the strain-hardened layer (**Figure 99a,b**);

(ii-) MnS inclusions, with different morphologies, played a role as stress concentrators, providing higher mass loss. This fact supports the better machinability of AISI 303 stainless steel (heterogeneous) compared with the homogeneous one. **Figure 100** shows the chips formed during the abrasive process, which are consistent with the values of the mass removed considering the different normal forces.

Figure 97 – Abrasion mechanisms transitions characterized by SEM (secondary electrons).

Figure 98 – Numerical results of removing mass by abrasion, obtained from surface finishing and microstructure features variation.

**Mass Removal Scale in the
Studied Materials**

Figure 99 – Effect of the surface finishing in terms of surface integrity in the mass removal: numerical results.

Figure 100 – Effect of the microstructure in terms of surface integrity in the mass removal: numerical results.

5.6.2 Crystallographic orientation

The EBSD analysis of the electropolished AISI 303 steel was performed to characterize its crystallographic orientation, and to test the hypothesis that it could affect the tribological behaviour of austenitic stainless steels during scratch tests. The indexed pattern obtained by EBSD of this material for the pre-test condition is provided in **Figure 101a**. It is possible to verify the presence of a face-centred cubic (FCC) lattice, whose grains are crystallographic oriented as indicated by the colours on the right side. This pattern is consistent with the results presented in the literature for the AISI 304 steel (LE PÉCHEUR et al., 2010), as can be seen in **Figure 101b**. In addition, annealing twins can be observed in the AISI 303 and 304 steels by means of the EBSD maps in **Figure 101**.

The EBSD map and the indexed pattern of the AISI 303 steel after the scratch test in micro-scale are shown in **Figure 102a**. These images suggest a misorientation and rotation of the analysed grains due the abrasive action over the material, which causes its plastic deformation. Particularly, the profile related to the orientation difference based on the test line (**Figure 102c**) indicates a low angle variation within a single grain. As pointed out by Dieter (1986), one of the main influences of the twins on the plastic deformation of the metallic single crystals is the orientation changes provided by them. These defects can allocate new slip systems in orientations that benefit the occurrence of the additional dislocation motion, when a stress is applied on the material.

The excessive plastic deformation of the AISI 303 grains tends to originate Thompson tetrahedrons, as observed in **Figure 102b**. Besides, a strain induced martensitic transformation may have occurred for the more severe micro-cutting condition (100 mN) seen in this figure. In this case, the material removal of the AISI 303 and 304 steels is lower than the AISI 310 steel (**Figure 62a**), which can be considered an indicative for martensitic transformation in the first two materials since the AISI 310 does not tend to form martensite during its abrasion (SOUZA, 2006). Gahr (1987) corroborates this assumption, indicating that the austenitic structures can show reduced values of the removed volume as a function of capacity to transform martensite induced by plastic deformation throughout abrasive processes.

Figure 101 – EBSD maps and their indexed patterns: (a-) AISI 303 steel prepared by electrolytic polishing; and (b-) literature reference for the AISI 304 steel.

(a-)

(b-)

Reference: (LE PÉCHEUR et al., 2010)

Figure 102 – EBSD maps and indexed patterns for the post-test specimen of the AISI 303 steel (a-) pattern with the crystallographic orientations; and (b-) misorientation levels of the grains; (c-) misorientation profile within the grain (test line in b).

(a-)

(b-)

(To be continued)

Figure 102 – EBSD maps and indexed patterns for the post-test specimen of the AISI 303 steel (a-) pattern with the crystallographic orientations; and (b-) misorientation levels of the grains; (c-) misorientation profile within the grain (test line in b).

(c-)

(Continuation)

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5.7 Final map of the abrasion resistance: mechanical behaviour x abrasive micro-mechanisms

Firstly, a graph containing the main tribological results derived from the numerical simulation by FEM is shown in **Figure 103** for all the materials tested here. A good exponential fit of the material removal data as a function of the average COF can be observed in this plot. For the tribological conditions studied, the removed volume tended to increase with the friction coefficient. The differences related to the material energies, particularly the frictional and cutting, can be responsible for providing an initial tribological ranking of the materials analysed during the scratch tests.

Figure 103 – Numerical results of the material removal of the materials studied as a function of COF: summary of the tribological data obtained by using computational modelling.

Secondly, a quantitative plot can be achieved to group the information about the microstructure, which affects the mechanical and damage behaviour of materials, and the mechanisms of abrasion, which determine the material removal resistance. As depicted in **Figure 104**, previous works (MURRAY; MUTTON; WATSON, 1979; GAHR, 1987) proposed schemes to combine these data of the abrasion resistance as a function of hardness, or the relation between the deformed material hardness and

material removal factor (f_{ab}), in which the denominated deformed material hardness (post-scratch test) can be calculated by eq. (32).

$$H_{def} \approx \frac{2W}{5hB} \begin{cases} B = \sqrt{h(2R - h)} & (\text{spherical part} - \text{abrasive}) \\ B = h \cot \theta & (\text{conical part} - \text{abrasive}) \end{cases} \quad (32)$$

Where: H_{def} is the hardness of the deformed material by the abrasion effect; W is the normal load; h is the depth of penetration; R is the abrasive tip radius; and θ is the attack angle.

Based on prior ideas, a map is designed here to join input and output variables of the numerical simulation, creating a relationship of the cutting resistance - (total removed volume / scratch length)⁻¹ – and the evaluated materials (H_{def}) added to the abrasive micro-mechanisms prevailing on them. The attack angle (θ) can be introduced into the analyses, in which it helps to define the micro-mechanism transitions, instead of applying f_{ab} due to the several uncertainties aforementioned. The quantitative graph in **Figure 105** confirms the linear behaviour between the abrasion resistance and the deformed material hardness (MURRAY; MUTTON; WATSON, 1979), allowing observing that:

- (i-) the linear functions with higher derivatives (slopes) correspond to the dominant ductile behaviour in the abrasion, in which the dominant effect of the austenitic matrix of the AISI 303 and 304 stainless steels supports the frontier definitions;
- (ii-) the linear function with the lowest derivative (slope) is designated for the dominant brittle effect on the abrasion represented by the heterogeneous hard material, which, for the most of cases, presented the prevailing influence of the brittle precipitates (NbC) on the tribological results.

Figure 104 – Some proposals of the literature to investigate the abrasive wear resistance for distinct materials.

(a-) Linear behaviour of the volumetric abrasion resistance as a function of ratio between the H_{def} and f_{ab} for different metallic materials (GAHR, 1987).

(b-) Schematic representation of the abrasion resistance x material hardness, developed by Murray; Mutton; Watson (1979), which indicated regions for the occurrence of dominant ploughing and cutting in steels, also including the fragmentation for brittle materials.

Figure 105 – Quantitative map developed from the numerical results of the present thesis, in which frontiers can be delineated to determine the dominant ductile or brittle features (mechanical and damage behaviour), and the prevailing abrasive micro-mechanism: abrasion resistance as a function of H_{def} /Attack Angle.

Figure 105 – Quantitative map developed from the numerical results of the present thesis, in which frontiers can be delineated to determine the dominant ductile or brittle features (mechanical and damage behaviour), and the prevailing abrasive micro-mechanism: abrasion resistance as a function of Hdef/Attack Angle.

As can be seen in **Figure 105**, the transition region between ductile and brittle includes the AISI 310 stainless steel, which tends to be closer to the brittle frontier than the other soft materials due to the influence of its brittle precipitates (TiN). Moreover, the AISI H13 steel is positioned in the transition of the dominant ductile-brittle frontiers. This material tends to be closer to its corresponding heterogeneous material because of this similar hard martensitic matrix.

The prevailing abrasive micro-mechanisms can be extracted from the map in **Figure 105** for the following conditions:

- (1) the micro-ploughing can be favored when the abrasion resistance is high and the attack angle is small ($\uparrow H_{def}/\theta$);
- (2) in turn, the micro-cutting can occur in the lower abrasion resistance conditions and when the attack angle is increased ($\downarrow H_{def}/\theta$).

In conclusion, **Figure 105** allows extrapolating the approach for a wide range of materials under abrasive action, e.g. for the purely brittle materials, the linear function will have the lowest slope, which indicates a low abrasion resistance being more susceptible to micro-cutting.

6 CONCLUSIONS AND FINAL REMARKS

The major contribution of the present thesis consisted in elaborating a computational model to study the abrasion effects in micro-scale by applying the Finite Element Method. Based on this purpose, the methodology and microstructural analyses conducted subsidise future designs of metallic alloys in terms of their cutting resistance, which can be correlated with abrasive manufacturing processes.

From the numerical procedure developed, which was experimentally validated, a combination of microstructures was investigated and their mechanical and damage properties were specified to assess the responses of the material removal due to abrasion. The characterization of the abrasive micro-mechanisms was approached in terms of local specific energy and removed volume in parallel to the apparent coefficient of friction. In turn, the specific energy was considered: frictional, strain and cutting.

Regarding the experimental step of the microstructural and mechanical characterization of the materials, which are performed to obtain input parameters of the numerical simulation, the following aspects can be pointed out:

(i-) microstructural characterization by SEM/EDX: the classification into homogeneous and heterogeneous materials was conducted based on the presence of second phase particles (sulphides, nitrides and carbides). For the homogeneous soft material, grains with different crystallographic orientations and annealing twins were evidenced, though;

(ii-) characterization by X-ray micro-tomography: the hexahedral elements generated by voxels, with compatible sizes to the micro-tomography resolution, were used, since they reduced convergence problems of the contact (abrasive-slab) and allowed more precise calculations of the removed volume by abrasion (damage criterion + element deletion). By means of this technique, the discretization of the cubic carbides and nitrides, and elongated sulphides are possible;

(iii-) instrumented indentation (nanoindentation): the measurement of mechanical properties of the phases of the stainless steels varied with the applied normal load, mainly due to the deformed layer formed throughout the mechanical polishing. As a decision criterion, the ratio between elastic modulus and indentation hardness (E/H), which is an indicative parameter of the cutting efficiency were used. Phases with a higher E/H ratio enable the material removal and promote the micro-cutting were selected, whilst phases that minimize the material removal effects were assigned to the low E/H ratios;

(iv-) conventional macro- and micro-hardness of the homogeneous soft material: results agree with the literature. The macro-hardness is statically equal for the different tested loads. However, the micro-hardness is significantly different depending on the applied load, which allowed estimating the thickness of the deformed layer resulting from the mechanical polishing;

(v-) constitutive equation for phases plasticity: the employment of the parameters of the Hollomon model (n and K_s) for the austenitic and the martensitic matrices were consistent with the results of the AISI H13 tensile and AISI 304 nanoindentation tests. No restrictions were observed related to low strain rates of the scratch tests ($10^{-2} \sim 10^{-1} \text{ s}^{-1}$).

Focusing on the overall numerical results, the conclusions can be pointed out as follows:

(vi-) Apparent COF for the abrasive-homogeneous material contact pair: the numerical and experimental mismatches found were due to the adhesion effect, which was not specified in the numerical model. In the case of the austenitic stainless steel, the differences were intensified due to the influences of anisotropy, grain boundaries and twinning, which were not considered on the modelling;

(vii-) Apparent COF for the abrasive-heterogeneous material contact pair: the hard precipitates tend to have prevailing effect over the matrix regarding the apparent COF, whereas the matrix effect is dominant of the soft precipitates in the COF behaviour.

Hard second phase particles provide the decrease of the average COF when compared to the results of the homogeneous matrices. For the heterogeneous materials, the strain energy is reduced because the plastic deformation effects of the hard second phase particles are minimum or non-existent. Therefore, the strain energy had a more pronounced influence than the fracture energy regarding friction losses;

(viii-) Abrasive micro-mechanisms were predicted based on the numerical simulation outputs of apparent COF as a function of attack angle. These numerical results did not support the Hokkirigawa; Kato; Li (1988) map (Penetration x Hardness);

(ix-) Average depth of penetration and removed volume: Numerical results are in accordance with the experimental results. Differences can be assigned to the mesh effects;

(x-) The relation between the depth of penetration and normal load is linear, in which the depth tends to increase linearly with the load, providing high correlation coefficients. Hard material presented a better fitting than soft materials, probably due to the work-hardening;

(xi-) There is not a remarkable micro-mechanism transition using the approach of the dimensional wear coefficient as a function of normal load. The alteration of the order of magnitude, despite occurring in some cases, seems to be slight. The same result was experimentally observed by Franco (2015), which indicated a predominance of a micro-mechanism in a single scratching, and sudden transitions were not observed;

Concerning the local numerical results, the discussed aspects were divided into:

(xii-) Hard second phase particles: decreased the local depth of penetration and, consequently, caused the reduction of local removed volume and an increase of the local specific energy. When the depth of penetration reached their critical size (often for the more severe micro-cutting), particles were pulled out or fragmented on the material matrix, increasing the removed volume and modifying the amplitude of the specific energy;

(xiii-) Soft second phase particles: considering the studied range of the normal load, they followed the matrix material removal behaviour, but these particles can show fluctuations in terms of the local specific energy up to their more severe abrasion case;

(xiv-) Stress triaxiality: the analyses utilizing this output of the numerical model seem to be promising to determine the ductile failure mode. The pursuit of the damage parameters can be conducted interactively based on the scratch tests in micro-scale and microstructural characterizations;

(xv-) Map of the abrasion resistance x deformed hardness/attack angle: this map contains two main types of information – the first one is the division between dominant ductile or brittle effect on the microstructure considering the abrasion; and the second one concerns in delineating frontiers of the abrasive micro-mechanisms (from mild to severe).

Regarding the experimental results in terms of surface finishing and crystallographic orientation, the conclusive issues can be indicated as follows:

(xvi-) Mechanical polished (MP) specimens presented a lower mass removal than the electropolished (EP) specimens as a result of their deformed layer. Nevertheless, the abrasive micro-mechanisms of the EP condition, characterized as a function of apparent COF for several normal loads, can be postponed in relation to the MP condition due to different deformation plastic levels caused by the roughness influence;

(xvii-) After the scratch tests, the EBSD maps suggest a grain misorientation (low angle) in the FCC lattice that seems to be promoted by the material plastic deformation throughout the scratching.

7 FUTURE WORKS

(a-) To include crystallographic orientation effects, by means of the different mechanical properties of the grain and recrystallization FEM sub-routines, and adhesion influence as a component of the friction coefficient (contact specification) on the modelling of scratch test in micro-scale to refine the numerical analyses;

(b-) To add the thermal effect to verify the response of the material removal and specific energy under controlled temperature. The plastic deformation that can be converted into heat can be considered in this evaluation. Also, a possible deformation induced martensitic transformation can be studied by strain and temperature fields;

(c-) To further investigate the relation between the local stress triaxiality and the sub-surface ductile failures to generate a map that contains their parameters. Later, to verify the several conditions of the hydrostatic pressure to rank optimal design conditions of the heterogeneous materials submitted to the abrasive processes;

(d-) To better understand and explore the elastic recovery as technological contribution, allowing a link between microstructural influence on the macroscopic analyses;

(e-) To conduct a multiscale analysis, from micro-scale (scratch test studies here developed) to macro-scale (machining processes with undefined tool edge geometry – e.g. grinding), in which the link between the scales could be built using tribological tests with multiple abrasives, such as pin-on-discs tests applying abrasive papers as counterbodies.

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ANNEX A – Poster presented at the 2nd Conference on Abrasive Processes (ICAP 2014).

2nd International Conference on Abrasive Processes – ICAP 2014

Numerical and experimental analyses of scratch tests on two-phase microstructure of tool steels

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Abstract

Abrasive wear is one of the main damage mechanisms leading to tool steel failure during forming processes^[1]. The present study has investigated the effects of abrasion micromechanisms (microploughing and micro-cutting) on the microstructure of a two-phase tool steel. Experimental scratch tests were conducted by sliding a diamond tip over a tool steel slab considering different values of normal load. The same procedure was simulated using the Finite Element Method, allowing the analysis of the interaction between the indenter tip and the microstructural components, as well as the analysis of the effect of the precipitates. The simulations considered elastic precipitates immersed on an elastic-perfectly plastic matrix. Results have indicated the effects of the second phase particles in terms of tangential force and depth of penetration (experimental) as well as failure mechanisms (numerical). Therefore, both experimental and numerical analysis could be considered complementary to understand the abrasive wear of two-phase materials.

Objective: To evaluate abrasion mechanisms during the scratching test of heterogeneous and homogeneous tool steels, varying primarily the normal load.

Experimental and Numerical Procedures

□ Scratch Tests

❖ **Conical Tip:**
5 μm radius (experimental and numerical)

❖ **Rockwell C Tip:**
200 μm radius (only experimental)

❖ **Materials:**
Diamond (rigid abrasive) and Tool steels (slab).
Homogeneous: AISI H13 steel;
Heterogeneous (two-phase): martensitic matrix and carbides (mainly niobium carbides).

- ❖ **Normal loads:**
50 -500 mN (conical tip)^[2] and 10-30 N (Rockwell C tip).
- ❖ **Scratch length:**
400 μm (experimental) and 200 μm (numerical).
- ❖ **2-D Numerical model:**
The matrix was considered elastic-plastic. Damage model and, element deletion were taken into account for the two-phase; aiming the abrasive wear study.

Results

□ Experimental:

❖ Homogeneous material - AISI H13 tool steel:

The transition lines of abrasion mechanisms graphics were based on Hokkingawa and Kato [3].

50 mN - Mechanism Transition

100 mN - Cutting

❖ Influence of microstructural features:

❖ Comparison between homogeneous and heterogeneous materials:

□ Numerical:

Chip formation mechanism during cutting: When the scalar stiffness degradation is unity, the material failure will occur promoting debris such as discontinuous chips agreement with the experimental results.

(1) Second phase failure - abrasive movement is affected by the hard particles. This fact allowed increasing the tangential force to overcome the barrier.

(2) After the second phase failure, the depth of penetration was increased because there were the contact area effects. The scratching sequence favors the damage accumulation in the matrix resulting again in the debris formation (chip) during the material cutting.

Results obtained by FEM agree with experimental information: The depth of penetration was reduced in the points corresponding to the second phase particles.

Preliminary Conclusions

> In this work, it was possible to simulate and compare the abrasion behavior between two-phase steel and AISI H13 tool steel.

> In contact with the diamond tip, the average coefficient of friction for heterogeneous material was lower than for homogeneous material. The second phase decreases the tangential force, resulting in a lower average value.

> The mass loss was increased with the normal load (both materials). Heterogeneous material presented lower mass loss than H13 steel when abrasion mechanism was cutting.

Acknowledgements

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ANNEX B – Poster presented at the 20th International Conference on Wear of Materials (WOM 2015).

Numerical and Experimental Analyses on the Influence of Two-phase Carbides on the Abrasive Wear Behaviour of Tooling Steels

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1. INTRODUCTION

In abrasive wear of tool steels, the **microstructure** affects directly the wear behavior. **Second phase particles** are commonly presented in a steel matrix, allowing the control of **abrasive wear mechanisms** and improving also the **abrasion resistance** of the material [1].

During the production of tool steels, some **multi-phase carbides** can precipitate, promoting specific wear behaviors due to their **different mechanical properties** (focusing on **hardness x toughness**).

Aim of this work: To investigate the influence of two-phase carbides on abrasion of tool steel by both experimental and numerical scratch tests. The qualitative analysis of the abrasive wear is conducted based on variables such as tangential load and depth of penetration.

2. EXPERIMENTAL AND NUMERICAL PROCEDURES

- EXPERIMENTAL

- Scratch tests in microscale (TI 950 Tribolindenter® - Hysitron)
- Constant normal load – range from 10 to 500 mN
- Scratch length: 400 µm
- Abrasive: diamond conical tip with 5 µm radius
- Slab: AISI H13 tool steel – hard matrix with two-phase carbides
- Mechanical properties of carbides: instrumented indentation tests - 9 mN and Berkovich indenter. An algorithm [2] was applied for obtaining the stress-strain curve of these phases.

- NUMERICAL SIMULATION (3-D model)

- Finite Element software: Abaqus® / Explicit (version 6.13)
- Constant normal load – between 10 and 500 mN
- Scratch length: 77 µm
- Slab dimensions: 95 x 95 x 65 [µm]
- Rigid Abrasive and Elastic-Plastic properties specified for the matrix and carbides (slab)
- Modified ductile damage model
- Failure properties of these phases were estimated based on literature.

Phase	Fracture Toughness [MPa.m ^{1/2}]	Energy Release Rate [J.m ⁻²]	Reference
Carbide - core (less brittle)	3.8	45.0	[3,4]
Carbide - shell (more brittle)	2.2	13.7	[4,5]
Matrix specify as AISI H13 tool steel	20.0	1,770.0	[6]

• Results of the scratch tests:

• Abrasive wear mechanisms:

3. RESULTS

- Characterization by SEM** (backscattered electron image and EDS analysis): a **brittle behavior** in the carbide core (**rich in Ti-Nb_{1-x}-C**) was **less pronounced** than in the carbide shell (**rich in NbC**), during the scratch tests.

- Instrumented Indentation Results:** Although the **hardness of the both carbides are similar**, **black core** is more resistant than **white shell** at high strains, which were verified due to scratching.

Carbide Region	Elastic Modulus [GPa]	Vickers Hardness [GPa]	Yield Stress [GPa]	Strain-Hardening Coefficient
Rich in Ti _x Nb _{1-x} -C	420.17	23.95	7.69	0.64
Rich in NbC	382.56	24.14	15.06	0.25

4. CONCLUDING REMARKS

- The results for two-phase carbides are in agreement with the behavior reported by Chen and Zhao [7] for Ti_xNb_{1-x}-C carbides. An **increase in Nb** and a **decrease in Ti** promotes **carbide brittleness**. Thus, **crack nucleation is expected at the shells**.
- Results indicate that **less brittle cores can hold cracks that propagate through more brittle external shells**, while more brittle cores promote the complete failure of the carbide. These results are coherent with the literature [3-5], which reports that higher energies are necessary to fracture the Ti-rich cores.
- Numerical and experimental results allowed discussion of the **abrasive wear mechanisms** as a function of applied **normal load**.

5. ACKNOWLEDGEMENTS

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ANNEX C – The results reported for the AISI H13 steel tensile tests at room temperature, elaborated by the Centre for Development and Characterization of Materials (CCDM) – In Portuguese.

CERTIFICADO MET14-000070

Cliente: Fundação de Apoio à Universidade de São Paulo - FUSP

Contato: Erika Fernanda Prados

Endereço: Avenida Afrânio Peixoto, 14 - Butantã

Cidade: São Paulo

CEP: 05507-000

OS: 7190/7465-3

Estado: SP

Data de recebimento das amostras: 27/06/2014

Período de realização: 27/06/2014 – 18/07/2014

ENSAIOS DE TRAÇÃO EM AMOSTRAS DE AÇOS

1 – OBJETIVOS

Determinar as propriedades mecânicas em tração de amostras de aço.

2 – CONSIDERAÇÕES INICIAIS

Foram recebidas para análises no CCDM duas amostra de aços contendo três corpos de prova (cp's) para ensaios na temperatura de 450°C e três cp's para ensaios à temperatura ambiente de cada amostra, totalizando 12 corpos de provas usinados. As amostras foram identificadas como mostra a Tabela 1.

Tabela 1 – Identificações das amostras.

Identificação do CCDM	Identificação do Cliente
MET140590	Amostra H13 – 3 CP's –
	Temperatura ambiente
	Amostra H13 – 3 CP's –
	Temperatura 450°C

3 – MÉTODOS

Os ensaios de tração, à temperatura ambiente, foram realizados em uma máquina universal de ensaios modelo 5585H – INSTRON, com capacidade para 250kN e de acordo com a instrução de trabalho IT-META 213 rev.009. Os ensaios de tração à quente (temperatura 450°C) foram realizados no mesmo equipamento, utilizando um forno Instron modelo SF-16 2230 acoplado.

O objetivo do ensaio de tração é a determinação das propriedades mecânicas da amostra, por meio de sub-rotinas de cálculo pré-existentes no *software* (INSTRON *Bluehill*) e por meio de verificações dimensionais antes e após a realização do ensaio. As propriedades de interesse são descritas na Tabela 2; os parâmetros utilizados são informados na Tabela 3.

Tabela 2 – Propriedades obtidas no ensaio de tração.

Propriedade	Equação	Descrição
Tensão de escoamento $\sigma_{0,2\%}$	$\sigma_{esc} = \frac{F_{esc}}{A}$	<p>A tensão de escoamento é definida pela razão entre a força de escoamento e a área da seção transversal.</p> <p>Seu valor é obtido na interseção de uma reta, paralela à região elástica da curva obtida, traçada a partir de 0,2% de deformação, com a curva do ensaio em si.</p>
Limite de Resistência à Tração σ_{LRT}	$\sigma_{LRT} = \frac{F_{max}}{A}$	<p>O limite de resistência à tração é a tensão máxima atingida durante o ensaio, qual seja a razão entre a carga máxima atingida e a área da seção transversal original.</p>
Alongamento percentual total na fratura A	$A = \left(\frac{l_f - l_0}{l_0} \right) * 100$	<p>O alongamento total na fratura é uma medida percentual da relação entre a deformação axial, decorrente do ensaio, e o comprimento útil original.</p> <p>Na prática, marca-se o comprimento útil no corpo de prova e, após a realização do ensaio, obtém-se o valor do comprimento final, alimentando estes valores na equação.</p>

Tabela 3 – Parâmetros do ensaio de tração.

	Comprimento útil – L ₀ (mm)	Comprimento Paralelo – L _c (mm)	Taxa de deformação até o escoamento (mm/min)	Taxa de deformação após o escoamento (mm/min)
Tração Temperatura 450°C	24 ^B	30 ^B	0,2 ^C	0,4 ^C
Tração Temperatura ambiente	24 ^A	30 ^A	1,5 ^D	1,5 ^E
Referência	24 ^{A,B}	30 ^{A,B}	$0,012 - 0,018 \frac{(\frac{mm}{mm})}{min}$ _D	$0,05 - 0,5 \frac{(\frac{mm}{mm})}{min}$ _E
			$0,003 - 0,007 \frac{(\frac{mm}{mm})}{min}$ _C	$(0,04 - 0,06) \times L_c$ $\frac{(\frac{mm}{mm})}{min}$ _C

^A ASTM E8/E8M-11; item 6.4. ^B ASTM E21-09; item 7.3. ^C ASTM E21-09; item 9.6. ^D ASTM E8/E8M-11; item 7.6.3.3

^E ASTM E8/E8M-11; item 7.6.4.

4 – RESULTADOS

Os resultados para as propriedades de tensão de escoamento, limite de resistência à tração, alongamento percentual e redução percentual de área estão expressos na Tabela 4. A Figura 1 exibe as curvas tensão-deformação representativa das amostras.

Tabela 4 – Resultados obtidos nos ensaios de tração da amostra MET140590.

H13 MET140590	Tensão de Escoamento $\sigma_{0,2}$ (MPa)	Limite de Resistência (MPa)	Alongamento em 24 mm (%)
CP1 – T. Amb.	1421	1629	6,8
CP2 – T. Amb.	1395	1601	5,8
CP3 – T. Amb.	1422	1643	6,2

MET140590 – H13 – Frio.

Figura 1 – Curva tensão-deformação representativa das amostras.