Study on Microstructure Characteristics of Steel in Two-Body Abrasive Wear

By

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Submitted in fulfilment of the requirements for the degree of

Doctor of Philosophy

Deakin University

August, 2016
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Though the following dissertation is an individual piece of work, this accomplishment was never possible without the guidance and insights of several people.

First and foremost, I would like to extend my heartfelt gratitude to Prof. Peter Hodgson, for offering me a Ph.D. position (Higher Degree by Research) at Institute for Frontier Materials, Deakin University. His unprecedented enthusiasm for research was quite infectious and motivational during my research career at IFM. I appreciate his contributions of time, ideologies, support for conferences and most importantly, providing me with the intellectual freedom to conceive my research.

I express my deepest sense of gratitude to my mentor and guide Dr. Hossein Beladi who has been my pillar of support during the tough times of my Ph.D. I can still remember his golden words, “If I could do this, then definitely you can”. I often envied his modesty and natural ability to meticulously perform a task with no fuss. My writing and presentation skills were greatly honed by his humongous experience and patience towards all my trivial queries. During my research stint, I have failed miserably on numerous occasions, but he was always there to pick me up. I am grateful for all his indefatigable encouragement and unparalleled belief in me.

Special thanks to Dr. Ilana Timokhina, Dr. Pavel Cizek and Dr. Qi Chao for their invaluable support and guidance in advanced characterization techniques. Also to the hours of non-scientific chat with Pavel, that covered from Dostoyevsky to Don Quixote.

I would also like to thank Dr. Daniel Fabijanic, Dr. Alireza Ghaderi and Prof. Mathew Barnett for their significant contributions and research inputs to this dissertation.

I would like to acknowledge all the technical officers and research staff at IFM, Deakin University. Especially, David Gray, Linton Leigh, Andrew Sullivan, Rosey van Driel, Dr. Huaying Yin, Rob Pow, Mohan Setty, Ivi Cicak, Helen Woodall, Marion Wright and Magnolia Beer. Special thanks to Darlene Barnett and Steve Atkinson for their high tolerance levels in signing my numerous ‘out-of-hours’ forms and accepting me as a student representative in IFM OH & S committee.
I am very grateful to Marilyn Fisher for her wholehearted support and co-operation. I am indebted to Jane Allardyce, Technical and Academic Editor for patiently correcting (grammatically!!!) my thesis and stressing the need for scientific words instead of poetic ones.

I am indebted to Anuradha Gupta, Nitin Sharma and Pawan Solanki who felt that I would be worthy addition to Deakin University. I place on record, my sincere thanks to the International Student Advisors, Helen Nicholls-Stary and Bridget Becker for making me feel home. A debt of gratitude to the Division of Student Life at Deakin for offering me the roles of ‘International Orientation Coordinator’ and ‘Senior Writing Mentor’. My time at Deakin was made more memorable due to the many friends that have become a part of my life. My special thanks extends to Murugesan, Varsha, Harish, John, Sri Balaji, Lakshmanan, Thirinath, Ajeesh, Ehsan, Emma, Emily and Marjorie for their warmth and encouragement.

Last but the least, I would like to thank my family for their unconditional love and support. To my brother, who would never let me wander alone in the dark. A big hug to my mom, for being a classic example of a tough and lovable cookie. Most of all, I owe this one to my dad, a hero, who showed me that the world is not full of rainbows. Thanks to the almighty for all the blessings throughout this Ph.D. journey.
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Abstract

A detailed investigation was conducted on the major components, namely ferrous microstructure and abrasive environment of two-body abrasive wear. A CSM high temperature pin-on-disc tribometer was extensively employed to simulate two-body abrasive wear. The dynamic two-body sliding abrasive wear induced simultaneous changes occurring both in the material (i.e. microstructure) and characteristics of the abrasive environment (i.e. deterioration of abrasive particles). Firstly, a fully pearlitic microstructure was subjected to two-body abrasive wear test under two different abrasive environments (e.g. silica and alumina). This study revealed the significant impact of abrasive particle characteristics (e.g. particle size, and density) in the process of material removal during abrasion. The specific wear rate of the pearlitic microstructure and the abrasive particle deterioration mechanisms were greatly influenced by their particle size, irrespective of the particle type. Attrition, shelling and fragmentation were some of the dominant material removal mechanisms observed in this study. In addition, interrupted two-body abrasive wear tests demonstrated the significance of abrasive particle density (i.e. number of particles/µm²) in determining their abrading efficiency for a given sliding distance.

Followed by this, the influence of microstructure constituents in two-body abrasive wear was investigated. In this study, four microstructures (e.g. bainite, pearlite, martensite and tempered martensite) with similar hardness levels displayed a distinct response towards the abrasive behaviour. Despite similar hardness levels, the unique friction coefficient curve of the microstructures was ascribed to the characteristics of the microstructure constituents. The study revealed that the multi-phase microstructures (bainite and pearlite) revealed better abrasion resistance than the single-phase microstructures (martensite and tempered martensite). Moreover, the two-body abrasive wear induced significant microstructural changes (i.e. severe deformation) in their sub-surface layers (i.e. region beneath the abraded surface). Surface profile and topography techniques highlighted the quantum of material loss in the microstructures. The distinct material removal mechanisms (e.g. ploughing and cutting) in the microstructures were observed through exclusive single wear track analysis. In general, microstructures with a combination of brittle and ductile metallurgical phases exhibiting work-hardening behaviour was more beneficial in
abrasive conditions. However, there was a need to identify a laboratory abrasive wear test that can simulate the actual industrial test conditions.

To address this, a high strain abrasive wear testing was chosen, where a robust indenter abraded the microstructure under the action of a normal load, which resulted in a groove. This isolated the effect of abrasive environment characteristics (i.e. deterioration of abrasive particles) in the abrasion, thereby focussing on the microstructure response. This resulted in a thorough understanding of the material removal mechanisms occurring in microstructures during abrasion. The groove characteristics (i.e. degree of penetration, \(D_p\)) were significantly influenced by the microstructure constituents and the normal load. As expected, multi-phase microstructures (bainite and pearlite) demonstrated better abrasion resistance than the single-phase microstructures (martensite and tempered martensite). In general, the microstructures experienced ploughing material removal mechanism at low loads (i.e. 200 N to 500 N), whereas, cutting was more dominant at relatively high loads (above 1000 N). Additionally, a positive correlation between the work-hardening behaviour and the abrasive wear resistance of microstructures was observed through the sub-surface layer characterization. This proved to be the driving force for the subsequent study in the abrasive wear behaviour of ultra-high strength bainitic steels (also known as nanobainitic steel). The presence of retained austenite in their microstructure matrix is known for superior work-hardening behaviour through TRansformation Induced Plasticity (TRIP) effect.

Therefore, a high-carbon high alloy steel was subjected to isothermal bainitic transformation at a temperature range of 200-350°C to produce fully bainitic microstructures consisting of bainitic ferrite and retained austenite. A decrease in the transformation temperature resulted in a significant microstructural refinement (i.e. from \(~300\) nm at 350°C to \(~60\) nm at 200°C) and change in retained austenite morphology (i.e. from film+blocky to film only). Furthermore, the characteristics of retained austenite (e.g. volume fraction, morphology and the carbon content) triggered a range of work-hardening behaviour (i.e. TRIP effect) during two-body abrasive wear test. However, fully bainitic microstructures that were formed at low transformation temperatures (i.e. FB-200°C and FB-250°C) displayed superior abrasion resistance due to the presence of the mechanically stable film morphology retained austenite in their microstructure matrix. Blocky retained austenite often resulted in an early onset
of TRIP effect, leading to the formation of coarse blocky martensite, which was more vulnerable to crack initiation and propagation. The detrimental effect of block austenite was confirmed by conducting a comparative study with the abrasive wear behaviour of a fully pearlitic microstructure obtained from the same chemical composition.

This research has revealed the paramount importance of microstructure constituent characteristics and the effect of work hardening in two-body abrasive wear behaviour.
Chapter 1

Introduction

1.1 Introduction

The phenomenon of wear is defined as the process of surface damage or material displacement from one or two solid surfaces during sliding, rolling or impact motion relative to each other. This phenomenon is quite common in most industrial applications leading to the deterioration of mechanical strength and service life of machinery. This leads to a rise in the expenditure for maintenance and replacement of machinery components. Among the different types of wear, abrasive wear is severe and accounts for almost 50% of industrial wear [1-5]. During an abrasive wear, material removal occurs when hard particles abrade against a relatively softer surface during their relative motion. This undesirable material removal occurs predominantly during surface mining operations (e.g. mining and mineral processing industries) resulting in a global loss of ~30,140 metric tonnes of steel/year [1, 6, 7]. The statistics is based on six major mining industries across the world. This greatly exposes the financial and productivity downturn caused due to abrasive wear. Therefore, one of the main objectives is to conduct a thorough study on the major components of a sliding two-body abrasive tribological system (i.e. ferrous alloy and abrasive wear environment).

Among, the different sliding abrasive wear experiments (i.e. two and three-body abrasive wear), two-body abrasive wear (e.g. pin-on-disc tribometer) offers more control and better reproducibility of results. The restricted movement of abrasive particles and their constant depth of cut results in equal wear of all phases in a microstructure [8]. However, the two-body sliding abrasive wear is quite a dynamic system involving simultaneous changes both in the material (i.e. microstructure) and
characteristics of the abrasive environment (i.e. deterioration of abrasive particles) [9-15]. The abrasive particle characteristics (i.e. particle type, size, shape and density) play a crucial role in instigating the process of material removal during abrasion [14, 16-18]. For instance, round or polyhedral abrasive particles can cause progressive abrading action, meanwhile, particles with sharp tips can easily cut through the material. Normally, this leads to significant differences in the material removal mode and characteristics of wear debris generation [10, 11, 19]. As mentioned earlier, the abrasive particles are subjected to continuous traversals resulting in significant deterioration during the course of two-body abrasive wear test [11, 19, 20]. This emphasis an underlying fact about the active evolution of abrasive particles and the need to investigate the abrasive particle deterioration mechanisms.

However, this raises a series of arguments over the efficiency of the abrasive particles and the active wear loss induced by them over a defined sliding distance [11, 20]. In an actual industrial digging or excavating operation, the steel is often subjected to a series of constant abrading action by fresh abrasive particles. Accordingly, it becomes imperative to conduct a laboratory abrasive wear test that can simulate and/or replicate the actual industrial condition [21]. To address these aspects, high strain abrasive scratch testing could be a valuable tool, where a robust indenter abrades the microstructure surface under the action of a normal load. One of the major highlights is that the indenter undergoes negligible changes (i.e. constant indenter tip geometry) during the course of the test leading to more control over the abrasive environment.

Concurrently, abrasion imparts appreciable changes (i.e. morphological and mechanical properties) in the microstructure of the material [12, 13, 15]. The two-body sliding abrasive wear induces high strain along with the dissipation of frictional energy as heat towards the microstructure surface [15, 22, 23]. As a result, the region beneath the abraded surface (i.e. sub-surface layer) undergoes severe deformation along with a marked difference in their mechanical properties (i.e. hardness and fracture toughness) from that of the bulk microstructure. However, the amount of frictional energy consumption depends primarily on the characteristics of the microstructure constituents [13, 15, 22].

Extensive studies have reported the beneficial effects of microstructures with high fracture toughness as it can accommodate high strain and undergo work-hardening during abrasion [24-27]. Meanwhile, a positive correlation between the bulk
hardness and the abrasion resistance is more restricted to single-phase microstructures (i.e. pure metals) [28-30]. Nevertheless, bulk properties such as hardness, fracture toughness etc., of a ferrous microstructure are largely influenced by the characteristics of the microstructure constituents [24, 27, 31-33]. In practice, industrial applications often employ complex multi-phase microstructures (e.g. dual-phase steels- ferrite and martensite) [34]. Consequently, the abrasive response of multi-phase microstructures is usually a collective action of the individual metallurgical phases. Therefore, the effect of microstructure constituents in the abrasion phenomenon needs to be considered.

It becomes increasingly obvious that the microstructure characteristics such as size, morphology, volume fraction and carbon equivalent of their metallurgical phases have a significant role on the mechanical properties of a material [35, 36]. Over the years, several investigations have enough stressed on the importance of multi-phase microstructures with a combination of brittle and ductile metallurgical phases (e.g. conventional bainitic steels consisting of ferrite, granular bainite/lower bainite, martensite and retained austenite phases) in high strain abrasive wear conditions. Most importantly, the work-hardening behaviour is a unique feature that results in a hardness increase of the abraded surface making them highly suitable for abrasive applications [37, 38]. As mentioned earlier, the mechanical properties, i.e. hardness, of a microstructure undergoes appreciable changes, thereby the wear performance is more confined to the mechanical properties of the abraded or deformed surfaces [12, 13, 15]. In reviewing these aspects, ultra-high strength bainitic steels (also known as nanobainitic steel) consisting of very fine bainitic ferrite and retained austenite has been found to be a potential candidate for high strain abrasive wear applications. These steels display a wide range of work-hardening behaviour depending on the retained austenite characteristics [39-42]. Therefore, advanced TRIP steels with nanobainitic structure have been employed to understand the impact of retained austenite characteristics and their effect of work-hardening in two-body abrasive wear behaviour.

The aim of this thesis was to conduct systematic separate investigations on the different components, i.e. microstructures and abrasive environment of two-body abrasive tribological system. This led to a thorough study on the effect of abrasive particle characteristics (e.g. particle size and density) in two-body abrasive wear,
where two different abrasive environments (e.g. silicon carbide and alumina) were subjected to a series of two-body abrasive wear tests. An attempt was made to understand the efficiency of abrasive particles through interrupted abrasive wear tests, as a function of sliding distance and abrasive particle characteristics. Meanwhile, the influence of microstructures in two-body abrasive wear was analysed by comparing four distinct microstructures (i.e. bainite, pearlite, martensite and tempered martensite) with similar hardness levels. This also included extensive studies on the sub-surface and topographic analysis of the abraded regions. Furthermore, advanced high strength bainitic steels transformed at different isothermal holding temperatures (200°C-350°C) were subjected to two-body abrasive wear. The main purpose of this study is to determine the significance of retained austenite characteristics and their effect on work hardening on the abrasive wear behaviour. Finally, the process of material removal in abrasion was investigated using a controlled high strain abrasive scratch testing. In this study, the process of material removal as a function of normal load and the characteristics of microstructure constituents were investigated.

1.2 Thesis outline

The following provides a brief outline and major highlights of each chapter.

Chapter 2 reports a detailed literature review on the fundamentals of abrasive wear and the steels that are commonly used to tackle abrasion in industrial applications. An in-depth investigation was carried out on the effect of abrasive particle characteristics on abrasive wear. The parameters that affect the abrasion resistance of a ferrous alloy was discussed in detail. The significance of microstructure constituents in abrasion and their ability to undergo work-hardening was highlighted. Among the different steels that were investigated, advanced TRIP steels were found to be a probable candidate, as it revealed superior work-hardening behaviour. Finally, the chapter identifies the gaps in the current knowledge and details the objectives for further work.

Chapter 3 introduces the experimental materials and methods used in this research. Heat treatments furnaces and rolling mill used for thermomechanical processing were described. Pin-on-disc tribometer and scratch test instruments that were exclusively employed for performing two-body abrasive wear experiments are discussed in detail. A number of characterization techniques such as metallography, XRD, electron microscopy and optical profilometry used in this study are explained.
Chapter 4 investigates the role of abrasive particle characteristics in the process of material removal in a two-body abrasive wear. This study also analyses the efficiency of the abrasive particles as a function of sliding distance and particle characteristics. A detailed analyses on the particle deterioration mechanisms are presented in this chapter.

Chapter 5 reports the two-body abrasive wear resistance of four distinct microstructures (i.e. bainite, pearlite, martensite and tempered martensite) with similar hardness levels. The unique response of the microstructures was evaluated in terms of specific wear rate, friction coefficient and sub-surface layer characteristics. The process of material removal was investigated through single-track characterization technique. The superior abrasive performance of bainite and pearlite was attributed to their microstructure matrix and ability to undergo work-hardening phenomenon. A schematic representation of the microstructures under the action of an abrasive particle was developed.

Chapter 6 determines the influence of retained austenite characteristics on the two-body abrasive wear resistance of ultra-high strength bainitic steels (i.e. nanobainitic steels). The abrasive wear behaviour of the fully bainitic microstructures consisting of bainitic ferrite and retained austenite with different characteristics (e.g. size, volume fraction, carbon content and morphology) is discussed. A detailed analysis on the impact of retained austenite morphology in triggering a range of TRIP effect is presented in this chapter. The significance of optimum work hardening in abrasion is also highlighted.

Chapter 7 focusses on the process of material removal in microstructures under the action of a normal load in a controlled high strain abrasive scratch testing. In this technique, a robust indenter is used to simulate the abrading action, thereby isolating the effect of abrasive environment in the material removal process. A high performance TEM–NanoMEGAS ASTAR characterization provided valuable information on the sub-surface deformed layers in the microstructure. In addition, a relationship between the material removal mechanism in the microstructure and the normal load subjected during the scratch testing is described in this study.

Chapter 8 comprises the conclusions drawn from this research and suggestions for future work.
Chapter 2

Literature review

2.1 Introduction

The phenomenon of friction and wear is inevitable in most engineering applications. During service conditions, wear phenomenon involves a significant amount of material loss. It has been estimated that 1% of gross national product of a nation can be saved by means of better friction reduction and wear control [1, 43]. Therefore, this chapter will discuss the wear mechanisms and their major types in detail. The parameters affecting the wear mechanisms will be thoroughly examined for a better understanding of this process.

This chapter will briefly discuss the severity and impact of abrasive wear in mining and mineral processing industries. Also, providing a detailed account on the properties of the abrasive environment and the different steels that are currently used in industries. In summary, it provides a thorough analysis on the interaction between the abrasive environment and the materials that are being subjected to abrasion.

2.2 Tribology

The word ‘tribology’ is derived from the Greek word tribos meaning rubbing. It refers to the ‘science of rubbing’. In general, it is defined as the science and technology of interacting surfaces in relative motion and related practices [44, 45]. A tribological system consists of surfaces of two components that are in moving contact with one another and their surroundings (Fig. 2.1). An in-depth knowledge on multi-disciplinary entities such as contact mechanics, material science, thermodynamics and material design is highly essential for a better understanding on the surface interactions. A tribological system is usually governed by two major phenomena, namely friction and
wear. The study of friction and wear measurements is known as *tribometry* and the equipment that aids in these measurements are called as *tribometers* [1, 46].

![Figure 2.1: Schematic representation of a tribological system [2].](image)

**What is the need for understanding wear and friction mechanisms?**

Friction and wear are two different phenomena that operate when two surfaces undergo sliding or rolling motion. Friction is accompanied by energy dissipation, whereas material removal occurs during wear. In engineering applications, friction needs to be controlled and wear must be reduced for a better service life [43, 47]. The following section will highlight the basic mechanisms of friction and wear, which are essential for selection of materials, coatings and surface treatment for an application.

**2.2.1 Friction**

Friction is defined as the resistance to relative motion (sliding or rolling, Fig. 2.2), when one body moves tangentially over another. It is expressed as a coefficient of friction, $\mu$, which is the ratio of tangential force, $F_T$, required to initiate or sustain relative motion to the normal force, $F_N$, that presses the surfaces together [43].

$$\mu = \frac{F_T}{F_N} \tag{2.1}$$

where $F_T$ is the tangential force, $F_N$ is the normal force.
Figure 2.2: Schematic representation of a) rolling and b) sliding friction [43].

Friction is divided into two types, namely sliding friction and rolling friction (Fig. 2). Frictional force arises mainly due to the mechanical interaction between the asperities on a microscopic scale. These asperities deform either elastically or plastically leading to an energy dissipation in the form of heat. The scenario of mechanical interaction is largely dependent on the surface profile of the mating surfaces [43, 46, 47].

a) Surface topography

The surface topography of a given surface consist of irregularities in the form of peaks and valleys (Fig. 2.3) when examined under an electron microscope. The surface topography assessment is performed using a profilometer, which measures the vertical and horizontal displacement across the given surface profile [48].

Figure 2.3: Schematic representation of surface irregularities [46].

The surface finish or roughness of any surface is quantified by the characteristics of peak and valleys [48]. The parameters that are primarily used in the measurement are explained below.
i) $R_a – \text{Arithmetic average roughness}$: It is the arithmetic average height of the irregularities from the mean line, over a defined distance (Fig. 2.4). They offer simplicity and they are widely used in quality control over surface finish operations [48].

ii) $R_q – \text{Geometric average roughness}$: It was earlier known as root mean square (or RMS), which provides a geometric average height of the irregularities (i.e. $Y_1$, $Y_2$, $Y_3$... and $Y_8$) over a defined distance, $L$. This parameter is more sensitive to irregularities, as they amplify the regions that are generally not in the range (Fig. 2.4). For example, the $R_q$ for a given surface is approximately 11% higher than its $R_a$ value [48].

![Schematic representation of $R_a$ and $R_q$](image)

iii) Peak and valley heights: In general, the irregularities (peaks and valleys) on the surface can be measured using the parameters $R_T$ and $R_Z$ (Fig. 2.5) [48]. Peaks are defined as the section of the surface profile in the positive direction from the mean line, whereas the section below the mean line (negative direction) are defined as valleys [48].
R_Z - The arithmetic peak to valley height of the roughness profile (i.e. Z_1, Z_2 and Z_3) over an evaluation length.

\[ R_Z = \frac{(Z_1 + Z_2 + Z_3)}{n} \] ........................................... 2.2

R_T or R_{max} – It is defined as the maximum peak to valley height within the roughness profile over the given evaluation length (Fig. 2.5).

### 2.2.2 Wear

Wear is a process of surface damage or removal of material from either one or two solid surfaces in solid-state contact. It occurs when the solid surfaces are in sliding or rolling motion relative to each other [50]. It is a system response and not a material property. In general, wear is defined based on the volume of material loss. When the material displacement is on a microscopic scale, the net change in volume or mass of the material is null. However, the phenomenon of wear occurs slow and steady as a continuous process. Wear can be classified based on their mechanism of material removal [1, 7, 46].

- Adhesive wear
- Fatigue wear
- Tribochemical or corrosive wear
- Fretting wear
- Abrasive wear

With the exception of fatigue wear, all of the above wear mechanisms have a common characteristic feature of a gradual material removal. In most cases, the wear does not
follow a single distinct mechanism but rather a combination of one or more mechanisms [1, 46].

a) Adhesive wear

Adhesive wear (also known as galling or scuffing) is mainly due to the adhesive forces (bonding) acting at an atomic scale [51]. When two nominally flat bodies are in sliding contact, adhesion occurs at the asperity contacts due to high local pressures (Fig. 2.6). These contacts are sheared during sliding, resulting in fragmentation from one surface and attachment to the other surface. During continuous sliding, the fragments come off or result in generation of loose wear particles. Since adhesive wear includes adhesion and fracture of the mating surfaces, the surface topography and environment play a crucial role.

![Figure 2.6: Schematic representation of adhesive wear [51].](image)

Archard’s equation explains that the hardness of a material has an impact on the adhesive wear behaviour. The wear coefficient $K$ is dependent on the material properties and their surface topography [46]. Archard’s equation is expressed as volume of material removed per unit sliding distance $W_{ad}$:

$$W_{ad} = \frac{V}{L} = \frac{KF}{H} \quad \text{…………………………………. 2.3}$$

where, $K$ = Wear coefficient,

$V$ = Volume of worn material removed (mm$^3$);

$L$ = Sliding distance (m);
F = Normal load (N);
H = Hardness of the softer material (HV) [46].

b) Fatigue wear

Fatigue wear or failure is associated with the repeated stress cycles on a material during sliding or rolling applications. The effect of stress is mainly on the surface or subsurface without any physical contact of surfaces. Lang [52] observed this phenomenon, when surface fatigue failure occurred in journal bearings, despite the interacting surfaces being fully separated by a lubricant film. This is mainly because the maximum shear stress lies at some distance below the surface, with zero tangential stress at the surface. Thereby, it leads to the occurrence of subsurface cracks during service (Fig. 2.7).

![Figure 2.7: Schematic of fatigue failure – subsurface cracks [52].](image)

c) Tribochemical or corrosive wear

Tribochemical wear involves the dynamic interaction between the mating surfaces and the environment. It occurs in industries (e.g. mineral processing, mining etc.) where corrosive environments are prevalent. Friction tends to modify the kinetics of the chemical reactions of the sliding bodies with the environment. Therefore, high temperature reactions occur at ambient temperature. This process of altering the chemical reaction by friction or any mechanical force is termed as *tribochemistry* and the wear associated with such a phenomenon is called as *tribochemical wear*. A significant amount of material loss is involved in this wear, due to the combined effect of friction and chemical reactions with the environment [44, 53].

d) Fretting wear
Fretting occurs when low amplitude oscillatory movements take place in a tangential direction between the contacting surfaces, which are at rest. It is a form of adhesive wear, where there is adhesion between the asperities due to the normal load and cracks occur during external vibration [54]. When fretting is combined with corrosion, the wear mechanism is defined as fretting corrosion. Fretting wear is determined by variables such as slip, normal load, frequency of vibration and type of contact between the bodies. Such wear is predominant in splines, shrink fits and bolted parts [54].

e) Abrasive wear

Abrasive wear (also known as scratching, scoring or gouging) is often described as the damage to a surface by a harder material. In abrasive wear process, the asperities of a rough and harder surface press or slide into a relatively softer surface and cause damage [55, 56]. In the case of ductile materials, there will be plastic flow of the softer material, but in the case of brittle materials wear occurs by brittle fracture [3].

In industrial applications, the impact of wear is quite significant. The type of wear that are normally encountered are: abrasive wear (50%), adhesive wear (15%), erosion (8%), fretting (8%) and chemical wear (5%) [2]. The distribution of the specific wear rate of different metallic materials exhibit the severity of different wear regimes [5] (Fig. 2.8). Abrasive wear occurs mainly in mining, mineral beneficiation, agriculture and earth moving machineries [57]. Tribological losses (both friction and wear) incurred in mining and mineral processing sectors account for almost half of the nation’s wear. It has been estimated that surface mining operations (e.g. exposing and digging) results in a loss of ~30,140 metric tons of steel every year [6]. Considering the financial impact and the amount of material loss involved in abrasion, further discussion will be focused on abrasive wear. Therefore, the development of materials that are more resistant towards abrasive wear is economically important. It is important to study the mechanism of abrasive wear in order to simulate the service wear conditions in the laboratory [21].
2.3 Classification of abrasive wear processes

The abrasive process is classified based on three important parameters:

- Number of bodies involved in contact: Two-body and three-body body abrasion wear
- Ability of the abrasive particles to move within the mating surfaces: Open and closed.
- Stress levels between the abrasive particles and the solid surfaces: Gouging, high stress and low stress [4].

2.3.1 Two-body and three-body abrasion

In two-body abrasive wear, a hard material slides against a softer surface to remove material. The harder material could be the abrasive particles when slid across the surface results in a groove.

In the three-body abrasive wear, the two mating surfaces and the abrasive particles are involved in the abrasion process. The particles are loose and they can move or rotate while sliding across the mating surfaces (Fig. 2.9). The three-body abrasive wear is more common and complex than the two-body abrasive wear. Since the abrasive particles have different freedom of movements (such as slide and roll), a wide range of wear rates have been found [58].
2.3.2 Open and closed abrasive wear

The three-body abrasive wear is further classified into open and closed, based on the ability of the abrasive particles to move within the mating surfaces [21]. In closed three-body abrasive wear, fine abrasives are trapped between the mating surfaces, which are close to one another. Open three-body abrasive wear occurs when there is a thick bed of abrasive between the surfaces. Here, the surfaces are either far apart or only one of the surfaces will be influential in the wear process [21].

2.3.3 Gouging, high and low stress abrasive wear

Avery [59] proposed a further classification of the abrasive wear into three groups: gouging, high stress and low stress. Gouging abrasion involves coarse abrasive particles (such as rocks) and higher stress resulting in larger material removal (Fig. 2.10a). In some cases, the abrasive particles are compressed between the surfaces under high stress (known as grinding), like in the case of ball mill grinding. The high stress levels cause dents, scratches and crushes the abrasive particles (Fig. 2.10b). On the other hand, when there is no damage or fracture to the abrasive particles, it is termed as a low stress abrasive wear (known as scratching, Fig. 2.10c). Due to the low stress levels, the cutting and ploughing of the material is on a microscopic scale. However, there is very little difference between low and high stress abrasive wear [21].
2.4 Mechanism of abrasive wear

There are several mechanisms to explain the material removal during an abrasive wear. Due to the complex nature of abrasion, it is difficult to account one major mechanism for the material loss. Plastic deformation, fracture, fatigue and grain pull out are some of the mechanisms, which are widely seen during abrasive wear [7, 60, 61].

2.4.1 Plastic Deformation

This mechanism is mostly associated with ductile materials. When a ductile material slides against the abrasive particle, it results in either one or more combination of three abrasive wear modes, i.e. ploughing, wedge formation and cutting (Fig. 2.11). Ploughing involves the formation of wear tracks (grooves) by displacement of material to their sides (Fig. 2.11a). In the case of wedge formation, the material is pushed along ahead of the particle (Fig. 2.11b). In both these modes, there is little wear loss or negligible material removal. However, in the case of cutting, the material is displaced as wear debris or micro-chips with very little material displacement to the sides (Fig. 2.11c) [7, 62].
A two dimensional abrasive model by plastic deformation has been proposed by Rabinowicz. It is based on an abrasive particle (assumed to be sharp cone) with a semi angle, $\theta$, known as attack angle (i.e. the angle between the abrasive particle and the material surface) under a load, $F$, when dragged across a ductile surface of hardness, $H$ (Fig. 2.12). It results in the formation of a groove with material displaced from it. The volume of the groove, $V$, per unit length is given by,

$$\frac{V}{L} = \frac{2F}{\pi \tan \theta H} \quad \text{............... 2.4}$$

The wear rate, $Q$, is defined by Rabinowicz equation,

$$Q = K \frac{F}{H} \quad \text{............... 2.5}$$

Where, $K$ is the wear coefficient and is given by,

$$K = \frac{2\eta}{\pi \tan \theta} \quad \text{............... 2.6}$$

Where, $\eta$ is the fraction of the material removed from the groove. From the above equations, it is clear that the wear rate is directly proportional to the load and inversely proportional to the hardness of the material [61, 63].
2.4.2 Fracture

Even though plastic deformation governs the rate of material removal in ductile materials, the effect of fracture cannot be neglected [64]. Nevertheless, the material removal process is vastly controlled by micro-cracking or fracture mechanism, in the case of brittle materials. Evans and Wilshaw [65] developed a model for the abrasive wear by fracture.

When a brittle material is subjected to abrasion, very high stresses occur at the point of contact, $D$ (Fig. 2.13). As the load increases above a critical level, a median vent crack, $M$ is initiated at the vertical mid-plane. The crack extends further down with an increase in the load. As the sliding contact continues, there is a reduction of load at $D$ followed by the formation of lateral vent cracks, $L$. These lateral cracks continue to grow upwards and terminate at the surface, resulting in the detachment or fragmentation of material.

Figure 2.13: Schematic representation of crack formation in a brittle material:
Normal load increases from (a-c) and then decreases from (d-f) [65].
Based on this theory, a model was developed focusing on the lateral cracks that grow upwards and the residual stress associated with the material. Wear rate per unit sliding distance, $Q$, is given by,

\[ Q = \alpha \frac{F^{5/4}}{A^{1/4}} \frac{\sqrt{d}}{\sqrt{K_c}} \]

where $\alpha$, $F$, $d$, $A$, $K_c$ and $H$ are the material independent constant, load, diameter of the abrasive particle, contact area, fracture toughness and hardness of the material, respectively [65].

### 2.4.3 Fatigue

A repeated strain caused by the abrasive particles on the surface can lead to metal fatigue. A transverse section of an abrasive groove with sideways material displacement is shown in Figure 2.14a. Sideways displacement of material due to repeated deformation and subsequent fracture is evident. This mechanism type is relatively mild because repeated deformation is required to produce the wear particle [44].

![Figure 2.14: a) Schematic representation of the sideways material displacement due to fatigue and b) grain pull-out mechanism [44].](image)

### 2.4.4 Grain Pull-Out

This mechanism is mainly confined to ceramics and it is relatively rare. This occurs when the inter-grain bonding is weak and the size of the grain is large [4]. Poor deformation ability of the material and highly concentrated stresses result in detaching large wear fragments from the wearing surface (Fig. 2.14b). The volume of lost material is generally higher than the volume of wear track [44].
2.5 Variables affecting abrasive wear

The variables affecting abrasive wear can be classified into three groups, 

- Environment;
- Abrasive particle characteristics and
- Properties of the material [66].

2.5.1. Environment

During the abrasive wear, the mating surfaces are subjected to compressional stress and the temperature rises. Most of the energy that is spent during the plastic deformation of the material is dissipated as heat. The rise in temperature affects the mechanical properties and the phase changes of the mating surface or the material. This effect is more significant in the case of metals, as an increase in the temperature leads to a decrease in the hardness of the worn surface and the abrasive, eventually leading to higher wear rate [67, 68].

The moisture in the atmosphere can have contrasting effects on the abrasive wear rate. The abrasive particles can be weakened by the prevailing moisture in the atmosphere, leading to fine abrasive particles. Meanwhile, the surface is also affected by the moisture [44].

2.5.2. Abrasive particle characteristics

The characteristics of the abrasive particles play a significant role in affecting the amount of material loss during abrasion. The variables related to the abrasive particles (e.g. morphology and hardness) play a major role in determining the efficiency of the abrasive particles [17],

a) Morphology

The morphology of the abrasive particle (shape and size) greatly influences the wear rate. However, the distribution of abrasive particle shape is not uniform in a given abrasive environment. Also, the abrasive particles of similar sizes are most likely to have different shapes and orientations [17]. Therefore, it becomes increasingly hard to study the individual effect of abrasive particle shape and size on abrasion. Thereby, a combined discussion on the impact of abrasive particle shape and size has been dealt here.
Researchers have shown that the particle shape determines the cross sectional area of the groove (wear track) that is formed during abrasive wear. After a detailed analysis on the various abrasive particles, they are characterized based on parameters such as aspect ratio (width/length) and roundness factor, \( F \).

\[
F = \frac{4\pi A}{P^2} \quad 2.8
\]

where \( P \) and \( A \) are the perimeter and area of the projection [7]. In general, the wear rate is usually higher for sharp pointed abrasives (pyramidal or conical) than for blunt and round abrasives (spherical type) [69]. For pyramidal or conical asperities, the angularity (attack angle, \( \theta \)) determines their ability to actively cut through the material. There is a critical attack angle, \( \theta_c \), where there is a transition in the abrasive wear mode. Particles with higher attack angles (\( \theta > \theta_c \)) favour cutting mode, whereas lower \( \theta_c \) result in other abrasive wear modes (Fig. 2.15). However, it is often quite difficult to characterize the angularity due to the complex shape of the abrasive particles. In the case of spherical asperities, the material removal is measured through their degree of penetration (i.e. ratio of depth to width) [20, 70].

![Figure 2.15: A transition in the abrasive wear mode as a function of attack angle](image)

There has been a strong influence of particle shape on the abrasive particle size effect. Coarse particles (>60 \( \mu m \)) were characterised with multiple round or polyhedral edges, whereas sharp tips were observed in fine particles (<15 \( \mu m \)). Therefore, coarse particles with small attack angles can penetrate deep. When these coarse particles are broken down, it leads to more sharp tips, resulting in higher material loss. On the other
hand, fine particles with sharp tips can initially cut through, although they are more vulnerable to fracture.

In general, the wear rate increases with an increase in the size of the abrasive particles up to a certain point (~100 μm) known as the critical particle size (CPS). Above which, three distinct phenomena may occur (Fig. 2.16):

- the wear rate can increase slightly (curve 1 in Fig. 2.16)
- the wear rate becomes independent of the particle size (curve 2 in Fig. 2.16)
- the wear rate tends to decrease (curve 3 in Fig. 2.16) [71].

![Figure 2.16: Effect of particle size on wear. CPS represents critical particle size](image)

As mentioned earlier, not all abrasive particles are involved in the process of two-body abrasion due to the presence of non-uniform distribution of particle shapes in the environment. Nevertheless, the number of particle contacts with the material is closely associated with the orientation of the particle [17]. The packing nature (i.e. number of particles over a defined area) of the particles in an abrasive environment also proves to be a crucial factor in estimating the material loss.

**b) Hardness**

Abrasive wear is also determined by the particle hardness. It depends mainly on the ratio of the hardness of surface, $H_S$, to the hardness of the abrasive, $H_a$. The rate of material removal depends on the critical ratio of $H_S/ H_a$, which is found to be 1.2 irrespective of the particle shape [72, 73]. When $H_S/ H_a < 1.2$, there is less scratching or wear and is termed as soft abrasion. If the ratio $H_S/ H_a$ exceeds 1.2, it is called as hard abrasion (Fig. 2.17) [74].
This is largely based on the contact mechanics between the abrasive particle and the surface. The plastic flow of material from the surface occurs, when the contact pressure reaches \( 3Y \), where, \( Y \), represents the uniaxial yield stress of the material. This causes indentation on the surface, provided the particle can withstand the pressure without any deformation, especially in the case of \( H_s/H_a > 1.2 \) (Fig. 2.17a). However, in the case of \( H_s/H_a < 1.2 \), there is a particle fracture, leading to negligible plastic flow on the material surface (Fig. 2.17b) [75].

![Figure 2.17: Schematic representation of particle contact on a plane surface under a normal load](image)

In general, the characteristics of the abrasive particles (e.g. morphology, orientation, distribution etc.) play a pivotal role in affecting the process of material removal. However, the abrading efficiency of the particles is often debatable, due to their constant deterioration during the abrasive wear. Therefore, such a complex and dynamic abrasive environment needs a thorough analysis to study the abrasive particle deterioration mechanisms and their process of material removal during abrasion.

2.5.3. Properties of the material

Metallic alloys form a major part of the industrial components that are being subjected to wear. As discussed earlier, the stress levels associated with abrasive wear determine the severity of the material loss. Therefore, factors such as mechanical properties (hardness, fracture toughness and work hardening) and metallurgical properties (microstructure and alloying elements) of the metals influence their abrasive resistance [76]. The following sections will brief about these factors.
a) Mechanical properties-hardness and fracture toughness

Hardness is defined as the resistance to indentation and is considered to be one of the principal variables that influence abrasive wear. Hardness is, in general, related to the flow stress of the material. The indentation hardness is approximately three times the uni-axial flow stress at the value of the strain produced by the indentation.

The effect of hardness is quite dominant when comparing the abrasion resistance of different material types (such as pure metals and ceramics, Fig. 2.18 [76, 77]). The abrasion resistance of the material increases progressively with an increase in the bulk hardness of the material. This argument holds true especially in the case of pure metals, where the effect of structure and elemental compositions are less significant [30]. Moreover, the abrasive wear resistance depends on the hardness coefficient, $K_T$ ($K_T = H_S / H_a$, where $H_S$=hardness of surface and $H_a$=hardness of abrasive). When $H_S / H_a \geq 0.5/0.6$, there has been significant increase in the wear resistance [78].

![Figure 2.18: Effect of abrasive hardness on wear behaviour of metals and ceramics](image)

However, materials with similar bulk hardness (e.g. grey cast iron, white cast iron and ceramics, Fig. 2.19) tend to display a range of distinct abrasion behaviour. Interestingly, materials with higher bulk hardness displayed a poor abrasion resistance.

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This is mainly due the ratio $E/H$, where, $E$=elastic modulus and $H$=hardness of the material. Low values of $E/H$ lead to cutting abrasive mode, as discussed in 2.4.1. This argument explains the higher material loss in ceramics in comparison with metals, despite having a similar hardness level (Fig. 2.19) [8].

Moreover, in ferritic alloys, the surfaces are subjected to a high degree of deformation, resulting in a work-hardening phenomenon (i.e. hardness increment on the wearing surface). This work-hardening behaviour depends largely on the type of alloy and the intensity of strain levels during abrasive wear. Researchers have shown that there is a linear relationship between the work-hardening behaviour and the abrasion resistance of the alloy. Also, prior work-hardened surfaces have very little or null effect on increasing the abrasive wear resistance [76, 79]. Eventually, this shows that the hardness of the worn surface is of prime importance than the hardness of the undeformed surface.

Fracture toughness is a key parameter in resisting the crack propagation in materials. Especially in the case of brittle materials, above the critical loading, as discussed in 2.4.2, micro-cracking occurs. It has been shown that increasing the ratio of hardness to fracture toughness favour increased material loss by micro-cracking [26]. In addition, a strong relation between fracture toughness, hardness and wear resistance has been observed (Fig. 2.20). Materials with higher fracture toughness
undergo abrasive wear by plastic deformation (ploughing and wedge formation), whereas micro-cutting or fracture occurs in low fracture toughness materials (i.e. brittle materials). This clearly indicates that mechanical properties such as hardness and fracture toughness play a major role in determining the abrasion resistance of a material. Nevertheless, the mechanical properties of a material are indeed influenced by their metallurgical structure (microstructure) [27].

![Figure 2.20: Relation between fracture toughness and hardness of a material [27].](image)

**b) Metallurgical factors**

Microstructure of materials have a significant impact on their abrasive wear behaviour. The chemical composition and production techniques influence the microstructure of a given material (Fig. 2.21). In the case of ferritic materials, the effect of alloying elements play a major role in determining their abrasion resistance. Compared with other alloying elements, carbon has a greater impact because of its strengthening effect [80-82]. Carbide forming elements also contribute towards a slight increase in abrasive resistance. Addition of nickel can greatly improve the toughness of the steel. Moreover, the presence of alloying elements can delay transformation reactions that can result in undesirable phases in the microstructure [83].
The microstructure can be produced by mechanical, thermal (heat) and thermomechanical treatments. Cold working process such as rolling, deep drawing etc. can induce structural changes in the microstructure. However, there is little or negligible effect of cold working on abrasion resistance of a material, as the abrasive wear process induces much higher surface strains [7]. Thermal (heat treatments) processing techniques such as annealing, tempering etc. are used in steels and ageing of aluminium alloys. In general, a combination of thermal and mechanical (i.e. thermomechanical) treatments are widely employed to produce a desired microstructure with improved properties (e.g. high rolling temperature in high strength low alloy steels). Moore [77] explained the effect of microstructure, thermal treatment and the chemical composition on the abrasive wear resistance of steels against an alumina abrasive environment (Fig. 2.22). In general, the abrasion resistance of a steel can be varied by producing different phases. Nevertheless, the properties of these microstructural phases are primarily dealt by their constituents [17, 77, 84]. A detailed discussion on the effect of metallurgical phases on abrasive wear will be done later on.
Figure 2.22: The effect of microstructure and hardness on the abrasive wear resistance of steels against an alumina abrasive environment [77].

The abrasion response of the microstructure is also determined by the severity of the abrasive environment (high and low stress abrasive wear). Homogenous microstructures (e.g. pure metals) offer very little resistance to the high stress levels associated during abrasion. Multi-phase microstructures (e.g. white cast irons, composites, hard transition metal carbides etc.) consisting of hard and ductile phases are quite effective in such severe abrasive conditions. This is often determined by the size, spacing and volume fraction of the harder phase [25]. When the width or indentation of the abrasive particle is greater than the hard phase (finely dispersed hard phase), it often leads to increased flow behaviour (i.e. formation of ploughing and wedge formation modes, as discussed in 2.4.1), ultimately leading to better abrasion resistance [7, 62]. However, very high volume fraction of hard phase or a brittle matrix can lead to decreased abrasion resistance. The abrasive particle action on such large hard phases can cause fracture wear mode as the cracks can initiate at the vulnerable particle-matrix interface [85, 86].

Despite hardness and toughness being key factors that influence abrasive wear, the abrasive wear resistance of a material is largely governed by its microstructural characteristics. Literature reports a linear relationship between hardness and the abrasive wear resistance of homogenous/single-phase microstructures (e.g. pure metals). However, in the case of complex/multi-phase microstructures, a cumulative response of the individual phases determines their abrasive wear resistance.
Therefore, a comparative study on the abrasive wear behaviour of single and multi-phase microstructures can aid us in deriving a relation between hardness, microstructure characteristics and the abrasive wear resistance of a material.

2.6 Laboratory abrasive wear tests

To quantify or simulate the abrasive conditions, aforementioned factors must be considered. The following section will briefly outline the different laboratory tests that are currently used to understand abrasive wear. Since the wear performance is mostly system related, i.e. depending on the characteristics of the abrasives and the sliding conditions, a proper selection of test method and equipment is vital. Based on the abrasive particles’ freedom of movement, the laboratory test has been classified into two major groups to quantify abrasive wear.

- Abrasives are fixed relative to the wearing specimen (two-body abrasive wear)
- Abrasives are free to move with respect to the wearing specimen (three-body abrasive wear) [87].

Commonly used laboratory test for abrasive wear include a pin on disc/drum two-body abrasive test (Fig. 2.23a), where a pin slides across the fixed abrasive to simulate the two-body abrasion. In the case of three-body abrasive wear, a wheel (rubber or steel) rotates against another with loose abrasive particles being continuously fed from a hopper (Fig. 2.23b) [88].

![Figure 2.23: Schematic illustration of the different laboratory set-ups to demonstrate two- and three-body abrasive wear [88].](image)

For appropriate selection and effectively characterize the abrasive test conditions, important parameters were summarized in Table 2.1. Moreover, factors such as the precision of the equipment and reproducibility are of paramount importance. In two-body abrasive wear, the freedom of the abrasive particles is restricted and the depth of cut remains constant irrespective of the type of the micro
constituents of the material. Thereby, equal wear occurs for all phases present in the material [8]. Rabinowicz [63] demonstrated that the amount of wear is more in the case of two-body abrasive wear mode than in a three-body abrasive type. This is due to the fact that in a three-body abrasive wear, the particles abrade the surface around 10% of the total time. Considering the above, two-body abrasive wear test (fixed abrasive tests) offers more control and ease than a three-body abrasive wear (loose/free abrasive tests). However, it is important to understand that the abrasive environment undergoes significant changes (in size, shape and attack angle of the abrasive particles) in the above mentioned tests. During the course of a two and three-body tests, it is more likely that the abrasive particles tend to lose their abrading efficiency and are no longer involved in the process of abrasion. As discussed earlier, in an actual abrasive environment the material is subjected to a constant abrading action [4]. Therefore, it is quite essential to choose an abrasive wear test that can actively replicate such continuous abrading action and evaluate the abrasive wear resistance of the material.

Table 2.1: Salient features of different abrasive tests.

<table>
<thead>
<tr>
<th>Type of abrasive tests</th>
<th>Test characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Two-body abrasive test</td>
<td>1. Type &amp; size of abrasive particles-limited by availability of grit papers.</td>
</tr>
<tr>
<td></td>
<td>2. Pressure acting on the abrasive or the mechanics of abrasive – wearing surface is almost constant [4].</td>
</tr>
<tr>
<td>Three-body abrasive test</td>
<td>1. More freedom to select the size and shape of abrasive particles (even from actual service conditions).</td>
</tr>
<tr>
<td></td>
<td>2. Difficult to characterize the load acting per unit area, due to constant variation [4] [63].</td>
</tr>
</tbody>
</table>

Scratch test is one such test that can simulate high-stress abrasive wear mechanism offering good control and repeatability. In a standard scratch test, an indenter acting as an abrasive particle abrades/slides against the material of interest (Fig. 2.24). The robust design allows negligible or no changes in the indenter during the course of an abrasive test. Moreover, a constant load and sliding velocity greatly regulates the abrasive wear test conditions. This primarily imparts more control over the abrasive environment and focus on the material removal process. Except for the limitations in the type of abrasive environment (i.e. indenter material), this test
provides more scope in understanding the abrasive wear resistance of microstructures under controlled test parameters.

![Figure 2.24: Schematic illustration of a scratch test.](image)

**2.7 Energy balance principle in two-body abrasive wear**

A tribological system is quite dynamic as it involves a significant amount of changes in the system during the combined action of friction and wear. These occurrences are mainly based on the energy balance principle of a system. For a given two-body sliding abrasion system,

The input energy equals to useful work (i.e. abrading action) + energy loss to other systems and surroundings [15, 22, 23].

Based on this equation, a major section of the input energy is used for the abrading action and rest is lost to other systems. Since abrasion involves friction, the input energy is provided for overcoming the frictional force. In general, mechanical work is defined as the product of force times the distance. In the case of sliding, the frictional force, $F$, times the sliding distance, $L$, gives the frictional work, $FL$. This frictional work is consumed in the process of friction and wear. The frictional energy is dissipated to different sections of a system. The energy consumption during sliding, $\varepsilon$, for the given volume of material removed, $W$, under the action of a normal load, $P$, is given by the equation: $\varepsilon = (FL)/W = (\mu PL)/W$ [15, 22, 23].

Therefore, the amount and mechanism of energy dissipation are critical as it affects the system properties, such as microstructural changes in the material, mechanical properties etc. [89]. In this context, the energy consumption is largely unique for different microstructures due to their microstructural constituents [15, 22,
23]. Literature suggests that microstructures with high fracture toughness and work-hardening component are likely to consume more energy during sliding action [15, 23]. Accordingly, the microstructural constituents that can accommodate the high strains during abrasion can yield better resistance towards abrasion.

*From the above discussion, it is clear that the abrasive wear behaviour of a system is based on a cohesive action of the abrasive environment and the abraded material. Hence, this tribological system is quite dynamic that leads to significant changes occurring simultaneously in both the entities (i.e. material and the abrasive environment). Therefore, separate investigation needs to be carried out to fully understand the individual effect on abrasive wear behaviour.*

2.8 Abrasive wear in steels

Abrasive behaviour of steels is largely based on the characteristics of their microstructure [28, 29, 90]. A wide range of microstructures can be produced in steels depending on composition and heat treatment procedure. In general, microstructures having superior work-hardening component is most likely to exhibit better abrasion wear resistance [27, 76, 79]. In two-body sliding abrasive wear, the steel abrades against an abrasive surface. During which, the friction curve consists of an initial running-in period (i.e. A to B in Fig. 2.25), followed by a steady-state period (i.e. B to C in Fig. 2.25). There is a steady rise in coefficient of friction during the running in period, as it abrades the particles. A significant amount of deformation is involved during the initial running-in period, after which the coefficient of friction reaches a near constant level known as the ‘steady-state period’. During this period, the indented abrasive particle encounters a highly deformed surface. Such deformation or the work-hardening behaviour of the surface is largely governed by the surface hardness rather than the bulk hardness of the material. Also, the wear response is directly related to the properties of the layer beneath the wear surface rather than the properties of the bulk material [13]. Hence, abrasive wear behaviour can be considered to be dependent on the deformation behaviour, which is a strong function of hardness, ductility and fracture characteristics [12]. Eventually, the microstructural features are vital in abrasion resistance as they influence the flow stress at higher strains [25].
On a general note, the characteristics of the microstructural constituents have a great impact on the abrasion resistance of the steel alloy. Different microstructures tend to have a unique response to abrasion due to their distinct microstructural characteristics. The following sections will discuss the commonly used steels in industries, outlining the different heat treatment procedures and their corresponding microstructures response towards abrasive wear.

2.8.1 Commonly used steels in abrasive environments

Carbon steels are commonly used in industries, as they offer a wide range of mechanical properties at an affordable cost. The carbon content and their relatively simple heat treatment techniques can produce different microstructures with one or more phases [91, 92]. A pearlitic microstructure (a mixture of ferrite and cementite phases) is produced in steels through eutectoid transformation with a carbon content of ~ 0.8 (in wt. %). The mechanical properties of pearlite is inversely proportional to the square root of the interlamellar spacing between ferrite and cementite lamellae [12]. Also, a combination of hard martensite and soft ferrite phase can be obtained through intercritical annealing (between $Ae_1$ and $Ae_3$ temperatures) of plain carbon steels. These steels are also known as dual phase steels known for their high strength to weight ratio and better formability characteristics [93]. Highly dislocated martensitic structures with different morphologies (lath and plate) are obtained by
altering the carbon content and cooling rate (e.g. quenching treatment). These structures have superior strength, but their brittle nature can often be vulnerable during high-stress abrasive conditions [94]. Therefore, abrasive environments often demand steels that can offer both better strength and toughness. Especially, conventional bainitic steels formed through isothermal transformation provide better abrasion properties. This is due to the presence of ductile retained austenite phase in combination with different forms of bainite (upper and lower bainite) [37]. A detailed study on the abrasive wear behaviour of the aforementioned steels will be dealt in the subsequent sections.

a) Pearlitic steels

Pearlite microstructure is a eutectoid mixture of ferrite and cementite lamellae. In pearlitic steels, the abrasion wear resistance depends mainly on the pearlite characteristics, which are a function of carbon content and the heat treatment. An increase in carbon content, increases the abrasion wear resistance [76, 95, 96]. This phenomenon is effective in hypo-eutectoid steels (< 0.8C wt. %) than in hyper-eutectoid steels (> 0.8C wt. %). The mechanical properties of eutectoid steels generally follow the Hall-Petch relationship. The hardness, strength and toughness are inversely proportional to the square root of interlamellar spacing. However, studies have found that the wear rate does not follow the Hall-Petch relationship. On the contrary, the wear rate is reduced with a decrease in the pearlite interlamellar spacing [96-100].

In the case of pearlite, the abrasion resistance is high due to the synergetic action of cementite and ferrite. The ductile nature of the ferrite region between cementite lamellae favours work-hardening behaviour during abrasion. As a result, it leads to the plastic deformation of hard cementite lamellae in the sub-surface layer (i.e. layer beneath the worn surface). Moreover, the pearlite lamellae fractures and forms nano size particles during abrasion. These particles attach themselves to the sub-surface layer resulting in a reduction of the wear rate. The ability to form such hardened sub-surface layers depend on the pearlite characteristics (i.e. cementite thickness and interlamellar spacing). For example, in the case of coarse pearlite, the tendency to form hardened sub-surface layers is relatively less, thereby leading to higher wear rates. Primarily, the wear resistance of pearlitic steels increases with a decreasing interlamellar spacing. A reduce in the interlamellar spacing diminishes the cutting efficiency of the abrasives leading to a decrease in the wear rate (Fig. 26).
However, beyond a threshold level, a further reduction in their interlamellar spacing has a negligible effect on their wear resistance [12, 95, 97, 98, 100].

![Interlamellar Spacing vs Wear Resistance](image)

Figure 2.26: Effect of interlamellar spacing on the abrasive wear resistance of a pearlitic steel for different sliding distances [12].

**b) Dual phase steels**

Dual phase steels consisting of hard martensite and soft ferrite phases are known for their high strength to weight ratio and better formability characteristics. Such steels are produced by intercritical annealing (between Ae₁ and Ae₃ temperatures) of plain or low carbon steels [101]. The mechanical behaviour of these steels are based on the volume fraction and size of the existing phases (ferrite and martensite). The characteristics of these microstructural phases can be altered by suitable heat treatment cycles [34]. The hard/brittle martensitic phase provides the abrasion resistance, whereas the ferritic phase improves the work-hardening capability and facilitates crack tip blunting. This work-hardening behaviour of ferrite ultimately results in an increment in the hardness of the deformed and sub-surface layers. In general, the wear resistance of a dual phase steel is proportional to the volume fraction of martensite [34, 102] (Fig. 2.27). Moreover, it was found that coarser martensite was more efficient in resisting the abrading action, than the finer martensite islands.
c) Quenched and tempered steels

Fully martensitic steels are seldom used in industries due to the highly dislocated martensitic structures (lath/plate morphologies with a BCC/BCT lattice structure depending on carbon content) in their matrix. Despite superior strength, these structures are highly brittle as they formed by shear transformation mechanism at a very high cooling rate [91, 94, 103]. Therefore, such martensitic microstructures are tempered to impart the desired ductility to the microstructure. During tempering, the martensitic structures undergo annihilation process and the laths decompose into ferrite and cementite particles. Therefore, their microstructure matrix consists of cementite particles distributed in a ferrite matrix. These steels undergo very little microstructural changes during abrasion. Furthermore, their work-hardening behaviour is less leading to a lower abrasion resistance. The resistance towards abrasion is largely determined by the volume fraction of ferrite and the cementite distribution [25]. However, the mechanical properties of such steels can further be improved by tempering in one or more stages. After quenching, a series of tempering heat treatment cycles can produce a tempered martensitic structure with evenly distributed carbides (primary and secondary). These steels are also known as tool steels [91, 94, 103]. During abrasion, the tempered martensitic matrix is worn out by the
abrasive particles leaving the carbides unaffected and protruding from the worn surface. Thereby, the carbides are responsible for bearing most of the applied loads. As the wear progresses, the carbides crack and are removed as fragments, leading to cavities on the surface of the worn track (Fig. 2.28). However, the ability to pull out the carbides largely depends on the penetration depth of the abrasive particles (i.e. abrasive particle size and severity of abrasive wear). Studies have found that the volume fraction and inter particle distance between the carbides plays a critical roles in the abrasion resistance in these steels [104].

Figure 2.28: Worn out section of AISI D2 tool steel subjected to three-body abrasive wear [104].

d) Bainitic steels

Bainitic steels were often considered as a potential replacement of pearlitic steels in railways [105]. Rail steels are often subjected to a combination of both adhesive and abrasive wear due to rolling contact fatigue (RCF) (i.e. constant wheel-rail interactions) [106-108]. Recently, low carbon bainitic steels were employed in railway crossings (especially in nose) to display superior impact wear resistance. In addition, laboratory tribological experiments simulating on-site rail conditions were performed on bainitic steels in comparison with pearlitic steels. The former displayed superior resistance to RCF cracks, contrarily their wear rate was higher than the latter [109]. Despite limited investigations revealing contradictory results, researchers have shown a lot of promise towards development of bainitic steels for tribological applications [110-115].
Davenport and Bain (1930) discovered bainite microstructure as an ‘acicular, dark etching aggregate’, during the isothermal decomposition of austenite [37]. In general, bainite has been classified into ‘upper’ and ‘lower’ bainite based on the transformation temperature and alloy composition [103, 116, 117]. Upper bainite is formed at higher temperatures, in a range of 550-400°C, with the carbides between the ferritic plates. In lower bainite, the low transformation temperature, in a range of 400-250°C, permits a limited amount of carbon to precipitate in the growing ferritic plates [116, 118]. On continuous cooling, bainite with other morphologies can be obtained such as granular bainite, where the microstructure consists of a ferrite matrix, islands of martensite and retained austenite [119]. Thereby, the term ‘bainite’ denotes a variety of complex microstructures with varied mechanical properties in terms of strength and toughness [120-123].

Recently, a combination of alloy addition (Si and Mn) and thermomechanical processing have produced a simpler bainitic microstructure (TRIP steels) consisting of bainitic ferrite plates surrounded by films of carbon rich retained austenite [41, 124, 125]. The carbide-free bainitic microstructure enhances the strength-ductility balance in steels [126, 127]. During plastic deformation, the retained austenite is transformed into martensite increasing the strain-hardening rate, termed as TRansformation Induced Plasticity (TRIP) effect [128-131]. The wear behaviour of TRIP steels matched the performance of pearlitic steels in rail industries [124]. Subsequently, the existing carbide-free bainitic microstructures were further refined to nano scales to produce advanced high strength TRIP steels, also referred as nanobainitic steels. The microstructure consists of very fine bainitic ferrite and retained austenite (i.e. film or blocky morphologies, Fig. 2.29). Such a unique microstructure provides an unusual combination of superior strength (~ 2 GPa) and fracture toughness (130 MPa/m^1/2) [127, 132, 133]. The presence of retained austenite in the matrix favours work-hardening behaviour (i.e. TRIP effect) resulting in better abrasion and adhesion wear resistance [37, 41, 121, 122, 134].
In general, the characteristics of the nanobainite microstructure are largely dependent on the isothermal bainitic transformation temperatures [135, 136]. Depending on the transformation temperature, the characteristics (morphology, size, volume fraction and carbon content) of bainitic ferrite and retained austenite can be varied [37, 121, 122, 137, 138]. Especially in the case of retained austenite, distinct morphologies can greatly influence the strain-induced martensitic transformation (i.e. TRIP effect). For instance, film morphology retained austenite exhibits relatively higher mechanical stability than the blocky ones [137, 139]. If the mechanical stability of the retained austenite is low, then the retained austenite will transform at lower strains (i.e. lower than yield strength, $\sigma_y$) and will not contribute towards work-hardening behaviour. On contrary, highly stable retained austenite does not provide any beneficial effect on mechanical properties as the required strain for transformation may exceed the ultimate tensile strength, $\sigma_{uts}$, of the steel [108, 140-143]. Indeed, the retained austenite with a stability that lies between a range of yield and ultimate tensile strengths (optimum) has the most beneficial effects on the work-hardening behaviour and resultant mechanical response of steels [144, 145] (Fig. 2.30). Consequently, the stability of retained austenite strongly depends on different parameters such as carbon equivalent [140], size, morphology [145] and the surrounding phases [143]. Moreover, the aforementioned factors are interlinked with one another to affect the mechanical stability of retained austenite. This clearly outlines the broad spectrum of mechanical properties offered by the different class of bainitic microstructures (i.e. conventional, carbide-free and nanobainite) and also in identifying themselves as a probable candidate for tribological applications.
The abovementioned steels and their corresponding effective microstructural parameters towards abrasion have been summarized (Table 2.2). This clearly shows that different phases in a microstructure act in synergy towards abrasion. In other words, each phase (i.e. microstructural constituent) can display a unique abrasive wear behaviour. This can be explained by the following equation.

\[ \varepsilon = V_\alpha \varepsilon_1 + V_\beta \varepsilon_2 \]  \hspace{1cm} 2.9

where \( V_\alpha \) and \( V_\beta \) are the volume fraction of \( \alpha \) and \( \beta \) individual phases and \( \varepsilon_1 \) and \( \varepsilon_2 \) are the wear resistance of their corresponding phases [146]. However, this equation holds true for simple microstructures (i.e. number of phases) and depends on factors such as work-hardening [146].
Table 2.2: Different steels and their corresponding effective microstructural parameters towards abrasion [25, 34, 72, 76, 91, 94, 95-104, 120-123]

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Different types of steels in abrasive wear</th>
<th>Microstructure</th>
<th>Effective microstructural parameters</th>
<th>Work-hardening behaviour</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Pearlitic steels</td>
<td>Pearlite</td>
<td>Interlamellar spacing between ferrite and cementite lamellae.</td>
<td>High</td>
</tr>
<tr>
<td>2.</td>
<td>Dual-phase steels</td>
<td>Ferrite and martensite</td>
<td>Volume fraction of ferrite and martensite.</td>
<td>Medium to high</td>
</tr>
<tr>
<td>3.</td>
<td>Quenched and tempered steels</td>
<td>Ferrite and cementite</td>
<td>Volume fraction of ferrite and morphology of cementite.</td>
<td>Low to medium</td>
</tr>
<tr>
<td>4.</td>
<td>Quenched and tempering (in stages-tool steels)</td>
<td>Tempered martensite and carbides (Fe₃C)</td>
<td>Volume fraction and inter-particle distance between carbides</td>
<td>Low to medium</td>
</tr>
<tr>
<td>5.</td>
<td>Bainitic steels (TRIP steels)</td>
<td>Bainitic ferrite and retained austenite</td>
<td>Volume fraction, morphology and size of retained austenite</td>
<td>High</td>
</tr>
</tbody>
</table>

Furthermore, the ability of the microstructure to undergo work-hardening makes them highly suitable for abrasive conditions. A beneficial impact of the work-hardening behaviour (i.e. hardness increase on worn surface) on the abrasive wear resistance of a microstructure has been largely emphasized. However, the hardness factor is a variable one, as the process of abrasion induces more microstructural changes leading to change in the hardness before and after the wear test. On the other hand, there is a linear relationship between the wear resistance and the bulk hardness of pure metals and/or homogenous microstructures. Eventually, this leads to the contemporary debate on the effect of surface and bulk hardness on abrasion. Ultimately, this implies that the abrasive wear behaviour of the microstructures is complex and governed by a system of interlinked parameters.

_Extensive research has shown that steels with high work-hardening behaviour are better equipped to combat high-stress abrasion. There has been substantial evidence to emphasize the importance of final surface hardness or hardness increment_
after abrasive wear. However, there is still a lack of knowledge on the optimum level of work-hardening, which can be beneficial to abrasion. To address this, the abrasive wear behaviour of a microstructure (e.g. advanced TRIP steels- with nanobainite structure) that can display a wide range of work-hardening behaviour needs to be studied.

2.9 Summary

This chapter discussed the complex tribological system with regards to abrasive wear and a detailed explanation on the factors affecting it. Salient points of this review are summarized as follows:

1. Among the various types of wear encountered in industrial applications, the impact of abrasive wear is quite severe (~50%) involving significant revenue losses. The abrasive wear process is classified based on the severity of particle indentation and their freedom of movement within mating surfaces. In addition, the abrasion induces one or more material removal mechanisms accounting for the material loss in ferrous alloys.

2. An appropriate selection of laboratory abrasive wear test is vital, as their wear performance is system related. The two-body abrasive wear test (pin-on-disc) offers more advantages (i.e. in terms of ease and reproducibility of tests) than a three-body abrasive wear test. However, a scratch test provides a better understanding on the material removal mechanism, as it facilitates better control over the tribological systems (e.g. abrasive environment).

3. In a general note, the characteristics of the abrasive particles (morphology-size, hardness and toughness) influence the abrasive wear to a greater extent. However, for a given two-body abrasive environment, there is often a high probability of non-uniform abrasive particle distribution, i.e. in terms of morphology and orientation (attack angle). Therefore, an attempt will be made to study the individual effect of abrasive particle characteristics and understand their response during abrasive wear.

4. Since metallic alloys form a major part of industrial equipment, factors (mechanical and metallurgical properties) affecting the abrasive wear resistance
of ferrous alloys were discussed. In the case of mechanical properties, a strong
correlation was observed between hardness, toughness and the wear resistance of
a steel. Moreover, there has been a lot of uncertainty for a possible explanation
over the differential abrasive wear response of metallic alloys heat treated having
similar hardness.

5. Several theories have elucidated the role of hardness in abrasive wear resistance.
Amongst these models, there has been a strong inclination in linking the study of
hardness towards the plastic flow of the material. On the other hand, there has
been several arguments stressing the importance of worn surface (i.e. work-
hardened) hardness rather than the bulk hardness. Therefore, the main objective
of this thesis is to study the abrasive wear behaviour of steels heat-treated to
similar hardness levels and understand how the initial hardness affects their
abrasive wear resistance.

6. Despite hardness and toughness being key factors that influence abrasive wear,
these factors are greatly governed by the characteristics of a microstructure (of a
steel alloy). Extensive research has revealed that multi-phase microstructures (i.e. a
combination of one or more phases) are beneficial in high-stress abrasive
environments (e.g. mining and mineral processing industries). The presence of a
ductile phase in their matrix induces work-hardening phenomenon, thereby
leading to a reduced material loss. Conversely, there is very little information on
the extent or the optimum level of work-hardening that can enhance the abrasive
wear resistance of a steel alloy.

7. Different grades of steels used in various abrasive environments were briefly
discussed. In general, pearlitic steels displayed superior performance that was
mostly ascribed to their interlamellar spacing (i.e. space between ferrite and
cementite lamellae) and work-hardening behaviour. Meanwhile, bainitic steels
(e.g. advanced TRIP steels) with the presence of retained austenite in their
microstructure matrix were found to undergo TRIP effect (i.e. work-hardening
behaviour). The ability to investigate the TRIP effect by altering the
characteristics of retained austenite (volume fraction, morphology and carbon
content) makes the bainitic steels highly attractive for the current study.
Consequently, this study can aid us in determining the optimum level of work-hardening that can be advantageous for the abrasive wear.

2.10 Objectives

The potential research gaps mentioned in this section can be addressed through the following objectives:

- To evaluate the role of abrasive particle characteristics on the process of material removal in two-body abrasion. Thereby, understanding their abrading efficiency through different particle deterioration mechanisms.
- To understand the effect of microstructure characteristics on the two-body abrasive wear behaviour.
- To study the influence of microstructural constituents on the two-body abrasive wear behaviour of advanced TRIP steels (nanobainite).
- To investigate the abrasive wear behaviour of microstructures under controlled test conditions using a scratch-test method.
Chapter 3

Experimental procedure

3.1. Introduction

This chapter will discuss the materials and the experimental techniques employed in the current study. In addition, the different tribological instruments (Pin-on-disc and scratch tester) for conducting the two-body abrasive wear tests will be explained in detail here.

3.2 Experimental materials

3.2.1 Steel alloys

Four steels with different chemical compositions were used in the current investigation (Table 3.1). All steels were subjected to a series of heat treatment schedules to achieve the desired microstructures, except steel C which was used in the as-received condition with a fully pearlitic microstructure (Table 3.1). In Chapter 5, four distinct microstructures namely: bainite (steel A), tempered martensite (steel A), martensite (steel B) and pearlite (steel C) were subjected to a silicon carbide, i.e. SiC abrasive environment to study the impact of microstructure characteristics in two-body abrasive wear. Further, two different abrasive environments, namely, SiC and Alumina with distinctive characteristics (i.e. particle size and density) were chosen to understand the role of abrasive particles in two-body abrasive wear (Chapter 4). Industrial grade abrasive papers of SiC and Alumina with distinctive characteristics (particle size, density, hardness, etc.) were employed to understand their particle deterioration mechanisms and efficiency during abrasion. A high carbon-high alloying content (Steel D) was chosen to investigate the impact the microstructural constituents in two-body abrasive wear (Chapter 6). The chemical composition of the steel D enabled us to produce fully bainitic microstructures through isothermal bainitic transformation.
with distinctive microstructural constituents (size, morphology and carbon content). Furthermore, the process of material removal in microstructures was evaluated under controlled test conditions using a scratch-test in Chapter 7. A detailed description of the scratch-test set-up and the parameters will be discussed in detail in 3.4.2.

Table 3.1: Chemical composition of steels (in wt. %).

<table>
<thead>
<tr>
<th>Steels</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>Al</th>
<th>Co</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.261</td>
<td>1.61</td>
<td>3.51</td>
<td>1.05</td>
<td>0.275</td>
<td>1.69</td>
<td>0.78</td>
<td>0.49</td>
</tr>
<tr>
<td>B</td>
<td>0.046</td>
<td>0.264</td>
<td>1.84</td>
<td>0.0078</td>
<td>0.251</td>
<td>0.0087</td>
<td>0.0702</td>
<td>0.0066</td>
</tr>
<tr>
<td>C</td>
<td>0.844</td>
<td>0.27</td>
<td>0.67</td>
<td>0.02</td>
<td>0.006</td>
<td>0.04</td>
<td>0.002</td>
<td>0.004</td>
</tr>
<tr>
<td>D</td>
<td>0.79</td>
<td>1.5</td>
<td>1.98</td>
<td>0.98</td>
<td>0.24</td>
<td>---</td>
<td>1.06</td>
<td>1.58</td>
</tr>
</tbody>
</table>

3.3 Thermomechanical processing

3.3.1 Furnaces

Two different furnaces namely: ceramic tube and muffle furnace were employed in the current study. The ceramic tube furnace (Fig. 3.1) was exclusively used for homogenizing the hot rolled samples, as it offered a high working temperature of 1550°C and an effective heating chamber. The chamber is a ceramic tube (~80 mm Ø and 1400 mm long), which runs through the middle of the furnace and surrounded by a heating element. This confined chamber can be purged with Argon gas, thereby offering more protection from oxidation and reducing the effect of decarburization in steels.
A Labec muffle furnace (Fig. 3.2) was primarily used for preheating and other subsequent heat treatment schedules, which will be discussed in detail in the corresponding chapters. The furnace is ceramic lined and heated by the electric elements with a maximum working temperature of 1200°C. In addition, it houses a rear vent for gas purging and an N type thermocouple for monitoring the furnace temperature. The temperature of the furnace can be controlled using a digital Proportional Integral Differential (PID) controller at the front.
3.3.2. Hot rolling

A laboratory mill capable of hot (up to 1100°C) and room temperature rolling was used in the current study (Fig. 3.3). The mill consisted rolls of 350 mm diameter, a face width of 450 mm and a surface speed of 16 m/min. During hot-rolling, the surface temperature of the rolled samples was measured using an optical pyrometer-Raytech: Thermo Hunter. The hot rolling process was mostly conducted to reduce the initial thickness of as-received steels.

![Figure 3.3: Rolling mill.](image-url)

3.3.3 Salt-bath furnace

A Nabertherm WB-20 martempering furnace (often referred to as salt-bath furnace) was used for austempering (isothermal bainitic transformation) process (Fig. 3.4). The furnace is filled with neutral salt that offers rapid heat transmission to the samples subjected to heat treatment. The bath consists of immersion heating elements providing a uniformity in the temperature. The working temperature of furnace is in a range of 180-500°C. A hardened steel cover at the top of the bath enables ease of operation and safety precaution from molten salts.
3.4 Tribological instruments

3.4.1 Pin-on-disc tribometer

Friction and abrasive wear properties of the microstructures were analysed using a CSM high temperature pin-on-disc tribometer (Fig. 3.5). The tribometer has been precisely calibrated to perform the tribological analysis at elevated temperatures, according to ASTM G99 and ASTM G133. The machine has a PC interface to acquire data and control the test parameters such as load, sliding distance and speed. The set up consists of a lever arm, which holds a pin sample (approx. 6 mm Ø and 50 mm long) at ~45° to a flat circular disc (Fig. 3.5a). During the test, the stationary pin comes in contact with the rotating disc. Multiple wear tracks can be studied by adjusting the lateral movement of the lever arm. A variable speed motor is responsible for maintaining a constant speed of the disc. Depending on the disc and/or pin material, different wear environments can be simulated. Friction coefficient measurements were based on lever deflections using a Linear Variable Differential Transformer (LVDT) sensor on the tribometer. The tribometer has an automatic shut-off, in the case of any emergencies (e.g. exceeding the friction coefficient threshold). The disc is housed in a heater with a thermocouple underneath it to measure the temperature changes during the tests (Fig. 3.5b). An efficient heating/cooling system ensures the desired temperature during the test conditions. In the current study, a minimum of four abrasive wear tests were conducted to ensure repeatability and the ± range is reported for each testing condition.
3.4.2 Scratch tester

Scratch tester is a dedicated instrument for evaluating the surface properties of films and coatings (i.e. adhesive strength). This technique is widely employed to evaluate the abrasive resistance of materials by scratch damage (Fig. 3.6a). The scratch tester is a servo-controlled single axis machine with a conical stylus (Fig. 3.6b). The stylus has a spherical tip radius of ~820 µm and is made of tungsten carbide and cobalt. This instrument is designed to create a controlled scratch on the surface of interest under the action of a normal load. A servo-hydraulic Instron testing machine supplies the load (100-4000 N) to the stylus. The sample (~55 mm × 45 mm × 8 mm) is fixed rigidly to the instrument base with the aid of fixtures. The robust design of the stylus tip generates a scratch under a constant or incremental normal load. There is a load cell in the scratch tester which measures the lateral (tangential) force during a progressive loading condition. In addition, it is a valuable tool for measuring the friction force during the scratch test.
3.5. Characterization techniques

3.5.1 Metallography

Metallographic observations were made on the longitudinal sections (~10 mm × 4 mm × 3 mm) of the heat-treated steel samples. Sectioning was carried out using a high precision cut-off machine (Accutom-50). The samples were mounted in a multi-fast, a conductive polymer and mechanically ground using 240, 600 and 1200 grit silicon carbide papers. The ground samples were successively polished in a series polishing pads of 9 µm, 6 µm, 3 µm and 1 µm using Tegra-Force semi-automatic polishing unit. This was followed by polishing the sample surface in a Tegra-Force semi-automatic polishing unit using 9, 6, 3 and 1 µm diamond paste. The samples were ultrasonically cleaned between different stages of polishing. Finally, a 4 vol. % nital solution was used to etch the polished samples for optical and scanning electron microscopy. Optical microscopic examinations of the microstructures were performed using an Olympus PMG 3 reflected Light Microscope equipped with an Olympus DP10 digital camera.

3.5.2 Micro-hardness tester

The bulk hardness of the microstructures before and after the wear test was measured using a Struers-Durascan 20 micro-hardness (Vickers) tester (Fig. 3.7). It is equipped with a vertical movable head, which consists of a six-position turret. The turret houses the optical lens (10x and 60x), zoom lens (1x and 2x), auto focus (AF) camera and indenter. The test loads between 0.098-98 N with a series of dwell time makes the equipment ideal for studying areas in micron scale. The instrument is interfaced with a PC and ecos Workflow software enables both manual and automatic hardness measurement [147].
3.5.3 XRD characterization

X-ray diffraction was employed in the current study to determine the characteristics of retained austenite phase (volume fraction and carbon content) in advanced TRIP steel microstructures. A PANalytical X’pert MRD XL diffractometer (Fig. 3.8) is a versatile instrument with a silicon monochromated CuK\(\alpha\) radiation and offering different scan types (e.g. point and line focus). This robust technique is widely used in measuring crystal lattice parameter, phase identification and residual stress. The diffraction pattern was analysed using HighScore Plus published by PANalytical Inc.
3.5.4 Electron microscopy

a) Scanning electron microscope (SEM): A high resolution scanning electron microscope (Supra 55VP FEG SEM) was extensively used to characterize the microstructures (Fig. 3.9). It encompasses a Schottky-type field-emission electron source and a beam booster for ensuring optimal electron performance at different accelerating voltages. In addition, the variable pressure (VP) mode enables the investigation of materials that offer little or no conductivity. The instrument incorporates several detectors such as, Electron Backscatter Diffraction (EBSD), Variable Pressure Secondary Electron (VPSE) detector, Angular Selective Backscatter (AsB) and Energy Dispersive X-ray (EDX) detector enabling versatility. However, only VPSE and EDX detectors were used in the current study for microstructure and post-wear examinations. FEI Quanta 3D FEG FIB-SEM microscope was used to prepare TEM foils (~6×8 µm²) from the deformed regions of the sub-surface layers in the abraded microstructures. The microscope houses a field-emission gun with a gallium focussed ion beam and a platinum gas-injection system. The focused ion beam (FIB) milling technique involved a series of precise steps namely, identifying the region of interest, platinum deposition, bulk-out, U- cut, lift-out, mounting, thinning and cleaning. The prepared foils were further characterized using a Transmission Electron Microscope, JEOL JEM-2100F.

Figure 3.9: Scanning electron microscope – Supra 55VP FEG SEM.

b) Transmission electron microscope (TEM): The characterization of the microstructural constituents was carried out using a transmission electron microscope, Philips CM-20 with a Lanthanum Hexaboride (LaB₆) filament and operating at an accelerating voltage of 200 kV (Fig. 3.10a). This instrument was highly conducive for ferromagnetic materials with negligible beam deflection. This enabled to conduct conventional electron microscopy in nanobainitic steel with austenite phase (FCC). However, a high-performance Transmission Electron Microscope, (JEOL JEM-
2100F, Fig. 3.10b) fitted with a NanoMEGAS ASTAR Automated Crystal Orientation and phase mapping was employed to investigate the sub-surface regions (i.e. beneath the worn out surface). The microscope was operated at 200 kV coupled with a Gatan Orius SC1000 fast-rate acquisition high-resolution camera of 11 Mpixel. The characterization was performed in a nanobeam mode using a condenser aperture of 10 \( \mu \)m, camera length of 15 cm and nominal spot size of 1.6 mm. The NanoMEGAS ASTAR system focussed the primary electron beam on the foil of interest and the resultant nanobeam spot diffraction spots were captured by an ultrafast CCD camera. One of the major highlights of ASTAR system is that, it allowed precession of the incident beam, where the incident beam rocked above the specimen in a conical fashion leading to a hollow cone illumination. Therefore such precession diffraction led to a greater reliability of orientation determination [148-152]. Crystallite orientation data (Euler angles) obtained as a text file, which was further exported to the HKL Technology/Oxford instruments Channel 5 for post processing.

![Figure 3.10: Transmission electron microscope: a) Philips CM-20 and b) JEOL JEM-2100F.](image)

### 3.5.5 Optical profilometer

The microstructure (i.e. pin sample) surface subjected to abrasion was analysed using an Alicona InfiniteFocus optical profilometer (Fig. 3.11). The profilometer combines
both 3D micro coordinate measurement and surface roughness techniques. The presence of LED ring and coaxial lighting facilitates analysis on surfaces with intricate geometry. The instrument is interfaced with a PC and a software to analyse the 3D topographical images. It can render high resolution 3D images based on the functionalities of the surface. These 3D images are contrast rich profiles, depending on the contours of the scanned surface and it can perform surface profile measurements [153].

Figure 3.11: Alicona InfiniteFocus optical profilometer [153].

The surface finish or roughness of any surface is quantified by the characteristics of peak and valleys [48]. The parameters that were primarily used in the current study are briefly explained.

a) $R_a$ – Arithmetic average roughness: It is the arithmetic average height of the irregularities (peaks and valleys) over a defined distance, $l$ (Fig. 3.12).

b) $R_q$ – Geometric average roughness: It (also known as root mean square, RMS), which provides a geometric average height of the irregularities over a defined distance, $l$. This parameter is a more sensitive to irregularities, making it more viable for surface roughness measurements (Fig. 3.12).

c) Peak and valley heights: The irregularities on the surface can be measured using the parameters $R_T$ and $R_Z$ (Fig. 3.12).

$R_T$- Maximum peak to valley height of roughness profile over an evaluation length.

$R_Z$- Mean peak to valley height of roughness profile over an evaluation length [48].
Figure 3.12: Schematic representation of surface profile irregularities and its parameters [8].
Chapter 4

Effect of abrasive particle characteristics in two-body abrasive wear behaviour

4.1 Introduction

In Chapter 2, the severity of abrasive wear and its impact on industrial applications was introduced. In two-body abrasive wear, the process of material removal appears when hard (abrasive) particles indent on a relatively soft surface of a material and move relative to it [2-5]. In this context, the characteristics of the abrasive particles (the freedom of movement, morphology, orientation, distribution etc.) play a pivotal role in affecting the amount of material loss [14, 16-18, 154-156]. Extensive research and several theories have been postulated based on the investigations of the abrasive environment.

The process of abrasive particle indentation and their mechanism of material removal is based on the particle size and morphology. There is a transition in the mode of material removal based on the particle morphology (e.g. rounded or sharp tips). Round or polyhedral particles can cause a progressive abrading action, whereas, sharp tips can easily cut through the material [10, 11, 19]. This can eventually lead to a difference in the characteristics of the wear debris generation and mechanism of particle deterioration. Moreover, in two-body abrasive system, the efficiency of the particles and the active wear loss over a defined sliding distance is often debatable. This is due to the restriction in their freedom of movement and repeated traversals induces significant particle deterioration during abrasion [11, 19, 20]. This emphasises the fact that the abrasive environment is quite dynamic and undergoes appreciable changes during abrasion.
As highlighted in section 2.5.2, not all abrasive particles are involved in the process of abrasion due to the non-uniform distribution of particle shape and orientation in a given abrasive environment [16, 17]. Therefore, it is vital to study such a complex and dynamic abrasive environment in two-body abrasive wear. In the current study, an attempt has been made to understand the efficiency of the abrasive particles during the abrasion process through interrupted abrasive wear tests. Topographical and EDX analysis were performed to understand the abrasive particle deterioration mechanisms and wear debris for different abrasive environments (e.g. particle size and type).

4.2 Experimental procedure

4.2.1 Materials

In the current study, steel C with a chemical composition of 0.84 %C, 0.27 %Si, 0.67 %Mn and 0.02 %Cr (in wt. %) was used in the as-received condition with a fully pearlitic microstructure. The steel sample (~10 mm × 4 mm × 3 mm) was mechanically polished using standard metallographic techniques, followed by etching in a 4 vol. % nital solution for microstructural characterization. The steel sample was machined in the form of a pin (~ 6 mm Ø and 50 mm long- Fig. 4.1a) for conducting the abrasive wear experiments in a CSM high temperature tribometer. The tip of the pin sample with an angular orientation of 45° was made to slide against an abrasive disc. The angular orientation of the pin ensured that the contact mechanics and the cross sectional area of the pin remained constant during the wear process. Different abrasive environments including silicon carbide (SiC) and alumina were employed by sticking industrial grade abrasive papers to the disc (Fig. 4.1b). ImageJ (Image processing and analysis in Java) software was used to measure the size of the abrasive particles. Five measurements were conducted for each condition and an average value was taken. The abrasive wear tests were performed at room temperature in an unlubricated condition with a constant speed of 0.2 m/s, a load of 9 N and a sliding distance of 300 m. Friction coefficient measurements were based on lever deflections using a Linear Variable Differential Transformer (LVDT) sensor on the tribometer. Before each test, the pin was ultrasonically cleaned in ethanol and weighed on a precise weighing balance to carry out the weight loss analysis after the wear test. The specific wear rate was computed based on the obtained weight loss data.
4.2.2 Characterization techniques

For the characterization of the abrasive papers, they were gold coated using a high vacuum coater, LEICA EM ACE600 operated at 40 mA for 100 s, to enhance the conductivity for electron microscopic investigations. The characterization of abrasive particles and the microstructure was investigated using scanning electron microscopy (SEM, SUPRA 55VP scanning electron microscope) with a SE2 detector operated at 10 kV and 20 kV for abrasive paper and microstructure characterization, respectively. EDX was also employed to analyse the chemical composition of the debris particles generated during abrasion. AZtec software was used to conduct the EDX analysis and map elements over a defined region. The topography of the deteriorated abrasive particles was investigated using an optical profilometer, Alicona-Infinite Focus by generating three-dimensional contrast rich images. Modular software supplemented the microscopic studies to produce the desired scans using the point selection technique and rendering optical 3D measurements based on depth profiles.

4.2.3 Types of abrasive wear tests

Tests undertaken in the current study concentrated on the influence of the characteristics of the abrasive particles (particle size and type) on the abrasive wear phenomenon. Therefore, two different types of wear tests were performed, namely: i) linear or progressive abrasive wear tests and ii) interrupted abrasive wear tests. In the linear wear test, the specific wear rate of the microstructure was calculated at the end, i.e. after the pin had completed its total sliding distance (i.e. 300 m) on a fixed track diameter. Meanwhile, in the interrupted abrasive test, the material loss of the microstructures was obtained at regular intervals (i.e. each 60 m) under identical test
conditions (i.e. same track diameter). It must be noted that the wearing surface of the microstructure was ultrasonically cleaned in ethanol, to eliminate the effect of clogging (accumulation of debris). This test was a valuable tool in revealing the extent of cutting efficiency of abrasive particles during the abrasion wear phenomenon. The specific wear rate of the pearlitic steel at different abrasive environments was calculated from the weight loss of the pins. The specific wear rate, \( K \), was determined by the volume of the material loss, \( V \), sliding distance, \( S \) and normal load, \( P \). Specific wear rate is given by the equation, \( K=\frac{V}{P*S} \) (mm\(^3\)/N.m) [157]. A minimum of four abrasive wear tests were performed for each testing condition (normal and interrupted abrasive wear tests) to ensure repeatability in the current study.

4.3 Results

4.3.1. Microstructure and abrasive particle characterization

The fully pearlitic microstructure consisted of ferrite and cementite lamellae with an interlamellar spacing of approximately 0.1 \( \mu \)m (Fig. 4.2a). The current study dealt with the evolution of abrasive particle characteristics during the abrasive wear behaviour of the pearlitic microstructure. A uniform cross-section (~ 500 \( \mu \)m \( \times \) ~ 500 \( \mu \)m) of the abrasive papers was analysed to measure the average abrasive particle size for different abrasive environments (silicon carbide and alumina papers, Figs. 4.2b-f). SiC abrasive particles displayed non-uniform morphology (particle size and shape) and were relatively less densely distributed than the alumina particles over a defined area (Figs. 4.2e and f). For SiC, the packing density (i.e. particle distribution over a defined area of ~ 500 \( \mu \)m \( \times \) ~ 500 \( \mu \)m) was measured as \( 1\times10^{-4} \), \( 1.68\times10^{-4} \) and \( 8.4\times10^{-4} \) (particles/\( \mu \)m\(^2\)) for the particle size of 58 \( \mu \)m, 26 \( \mu \)m and 15 \( \mu \)m, respectively (Figs. 4.2b-e). Similarly, the packing density of alumina was \( 2.68\times10^{-4} \) and \( 6.68\times10^{-4} \) (particles/\( \mu \)m\(^2\)) for the particle size of 41 \( \mu \)m and 20 \( \mu \)m respectively (Fig. 4.2e-f).
4.3.2 Effect of abrasive particle size on the specific wear rate

The abrasive particle size revealed a significant effect on the specific wear rate of pearlitic microstructure for both abrasive environments (Fig. 4.3). In general, the specific wear rate decreased with a reduction in the particle size. For SiC environment, the specific wear rate was measured as \((2\pm0.15) \times 10^{-4}\), \((1.6\pm0.07) \times 10^{-4}\), \((1.2\pm0.07) \times 10^{-4}\) (mm\(^3\)/N.m) for the particle size of 58 \(\mu\)m, 26 \(\mu\)m and 15 \(\mu\)m, respectively (Fig. 4.3a). Similarly, the specific wear rate for alumina environment was \((3.8\pm0.2) \times 10^{-4}\), \((2.7\pm0.3) \times 10^{-4}\) (mm\(^3\)/N.m) for the particle size of 41 \(\mu\)m and 20 \(\mu\)m respectively (Fig. 4.3b). It is interesting to note that the alumina abrasion environment instigated much higher material loss in the pearlitic steel compared with the SiC environment for a comparable particle size. For instance, the material loss induced by the 20 \(\mu\)m alumina particles was 1.5 times greater than that of 26 \(\mu\)m SiC particles (Fig. 4.3b). Similarly, 41 \(\mu\)m alumina particles led to a higher material loss when compared to a 58 \(\mu\)m SiC abrasive environment (Fig. 4.3a).
Figure 4.3: Effect of abrasive particle size on the specific wear rate of pearlite subjected to different abrasive particle sizes: a) SiC (58 µm, 26 µm and 15 µm) and b) Alumina (41 µm and 20 µm).
4.3.3 Effect of abrasive particle size on the coefficient of friction

Interestingly, the abrasive particle size also displayed a significant effect on the behavior of the friction coefficient curve. In the case of the silicon carbide environment, the friction coefficient was high during the initial running-in period, followed by a period during which it plunged down and then later it stabilized with a marginal increase in the friction coefficient for all particle sizes (Fig. 4.4a). However, the friction coefficient generally increased with a reduction in the SiC abrasive particle size. Frequent small fluctuations (peaks) were observed on the friction coefficient curves, though their amplitude gradually decreased as the particle size was reduced (Fig. 4.4a). The friction curve when subjected to alumina particles revealed unique characteristics compared with the SiC particles. The former displayed a pronounced increase in the friction coefficient after the initial running-in period, followed by a continuous rise in the friction coefficient. Besides small fluctuations, some drops in the friction coefficient curves appeared (Fig. 4.4b). In general, the friction coefficient increased with a decrease in particle size, similar to the SiC environment (Fig. 4.4).
Figure 4.4: Effect of abrasive particle size on the friction coefficient of pearlite subjected to different abrasive particle sizes: a) SiC (58 µm, 26 µm and 15 µm) and b) Alumina (41 µm and 20 µm).

4.3.4 Post wear analysis on the deteriorated abrasive papers

a) Microscopic characterization: The post wear analysis conducted on the wear tracks of the different abrasive environments (i.e. particle size and type) revealed the distinctive mechanism of abrasive particle deterioration (Fig. 4.5). In the case of the coarse particle conditions (SiC-58 µm and alumina-41 µm), it was observed that the majority of the particles lost their cutting edges, accompanied by regions of particle fracture with patches of debris (Figs. 4.5a and b). The severity of particle fracture increased with a reduction in the particle size and this phenomenon was common for both abrasive environments. Especially, in the case of finer SiC abrasive particles (i.e. 15 µm, Fig. 4.5c), there was a higher degree of abrasive particle fracture leading to an extensive debris layer. The debris was accumulated between the particles covering a major section of the wear track (as pointed out by arrows in Fig. 4.5c). Similar scenarios were observed in the case of abrasive particles of similar sizes (SiC-26 µm and alumina-20 µm, Figs. 4.5b and c). However, the debris layer and changes in the particle morphology (i.e. degree of particle fracture) was relatively less severe than that of SiC-15 µm.
After removing the debris, the extent of the abrasive particle deterioration can be clearly observed. In general, SiC displayed a higher degree of deterioration in comparison with alumina, irrespective of the particle size (Fig. 4.6). In the case of the coarse SiC-58 µm, most of the particles lost their cutting edges and voids were observed due to the particle detachment from the resin matrix (Fig. 4.6a). However, the scenario of particle fragmentation was more dominant with a reduction in the particle size (SiC-26 and 15 µm). Conversely, for the alumina abrasive environment (Alumina 41 and 20 µm) the abrasive particles were largely intact, though they mostly lost their cutting tips. It was interesting to note that particle detachment was more
confined to a coarse abrasive particle size (SiC-58 μm and alumina- 41 μm, Figs. 4.6a and d).

Figure 4.6: Microscopic analysis of deteriorated abrasive environment (without debris) on completion of 300 m sliding distance. a) SiC (~58 μm), b) SiC (~26 μm) c) SiC (~15 μm) d) alumina (~41 μm), and e) alumina (~20 μm).

b) Topographic investigations: Topographic investigations were employed on the uniform cross sections (~716 μm× ~514 μm) to examine the extent and mechanism of abrasive particle deterioration for both the SiC and alumina abrasive conditions (Figs. 4.7 and 4.8). The contrast rich profile revealed significant changes in the morphology of the abrasive particles before and after the abrasive wear test (i.e. on the completion of the total sliding distance of 300 m). It was revealed that the severity of the particle fracture (i.e. changes in particle morphology) increased as the particle size was reduced from 58 μm to 15 μm for SiC particles, similarly from 41 μm to 20 μm in the case of alumina. A reduction in the particle size also enhanced the particle fracture
phenomenon, progressively leading to debris accumulation on the wear tracks (i.e. SiC-15 µm and alumina-20 µm, Figs. 4.7f and 4.8d).

Figure 4.7: Topographic analysis of abrasive particles for different silicon carbide abrasive particle sizes: 58 µm: a - before wear test; b - after wear test; 26 µm: c - before wear test; d - after wear test and 15 µm: e - before wear test; f - after wear test.
Figure 4.8: Topographic analysis of abrasive particles for different alumina abrasive particle sizes: 41 μm: a - before wear test; b - after wear test and 20 μm; c - before wear test; d - after wear test.

The surface profile measurements of the abrasive papers prior and after the wear test determined the changes in the characteristics of the abrasive particles. This was evaluated based on the geometric average surface roughness, \( R_q \) and the mean peak to valley height, \( R_z \). Except for the finest abrasive environment (SiC-15 μm), the average geometric roughness, \( R_q \), decreased after the wear test (Table 4.1). A similar trend was observed with respect to the mean peak to valley height, \( R_z \), of the abrasive particles after the wear test. It was interesting to note that, the effect of \( R_q \) and \( R_z \) (i.e. amount of increase or decrease) on the abrasive particle size was significantly high for coarse (SiC-58 μm and alumina-41 μm) and fine abrasive environments (SiC-15 μm) (Table 4.1). In general, the surface profile measurements were consistent with the above topographic analysis.
Table 4.1: Surface profile measurements of different abrasive environments.

<table>
<thead>
<tr>
<th>Abrasive particle sizes</th>
<th>Rq (µm)</th>
<th>Rz (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before wear</td>
<td>After wear</td>
</tr>
<tr>
<td>SiC- 58 µm</td>
<td>11.4</td>
<td>4.7</td>
</tr>
<tr>
<td>SiC- 26 µm</td>
<td>5.7</td>
<td>5.2</td>
</tr>
<tr>
<td>SiC- 15 µm</td>
<td>1.6</td>
<td>2.9</td>
</tr>
<tr>
<td>Alumina-41 µm</td>
<td>10.2</td>
<td>6.35</td>
</tr>
<tr>
<td>Alumina- 20 µm</td>
<td>7.5</td>
<td>4.7</td>
</tr>
</tbody>
</table>

c) Debris analysis through EDX: The phenomenon of abrasive particle deterioration resulted in the generation of debris consisting of fractured abrasive particles and displaced material from the pearlitic microstructure. The nature of the debris obtained was largely unique for each abrasive environment (SiC and alumina, Figs. 4.9 and 4.10). Microscopic and EDX analysis of the debris generated during the wear tests revealed a significant influence of abrasive type and particle size on the material removal process (Figs. 4.9 and 4.10). The EDX maps of the abrasive papers confirmed the accumulation of metallic debris (green and red regions in Figs. 4.9b and c) between the abrasive particles (red and pink regions in Figs. 4.10b and c).
Figure 4.9: EDX analysis of the debris particles generated from SiC~58 µm (a-c) environment.
The debris were in the form of discontinuous chips for SiC, whilst continuous chips were a characteristic feature of alumina (Figs. 4.11a and d). Moreover, the level of particle deterioration was high for SiC, in contrast with the alumina particles that were largely efficient and intact, leading to the generation of continuous metallic chips (Figs. 4.11a and d). In the case of the former, the severity of the particle fragmentation increased proportionally with a decrease in particle size. However, the latter experienced relatively less fragmentation. Furthermore, the presence of metallic chips in the debris reduced substantially when subjected to the finer abrasive environment (SiC-15 µm, Fig. 4.11c).
d) **Interrupted abrasive wear tests:** Interrupted abrasive tests determined the unique evolution of the abrasive particles (SiC and alumina) during the test. The results also revealed the impact of abrasive type and the extent of cutting efficiency of the abrasive particles during abrasion (Fig. 4.12). For SiC, the amount of material lost was almost constant after a sliding distance of 120 m. In other words, the SiC particles were less active beyond the sliding distance of 120 m. In contrast, the threshold range of alumina particles (180 m) was higher than for the SiC. It must be noted that the contact area of the pin was cleaned after each interval. This drastically reduced the clogging effect of debris, leading to a significantly higher material loss than the normal abrasive tests.
In general, the alumina particles were more actively involved in the process of material removal than their counterpart, SiC.

![Figure 4.12: Effect of abrasive particle type as a function of material loss.](image)

The microscopic observations of the deteriorated abrasive particles (i.e. both SiC and alumina) at their corresponding threshold sliding distances aided in validating the interrupted abrasive test results. During the initial period of the wear test, fresh abrasive particles (SiC- 58 μm and alumina- 41 μm, Figs. 4.2b and e) were actively involved in the abrasion process. For the SiC-58 μm, the particles began to lose their cutting edges as the test progressed (Fig. 4.13). Meanwhile, some of the oversized (i.e. above the average size) SiC particles were detached from the resin matrix (i.e. the abrasive paper), leading to the formation of voids (Figs. 4.13a-e). The scenario of particle deterioration was more severe as the sliding distance increased (from 120 m to 300 m, Figs. 4.13a-c).
A similar phenomenon of particle deterioration was observed for the alumina-41 µm condition, but with very little particle detachment (Fig. 4.14). In other words, the alumina particles were largely intact with a negligible amount of voids (Fig. 4.14). Nevertheless, the fractured abrasive particles resulted in the accumulation of debris in the wear tracks from both the SiC and alumina environments. The debris accumulation phenomenon was dominant in alumina compared with the SiC particles (Figs. 4.13d-e and 4.14d-e). In general, the particles were largely inactive beyond the threshold level (i.e. SiC-120 m and alumina-180 m, Figs. 4.13 and 4.14), with the debris accumulating between the particles. Thereby, this led to a reduction in the cutting efficiency of the abrasive particles, which resulted in a near steady state material removal level beyond the threshold sliding distance (Fig. 4.12).
4.4 Discussion

The main aim of this chapter is to develop a critical understanding on the abrasive particle deterioration mechanisms during the two-body abrasive wear. This also examines the effect of particle characteristics, i.e. size and particle density in the process of material removal throughout abrasion. The following sections will also discuss the efficiency of abrasive particles throughout the two-body abrasive wear.

4.4.1 Effect of abrasive particle characteristics on particle deterioration mechanisms

In an abrasive wear regime, the level and nature of interaction between the abrasive environment and the test material has a dominant influence on the abrasive wear behaviour of the system. This is often true in the two-body sliding abrasive system,
where fixed abrasive particles exhibiting distinctive characteristics (particle size, type and packing density) result in a differential material loss [158-161]. The current study reveals that, in addition to the metallurgical factors of a material, the characteristics of the abrasive environment has a major impact on the amount of material loss (Fig. 4.3). The deterioration of abrasive particles through the course of abrasion involves a number of mechanisms. During the initial period of the abrasion test, the metallic pin is exposed to fresh abrasive particles, thereby leading to an active material removal process (Fig. 4.12) and this trend is quite common for both the SiC and alumina abrasive environments. However, as the test progresses the abrasive particles begin to lose their cutting efficiency (i.e. morphology and cutting edges) through their mechanical interaction with the pin surface, which is known as an attrition process [11, 162] (Fig. 4.6a). In most instances, the continuous interaction leads to an almost complete fracture (fragmentation) of the particles (Fig. 4.15b). Furthermore, with an increase in the number of traversals (sliding distance), shelling phenomenon occurs [11, 162] (Fig. 4.15a), which generally refers to the detachment of coarse particles from the resin. These are some of the dominant mechanisms that act either simultaneously or just in isolation, to drastically reduce the cutting efficiency of the abrasive particles.
Figure 4.15: Microscopic analysis of abrasive particle deterioration mechanisms: a) shelling b) fragmentation and c) simultaneous occurrence of attrition and fragmentation mechanisms and d) transition from abrasion to adhesion wear mechanism.

It is generally accepted that not all abrasive particles are involved in material removal process [10, 163]. This is largely due to the non-uniformity in the size, shape and orientation of the abrasive particles (Fig. 4.2). Nevertheless, the specific wear rate of the microstructure decreases with a reduction in the particle size for both abrasive environments in the current study (Fig. 4.3). Coarse particles result in a deep penetration into the abrading surface, leading to a significant material removal. Conversely for the finer abrasive environment, the particles are characterized with sharp tips and small contact area, resulting in relatively lesser penetration and material loss. On the other hand, the ratio of particle contact area with the resin to the total particle surface area (particle contact area/ total particle surface area) increases as the particle size decreases. This enhances a better bonding between the particles with the resin for the fine abrasive particle, leading to extensive abrading action during the wear testing. In other words, the fine particles were mostly subjected to a combined action of shear and normal forces during abrasion, leading to a pronounced fracture (i.e. fragmentation Fig. 4.15b). Conversely, the reduced particle contact area/total particle surface area ratio leads to weakening of the particle-resin bond strength for coarse particles. Furthermore, with the projection of a major portion of the particle outside the resin, it results in shelling (i.e. complete particle removal from the resin) [164]. Consequently, the coarse particles are not fully involved in the process of abrasion, leading to less fragmentation. In addition, their relatively less dense packing nature increases the average load acting per particle. This results in easier removal of the coarse particle (shelling phenomenon) compared with the fine, dense packed abrasive particle environment [164, 165]. It is important to note that for macro-scale abrasion (200-300 µm), the particle fracture follows Hall-Petch type relationship, i.e. the volume of material loss instigated is inversely proportional to the square root of the particle size. However, this does not hold true for micro-scale abrasion (10-50 µm), thereby finer abrasive particles wear comparatively faster than coarser particles [166]. It is also worth mentioning that abrasive environments with similar particle size (SiC-
26 µm and alumina-20 µm) are involved in triggering a differential specific wear rate (Fig. 4.3).

The above discussions are supported by the topographic observations from different abrasive environments (Figs. 4.7 and 4.8). The significant differences in their scales clearly depict the changes in the morphology (i.e. loss of cutting edges/fracture/shelling) of the abrasive particles after the wear test (Figs. 4.7b, d and e). The severity of particle fracture is dominant in the case of the fine abrasive particle environment, leading to the accumulation of debris on the wear tracks (clogging). It is necessary to note that the debris size and the space between the abrasive particles decreases with a reduction in the particle size, which facilitates clogging to a higher degree [10, 167]. Thereby, it produces a higher volume of metallic debris along with fragmented abrasive particles (i.e. scale bar augmentation in Fig. 4.7f), which is aptly supported by EDX analysis over the comparative debris accumulation for both abrasive environments (Figs. 4.9 and 4.10). In general, the clogging scenario is more dominant for the alumina abrasive environment (Fig. 4.8d) when compared with SiC. In fact, the dense packing nature of alumina enhances the abrading action over the SiC, resulting more metallic debris accumulation for a given particle size (Fig. 4.3). The extent of clogging influences the friction coefficient curve characteristics. Generally, the coefficient of friction increases with a reduction in the particle size (due to increased clogging).

Investigations on the extent and mechanism of abrasive particle deterioration are further strengthened by the surface profile measurements of the abraded papers (Table 4.1). A significant reduction in the $R_q$ and $R_z$ parameters for of SiC-58 µm and alumina-41 µm is observed at the end of the wear test, which may be attributed to dominant attrition mechanism (i.e. particles losing and/or blunting their cutting tips, Fig. 4.15a, Table 4.1). It is worth mentioning that the level of reduction in the mean peak to valley height, $R_z$, is substantially higher for SiC-58 µm than alumina-41 µm. This clearly indicates that SiC-58 µm abrasive particles are more prone to shelling (i.e. complete particle removal from the resin) than alumina-41 µm. In other words, alumina-41 µm is more efficient in the process of material removal. Conversely, the refinement of particle size leads to an increase in the geometric surface roughness, $R_q$ and the mean peak to valley height, $R_z$ parameters after the wear test. This in fact, emphasises the occurrence of clogging (i.e. dominant wear debris accumulation). As a
result, the metallic debris acts as a third body (interface) between the pin and the abrasive particles, ultimately leading to an adhesive action (i.e. metal-metal contact, Fig. 4.15d). These observations are quite explicit in deriving a conclusion that both adhesive and abrasive wear mechanisms are acting simultaneously for the finer SiC-15 µm abrasive environment, as reported elsewhere [10].

Furthermore, the metallic pin subjected to different abrasive environments can produce unique wear debris with respect to the nature of the metallic debris that are chipped away (Figs. 4.9a and 4.10a). Alumina produces continuous metallic chips, whilst discontinuous chips are generated by the SiC particles. Indeed, this would once again imply that the former is more efficient in abrading than the latter (Figs. 4.11a and d). In general, SiC abrasive particles lose their morphology by means of dominant particle deterioration mechanisms (attrition, shelling and/or fracture) leading to discontinuous chips. A decrease in the abrasive particle size (i.e. from 58 µm to 15 µm, Figs. 4.11a-c) has resulted in a reduction in the metallic chip formation in the debris. This could be attributed to the natural geometric effect of the abrasives, where coarser particles are more efficient in the material removal process during abrasion. The constituents or the characteristics of the debris are quite pivotal for the simultaneous occurrence of one or more wear mechanisms. As discussed earlier, the transition from abrasive to adhesive wear is possible mainly due to the concurrent occurrence of complete particle fragmentation (fracture mechanism) and chipping of metallic debris from the microstructure (Fig. 4.15d).

4.4.2 Evolution of abrasive particles during the two-body abrasive wear

Interrupted tests clearly show the evolution (i.e. deterioration) of the abrasive particles as a function of sliding distance during abrasion (Fig. 4.12). It is interesting to note that alumina with a relatively lower particle size (41 µm) is more efficient in instigating material removal than the coarse SiC particles (58 µm). This establishes the fact that apart from the characteristics (particle type) of the abrasive particle, other factors can significantly affect the abrasion wear phenomenon. For SiC-58 µm, attrition and shelling are quite dominant leading to significant deterioration of the particles (Figs. 4.13b-c). This is evident in the constant material loss instigated by the SiC abrasive particles from the pin (i.e. microstructure), after reaching the threshold sliding distance (i.e. 120 m, Fig. 4.12). Conversely, the alumina-41 µm particles are more proficient during the abrading action which is evident by the greater threshold sliding
distance (i.e. 180 m) than the counterpart SiC (Fig. 4.12). Nevertheless, attrition is quite pronounced, but with a negligible amount of shelling for the alumina (Fig. 4.6e). However, the alumina particles are largely intact on the resin paper, despite repeated traversals (Figs. 4.14b-e). It is also worth mentioning that abrasive environments with similar particle size (i.e. SiC–26 µm and alumina-20 µm) are involved in triggering a differential specific wear rate (Fig. 4.3). As mentioned earlier, the high packing density of alumina enhances its ability to continuously interact (more indentations) with the metallic surface, resulting in a significantly higher material loss than SiC.

These observations aid in identifying the dominant mechanisms that are actively participating in the abrasive environment during the abrasion process. The characteristics of the abrasive environment such as particle size, type and packing density are crucial in determining their abrading efficiency (threshold level) under a given condition. In general, the coarse particles (i.e. SiC-58 µm) are responsible for causing greater material loss due to their deep penetration capability. On the other hand, these particles are most likely to lose their morphology (i.e. attrition, Fig. 4.6a) and/or detach (i.e. shelling, Fig. 4.15a) from the resin. However, attrition and fracture phenomena occur simultaneously when examining finer abrasive particles (i.e. SiC-26 µm, Fig. 4.15c). Sharp tips and small contact area (i.e. SiC- 15 µm) can efficiently cut through the material, but they are more vulnerable to fragmentation (Fig. 4.15b), because of the high loads (combination of shear and normal forces) acting on them. To summarize, attrition, shelling and fracture are some of the principal mechanisms that govern the particle deterioration in a SiC abrasive environment. Conversely, parameters such as packing nature can have a large impact on the abrasion process. Especially, in the case of the alumina abrasives, their dense packing nature enables them to cause more indentations, leading to a high material loss through continuous metallic chips (Fig. 4.2e-f). The literature suggests that particle fracture during abrasion is largely based on its hardness and fracture toughness [168, 169]. However, despite alumina displaying a lower hardness (20 GPa) and fracture toughness (4.5 MPa/m^{1/2}), than SiC (hardness-23 GPa and fracture toughness-4.0 MPa/m^{1/2}) [170], their abrading efficiency is better than the SiC. This is because, that apart from the mechanical properties, factors such as packing nature can make a significant impact on the abrasion process. Consequently, the dense packing nature of alumina ensured a better abrading action, with the particles remaining largely intact, over a greater threshold level in a defined test condition.
4.5 Summary

In the current study, the effect of abrasive particle characteristics (size, type and particle density) in the two-body abrasive wear of a given microstructure (pearlite) was investigated. The following conclusions can be drawn from these investigations.

1. The characteristics of the abrasive environment (particle size and type) had a dominant influence in determining the abrasion wear behaviour of a material (i.e. microstructure). The specific wear rate of the microstructure decreased with a reduction in the particle size, irrespective of the particle type (SiC and alumina).

2. The abrasive particle size and distribution greatly governed the particle deterioration mechanisms. For example, attrition and shelling were dominant in the case of coarse abrasive particles, whereas, finer abrasive particles were more vulnerable to fragmentation.

3. The packing nature or density of the abrasive particles determined the abrading efficiency of an abrasive environment. The dense packing nature of the alumina abrasive environment led to a significantly higher material loss than SiC for similar particle size.
Chapter 5

*Influence of microstructures with similar hardness levels in the two-body abrasive wear behaviour*

5.1 Introduction

As explained in Chapter Two, abrasive wear is an undesirable material removal phenomenon occurring in most mineral and mining processing industries [2-5]. Sliding abrasion involves dissipation of frictional energy into heat. A major part of the frictional energy is expended towards the metallic surface (microstructure). In this context, the amount of frictional energy consumption has a major influence in determining the amount of material removal, which in turn, defines the abrasion resistance of the alloy. Moreover, it has been shown that there is a marked difference in the friction energy consumption of steels due to the distinct characteristics of the microstructural constituents [15, 22, 23].

Studies have shown a linear relationship exists between bulk hardness and abrasion resistance of microstructures with similar metallurgical structures but with different chemical compositions [24, 165]. However, it must be noted that the microstructural constituents significantly influence the bulk properties of steels such as hardness, flow stress and fracture toughness. It, is therefore, difficult to neglect the effect of the microstructural constituents in abrasion, as they influence the material removal mechanism in the microstructures [24, 27, 31-33, 76]. It was shown that an eutectoid steel heat-treated to different pearlitic structures (lamellar pearlite and spheroidized structure) produced different abrasive responses [23]. A significant difference in the abrasion resistance of commercial tool steels AISI D2 and O1 heat treated to similar hardness has also been observed due to the carbide morphology (i.e. plate-like and blocky ones) [104]. This emphasizes the fact that the metallurgical
structures can play an important role in determining the abrasion resistance of a material.

Moreover, in the two-body abrasive wear, dynamic changes occur both in the material (microstructure) and the abrasive particles (i.e. deterioration of abrasive particles) [163, 171]. In Chapter Four, it was demonstrated that the abrasive particle characteristics (size and particle density) greatly determined their abrading efficiency. Meanwhile, abrasion induces several morphological changes in the abraded surface, ultimately leading to a difference in its mechanical properties (hardness and fracture toughness) [172]. Despite hardness and toughness being key factors that influence abrasive wear, the abrasive wear resistance of a material is largely governed by its microstructural characteristics [9, 12, 13, 15, 25]. This has greatly motivated a comparative study on the two-body abrasive wear behaviour of single (martensite and tempered martensite) and multi-phase (bainite and pearlite) microstructures with similar hardness levels. Thereby, this can aid in understanding the relation between hardness, microstructure characteristics and the abrasive wear resistance of a material. An attempt has been made to analyse the sub-surface and topographical regions of the deformed microstructures. In addition, single-wear track investigation of microstructures has been undertaken, to understand their mode of material removal.

5.2 Experimental procedure

5.2.1 Materials

The materials used in the current investigation consisted of three different steel alloys (Table 5.1). These steels were subjected to different heat treatment routes to produce distinct microstructures, namely bainite, martensite, tempered martensite and pearlite all with similar hardness levels. Steels A and B were received as ingots and subjected to treatment at 1400°C for 24 hr in an argon gas atmosphere. Steel A was then austenitized at 1000°C for 30 min, followed by austempering in a salt bath furnace at 300°C. Afterwards, the samples were individually taken out from the salt bath at different holding times (0.1, 1, 3, 5, 10, 20, 30, 60, 180, 300, 360 and 1440 s) followed by water quenching to study the evolution of bainitic transformation. To produce a tempered martensitic microstructure, the fully austenitized structure of steel A was initially subjected to rapid water quenching resulting in a fully martensitic structure. It was then subjected to a set of tempering treatments (400°C, 500°C and 600°C at different times) to achieve the desired hardness level of ~ 350 HV0.01N. Based on this,
the tempering condition of 500°C for 3 hrs was chosen to achieve the target hardness. Steel B was austenitized at 900°C for 5 min, followed by rapid water quenching to obtain a fully martensitic microstructure. Steel C was used in the as-received condition and had a fully pearlitic microstructure.

Table 5.1: Chemical composition of the steels (in weight %).

<table>
<thead>
<tr>
<th>Alloys</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>Al</th>
<th>Co</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel A</td>
<td>0.261</td>
<td>1.61</td>
<td>3.51</td>
<td>1.05</td>
<td>0.275</td>
<td>1.69</td>
<td>0.78</td>
<td>0.49</td>
</tr>
<tr>
<td>Steel B</td>
<td>0.046</td>
<td>0.26</td>
<td>1.84</td>
<td>0.007</td>
<td>0.251</td>
<td>0.008</td>
<td>0.070</td>
<td>0.0066</td>
</tr>
<tr>
<td>Steel C</td>
<td>0.844</td>
<td>0.27</td>
<td>0.67</td>
<td>0.02</td>
<td>0.006</td>
<td>0.04</td>
<td>0.002</td>
<td>0.004</td>
</tr>
</tbody>
</table>

5.2.2 Characterization techniques

Hardness measurements were carried out at 0.01N with a dwell time of 15 s using a Struers, DuraScan micro-hardness machine. Ten hardness measurements were carried out for each microstructural condition (i.e. before and after the wear test) and an average was taken. Optical microscopy was performed using an Olympus PMG 3 Reflected Light Microscope fitted with an Olympus DP10 digital camera. Scanning electron microscopic (SEM, SUPRA 55VP operated at 20 kV with a SE2 detector) techniques were employed for microstructural characterization. The samples were prepared using standard metallographic techniques and etched in a 4 vol. % nital solution. To measure the volume fraction of retained austenite in the bainitic microstructure, the polished sample was further chemically treated using a solution of 80% hydrogen peroxide, 5% hydrofluoric acid and 15% water to minimize residual stress and avoid any phase transition during sample preparation. The volume fraction of retained austenite was then measured using the direct comparison method between the integrated intensities of \((200)_\gamma\), \((200)_\alpha\), \((220)_\gamma\) and \((220)_\alpha\). X-ray diffraction was undertaken using a Philips PW 1130 diffractometer with graphite monochromated \(\text{CuK}_\alpha\) radiation at 40 kV and 30 mA in the \(2\theta\) range of 30-120° at a rate of 0.02 degrees per 6 s. The topography of the abraded pin surface (i.e. surface roughness and groove characteristics) of different microstructures was studied three-dimensionally using an Alicona-Infinite Focus, optical profilometer. The surface roughness and the groove characteristics were analysed using optical 3D measurements. The modular software aided in defining the region of interest using a point selection technique, thereby
producing the desired scans or surface profile measurement. The surface profile of the abraded surfaces was quantified based on the characteristics of the peaks and valleys. $R_a$ and $R_q$ (also known as root mean square or RMS) represent the arithmetic and geometric average roughness, which is measured based on the height of the irregularities over a defined distance, denoted by $l$. $R_t$ and $R_z$ represent the maximum and mean peak to valley height in a surface profile, respectively [48].

### 5.2.3 Two-body abrasive wear tests

A CSM high temperature tribometer (as discussed in Chapter 3, Fig. 3.5) was used to study the abrasive wear behaviour of the microstructures. The heat treated samples were machined using an electron discharge machine into the form of a pin 60 mm long and 6 mm in diameter for the abrasive wear tests. The tip of the pin was chamfered to $45^\circ$, as the pin holder was inclined at $45^\circ$ to the disc. Therefore, the pin contact (i.e. the cross sectional area of the sample) remained constant throughout the test. A silicon carbide abrasive grit paper was stuck to the disc by an industrial glue. In the current study, silicon carbide abrasive grit papers of different particle sizes (58 μm, 25 μm and 12 μm) were employed. Subjecting the stationary pin to abrade against the abrasive disc simulates the two-body abrasive environment. The tests were conducted in an un lubricated condition with a constant speed (200 mm/s), load (9 N) and sliding distance (300 m). Before and after each test, the pin was ultrasonically cleaned in ethanol to minimize the presence of debris attached to the wear grooves. This was followed by the weight loss measurements. The specific wear rate of the microstructures was calculated based on the weight loss data. At least four tests were performed for each testing condition and an average specific wear rate was presented in the current study.

### 5.2.4 Single-track wear tests

To clearly understand the mechanism of material removal in the different microstructures, a single-track wear test was performed under controlled testing conditions. The chamfered tip surface of the pin was initially subjected to the standard metallographic technique. The pin was mechanically polished further using Oxide Polishing Suspensions (OPS). This led to a light etching of the surface enabling the observation of the interaction of the microstructural constituents with the abrasive particles. The pin was subjected to a minimal traverse (i.e. sliding distance of 20 mm
at a sliding speed of 20 mm/s) so that the wear tracks were not overrun by more than one particle. In other words, each wear track was created by a single abrasive particle. The nature of material displacement and the groove characteristics were investigated using scanning electron microscopy.

5.3 Results

The heat treatments were designed to produce distinct microstructures with similar hardness levels ranging from 330-360 HV$_{0.01N}$. The following describes the microstructural characteristics at different heat treatment conditions and their abrasive wear resistance in alliance with the abrasive environment.

5.3.1 Microstructural characterization

a) Bainite: The as-cast microstructure of steel A revealed an inhomogeneous and complex structure (Fig. 5.1a). Homogenization heat treatment resulted in a coarse martensitic microstructure with ~ 980 µm prior austenite grain size (as shown by dotted lines in Fig. 5.1b). The evolution of bainitic transformation during austempering was evident (Fig. 5.2). At an early stage of austempering, the hardness was significantly decreased from 415 HV$_{0.01N}$ at as-quenched condition (0.1 min) to 370 HV$_{0.01N}$ at 5 min holding time. Afterwards, the rate of hardness drop was gradually reduced with time. After the 300 min, the hardness became nearly constant (~350 HV$_{0.01N}$), suggesting the completion of bainitic phase transformation (Fig. 5.2). Therefore, the 300 min condition was chosen as an optimum time for the completion of bainite phase transformation in the current study. However, there was very little difference in the morphology of bainite and martensite, due to the low carbon content (0.26%C). Consequently, coarse plates were assigned as martensitic region and fine laths were considered as bainitic ferrite (Fig. 5.3). The ferritic lath, in general, tends to initially nucleate on the austenite grain boundary and grow in the grain interior. This was clearly evident here that the initiation of bainitic ferrite lath seemed to occur at the austenite grain boundary (as shown by arrows in Fig. 5.3).
It was apparent that the prior austenite grain size was relatively coarse (950 μm), and was comparable with the homogenization treatment of 1400°C (980 μm, Fig. 5.1b). This suggested that there would be some retained austenite present in the
microstructure that was subjected to the homogenization treatment. The presence of the retained austenite was not evident using SEM technique. Therefore, the XRD approach was used to clarify the presence of retained austenite in the microstructure. The volume fraction of retained austenite was calculated to be \(~18\%\) using direct comparison method, by comparing the integrated intensities of \((200)_\gamma\), \((200)_\alpha\), \((220)_\gamma\) and \((220)_\alpha\).

\textit{b) Tempered martensite:} As-quenched microstructure (i.e. martensite - \(415\pm5\) HV\(_{0.01N}\)) was subjected to tempering treatment (400\(^0\)C, 500\(^0\)C and 600\(^0\)C) and held for 2 hr. During this process, the highly dislocated martensitic structure underwent dislocation annihilation process (i.e. recovery) and the supersaturated martensitic laths decomposed into ferrite and cementite particles (Fig. 5.4). As a result, the hardness of tempered martensite decreased depending on the heat treatment condition (i.e. temperature and time, Fig. 5.5). In the current study, the hardness significantly decreased with an increase in the tempering temperature, though none of these conditions resulted in a hardness close to a desired value of \(350\) HV\(_{0.01N}\) (Fig. 5.5). Therefore, the tempering time was increased to 3 hr, resulting in a hardness of \(~357\pm3\) HV\(_{0.01N}\) at 500\(^0\)C tempering temperature.

Figure 5.4: (a) Optical and (b) scanning electron micrographs of tempered martensite microstructure in steel A tempered at 500\(^0\)C for 3 hours.
Figure 5.5: Hardness as a function of different tempering conditions in steel A.

c) Martensite: Steel B was reheated to 900°C and held for 5 min to obtain a fully austenitic microstructure followed by water-quenching. The resultant microstructure was fully martensitic consisting of laths with high dislocation density (Fig. 5.6). The hardness of the martensitic microstructure was 355±3 HV\textsubscript{0.01N}.

![Figure 5.6: (a) Optical and (b) scanning electron micrographs of martensite microstructure in steel B.](image)

d) Pearlite: Steel C at the as-received condition had a fully pearlitic microstructure consisting of ferrite and cementite lamellae (Fig. 5.7). The interlamellar spacing and thickness of the cementite were approximately 0.1 μm and 0.3-0.4 μm, respectively (Fig. 5.7). The hardness of pearlitic microstructure was 326±2 HV\textsubscript{0.01N}.
Figure 5.7: (a) Optical and (b) scanning electron micrographs of pearlite microstructure in steel C.

As aforementioned, four distinct microstructures (bainite, martensite, tempered martensite and pearlite with similar hardness (330-360 HV$_{0.01N}$) were successfully produced in three different alloy steels using a series of heat treatment approaches. The next step would be to compare the abrasive wear behaviour of these microstructures under the influence of the abrasive particles.

5.3.2 Two-body abrasive wear behaviour of the microstructures

a) Specific wear rate: The specific wear rate of the microstructures under a given abrasive condition (i.e. constant abrasive type and particle size, for example, SiC-240G) was studied (Fig. 5.8). The fully pearlitic microstructure displayed the lowest wear rate among all microstructures for the coarse silicon carbide paper corresponding to a ~58 µm particle size. If the relative wear rate of pearlite was defined as unity, then the relative wear rate of bainite, martensite and tempered martensite was found to be 1.36, 2.59 and 2.62, respectively. The pearlitic microstructure exhibited superior wear resistance in this condition, even though its hardness was relatively low (326 HV$_{0.01N}$) compared with the other microstructures (350-360 HV$_{0.01N}$). A similar trend was observed when the abrasive wear tests were conducted on silicon carbide grit papers with finer particle sizes of 600G and 1200G corresponding to sizes of ~25 µm and ~5 µm, respectively. The specific wear rates decreased with a reduction in the abrasive particle size for all microstructures (Fig. 5.8). However, the influence of abrasive particle size on the wear rate was less pronounced for the pearlitic structure compared with the others. As a result, the difference in the specific wear rate of the microstructures became less significant, revealing almost similar wear behaviour at the fine abrasive particle size of 1200G (~15 µm, Fig. 5.8).
Figure 5.8: Specific wear rate of distinct microstructures subjected to different SiC abrasive particle sizes.

b) Friction coefficient curves: The microstructure characteristics had a significant influence on the friction curve behaviour. In general, the curve consisted of an initial running-in period where the friction coefficient, $\mu$, was high, followed by a brief period during which it dropped down and later, either stabilized or increased continuously (Fig. 5.9). In the case of the pearlitic microstructure, the friction coefficient remained stable throughout the wear test with very few or negligible peaks in the friction curve. A similar trend was observed for the martensitic microstructure, although the number of peaks in the friction curve was significantly greater than for the pearlitic microstructure. After the running-in period, the coefficient of friction increased continuously for the tempered martensitic microstructure and its friction curve was characterised by numerous peaks or fluctuations (Fig. 5.9). The bainitic microstructure displayed a similar scenario of increasing friction coefficient although the rise in friction coefficient was not as high and the number of peaks was not as large as that of tempered martensite.
c) Surface topography of the abraded microstructures: Post wear analysis was carried out on the microstructures that were subjected to a coarse SiC of 240 G (~58 μm). A constant cross section (~716 μm×544 μm) of the abraded surface was topographically analysed for all microstructures. This was presented as a differential colour profile portraying the unique groove nature of each microstructure. Moreover, these groove features also characterized the amount of material removed (Fig. 5.10). The microstructure had a significant influence on the groove characteristics of the abraded surfaces. Pearlite and bainitic microstructures showed deep and narrow grooves (Figs. 5.10a-b), whereas, in the case of martensite and tempered martensite microstructures, the grooves were wide and shallow (Figs. 5.10c-d). The groove tracks in the pearlite microstructure were not uniform and continuous but were mostly terminated due to the pits (as shown by arrows in Fig. 5.10b). The bainitic microstructure revealed more continuous and deeper grooves (as shown by arrows in Fig. 5.10a). Wide and shallow grooves were characterized in both martensite and tempered martensite conditions, where heavy material loss was observed (as shown by arrows in Fig. 5.11d).
Figure 5.10: Topographical analysis of the abraded surfaces for different microstructures: a) bainite, b) pearlite, c) martensite and d) tempered martensite. Note: The scale is different for each microstructure.

d) Surface roughness and groove depth: The surface profile data were consistent with the above topographic images of the microstructures. The pearlitic microstructure showed the smoothest topography (surface roughness, $R_q = 519$ nm, Table 5.2) compared with other microstructures. In the case of bainite, the grooves were quite deep ($R_t = 4.42 \mu m$, Table 5.2) in comparison with pearlite. However, the average peak to valley height ($R_z = 2.07 \mu m$) was lowest in the pearlitic microstructure. This confirmed the relatively lower material loss and the presence of obstructions in pearlite (Fig. 5.10b). A higher surface roughness was observed in tempered martensite, where deep and wide valleys accentuated the surface profile, i.e. the maximum peak to valley depth, $R_t$ was very high (Table 5.2). Interestingly, there was a similarity in the wear rate of martensite and tempered martensite microstructures, despite the former ($R_a = 529$ nm) exhibiting a smoother profile than the latter ($R_a = 693$ nm, Table 5.2)
Table 5.2: Surface profile measurements of different microstructures.

<table>
<thead>
<tr>
<th>Microstructures</th>
<th>$R_a$</th>
<th>$R_q$</th>
<th>$R_z$</th>
<th>$R_t$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bainite</td>
<td>565</td>
<td>722</td>
<td>2.88</td>
<td>4.42</td>
</tr>
<tr>
<td>Pearlite</td>
<td>424</td>
<td>519</td>
<td>2.07</td>
<td>3.16</td>
</tr>
<tr>
<td>Martensite</td>
<td>529</td>
<td>659</td>
<td>2.46</td>
<td>3.25</td>
</tr>
<tr>
<td>Tempered martensite</td>
<td>693</td>
<td>968</td>
<td>4.12</td>
<td>6.62</td>
</tr>
</tbody>
</table>

The depth profile measurements for different microstructures provide a graphical representation of the peaks and valleys over a defined distance of ~ 636 µm. The valleys or the grooves in bainite were deeper than the pearlitic microstructure (as pointed by arrows in Figs. 5.11a and b). In the case of martensite and tempered martensite microstructures the valleys were wide and shallow (as pointed by arrows in Figs. 5.11c and d). These results are in accordance with the above topographical and surface profile studies (Fig. 5.11 and Table 5.2).

Figure 5.11: Profile of the abraded surface for different microstructures: a) bainite, b) pearlite, c) martensite and d) tempered martensite.
e) Wear track characterization: The groove characteristics of the microstructures were investigated at two different wear tests (i.e. normal abrasive wear test and single-track abrasive wear test, Figs. 5.12 and 5.13). SEM analysis of the wear tracks at both testing conditions was consistent with the earlier topographical results (Figs. 5.12 and 5.13). Wider grooves were found to be a characteristic feature of the martensitic microstructure (single-phase, Figs. 5.13a and b). The tempered martensite showed a similar trend although it consisted of martensitic laths with relatively low dislocation density and fine carbides formed during tempering (Figs. 5.13c and d). This suggests that the presence of carbides did not significantly influence the mechanism of material removal. Both microstructures displayed severe delamination at the edges of the grooves (Fig. 5.12a-d). However, the groove characteristics in multi-phase microstructures (i.e. bainite and pearlite) were largely dominated by the material displacement to the sides resulting in deep and narrow grooves (Figs. 5.12a-d). It is notable that the pearlitic microstructure experienced plastic deformation, realigning ferrite and cementite lamellae at the groove edges, which once again demonstrated a distinct material removal mechanism taking place in comparison with other microstructures (Figs. 5.12c and d).

![Figure 5.12: (a-c) Single and (b-d) multi-wear track analysis of different microstructures: Bainite (a and b) and pearlite (c and d).](image-url)
Figure 5.13: (a-c) Single and (b-d) multi-wear track analysis of different microstructures: Martensite (a and b) and tempered martensite (c and d).

f) Sub-surface characterisation of microstructures: Sub-surface characterisation of microstructures revealed that the layer below the abraded surface varied greatly in both properties and microstructural features from that of the bulk microstructure (Figs. 5.14 and 5.15). Multi-phase microstructures (pearlite and bainite) favoured plastic deformation or realignment of its constituents (i.e. ferritic laths and cementite lamellae) towards the abrasive sliding direction (Figs. 5.14a-d). On closer examination of the pearlitic microstructure, it was found that the cementite lamellae had been stacked together (i.e. plastically deformed) resulting in a mass of very fine sized microstructural constituents. The microstructural constituents were coalesced together making it hard to resolve or differentiate the phases (Figs. 5.14c and d). In addition, the groove edges of the pearlitic microstructure showed that ferrite and cementite lamellae realigned along the groove direction through severe plastic deformation (Figs. 5.14c and d). However, a featureless, white and non-etching layer was found below the worn surface of single-phase microstructures (martensite and tempered martensitic conditions). This could be due to the severe shear deformation that occurred during abrasion resulting in a highly dislocated white layer (Figs. 5.15a-d). Nevertheless, the extent of deformation varied for each microstructure, with pearlite
exhibiting the highest level of deformation, followed by bainite, martensite and tempered martensite (Figs. 5.14 and 5.15).

Figure 5.14: Sub-surface characteristics of microstructures: Bainite (a and b) and pearlite (c and d).

Figure 5.15: Sub-surface characteristics of microstructures: Martensite (a and b) and tempered martensite (c and d).
The abrasion led to an increase in the surface hardness for all microstructures (Fig. 5.16). However, the level of hardness increase on the deformed (worn out) surfaces was significantly affected by the microstructure characteristics (Fig. 5.16). Pearlite displayed the highest amount (i.e. 43%) of hardness increment, followed by bainite (i.e. 38%), tempered martensite (i.e. 6%) and martensite (i.e. 3%). It was interesting to note that these results were in accordance with the specific wear rate of the microstructures (Fig. 5.8). In general, multi-phase microstructures exhibited a relatively higher degree of hardening in comparison with the single-phase microstructures.

Figure 5.16: Micro-hardness of the microstructures and the amount of hardening after the wear test.

5.4 Discussion
In the current study, microstructures with similar bulk hardness levels displayed a distinctive two-body abrasive wear behaviour. This was supported by their unique sub-surface and single wear track investigations. Moreover, the amount of frictional heat generated on the microstructure (i.e. pin surface) is quite negligible. Considering, the total duration of each test (~ 25 mins) and the heat dissipation with the surroundings, there will be little frictional heat affecting the microstructure surface. To confirm this, a thermocouple was spot welded close to the pin surface for measuring the temperature gradient (i.e. pin surface and the surrounding temperature) during the pin-on-disc wear
test. The results revealed that the temperature gradient was not more than \( \sim 4^\circ C \). Therefore, the effect of frictional heat on microstructure was not considered in the current study.

5.4.1 Abrasive wear resistance of multi-phase microstructures (bainite and pearlite)

In a sliding abrasive system, the ability of a microstructure to actively absorb the frictional energy determines its abrasion resistance. This is largely governed by the characteristics of the microstructural constituents [15, 22, 23]. The present study showed the impact of microstructures with a similar hardness level (330-360 HV\(_{0.01N}\)) on the abrasive wear behaviour (Fig. 5.8). Despite similar hardness levels, each microstructure displays a unique response towards abrasive wear behaviour, with respect to parameters such as specific wear rate, friction curve and wear track characteristics (Figs. 5.8, 5.9, 5.12 and 5.13). It is interesting to note that the pearlitic microstructure that had a relatively low hardness level (326 HV\(_{0.01N}\)) exhibited superior abrasion resistance compared with the other microstructures (Fig. 5.8). This indicates that the metallurgical structures play a vital role in the wear resistance of ferrous alloys.

In general, microstructures consisting of brittle and ductile phases, i.e. multi-phase microstructures are more efficient in resisting abrasion than single-phase microstructures [173]. The current observations reveal that multi-phase microstructures such as pearlite (ferrite and cementite lamellae) and bainite (bainitic ferrite and retained austenite) exhibit better abrasion resistance compared to the examined single-phase microstructures (Fig. 5.8). This is in good agreement with observations reported elsewhere [25]. A brittle or hard phase offers more resistance towards the ‘penetration’ action of the abrasive particles; meanwhile the inclination towards ‘cracking or failure’ is largely reduced by the presence of a ductile phase [62]. In other words, multiphase microstructures are more efficient in combating abrasion as the ductile phase imparts support to the load bearing brittle phase. This theory is validated by the dominant abrasion resistance of pearlite and bainite microstructures when compared with the other microstructures consisting of a single phase.

During sliding abrasion, heavy deformation is induced in the sub-surface regions (i.e. layer beneath the wearing surface) leading to significant microstructural
changes [174]. The properties of the sub-surface layers may vary markedly from that of the bulk microstructure [25, 175, 176]. The initial microstructure constituents influence the extent and the characteristics of sub-surface layers (Figs. 5.15 and 5.16). The severely deformed region (i.e. sub-surface layer) appears either by realignment of microstructure constituents (e.g. pearlite and bainite, Figs. 5.14a-d) and/or through the formation of a featureless white layer (e.g. martensite and tempered martensite, Figs. 5.15a-d). The amount of hardness increment on the deformed surface reveals the extent of work hardening during abrasion (Fig. 5.16). Furthermore, it also denotes the superior ability of the multi-phase microstructures to accommodate high stresses that are involved during abrasion.

The realignment of microstructural constituents towards the sliding direction is largely attributed to the ductile ferrite phase in pearlite microstructure. On closer observation, the cementite lamellae were bent and stacked together, demonstrating the major role of the ductile ferrite phase in plastic realignment of lamellae during the abrasion process (Figs. 5.14d). The plastic realignment in the bainitic microstructure was not as significant as that of pearlite and this explains the relatively high material loss and specific wear rate in bainite. This is evident in the single-wear track tests, where the groove features and material displacement modes are distinctive for single and multi-phase microstructures (Figs. 5.12 and 5.13).

When the abrasive particles indent the metallic surface, wear tracks or grooves are formed on the surface irrespective of the type of material displacement mode. Three abrasive wear modes namely ploughing, wedge formation and cutting are primarily involved in the process of abrasion [62]. Ploughing and wedge formation modes result in the plastic deformation of the material, i.e. the material is displaced to the groove sides without causing any direct material removal. On the other hand, cutting leads to severe material loss through continuous or discontinuous debris detachment [177, 178]. There are distinct groove characteristics observed for different microstructures (Figs. 5.12 and 5.13). Microstructural constituents play a significant role on the material removal during abrasive wear. Consequently, the deep and narrow grooves of the multiphase microstructures (bainite and pearlite) reveal the ploughing mechanism with displaced material as ridges along the grooves (Figs. 5.12a and c). Furthermore, the formation of wedges strengthens the argument of the ploughing mechanism in pearlitic microstructure. When the hard SiC abrasive particles penetrate
deep into pearlite and bainite microstructures, the ductile phases (ferrite and/or retained austenite) facilitate the material displacement or realignment of constituents towards the sliding direction. Subsequently, as the wear progresses, very little material is lost as debris through the cutting mechanism. The hard cementite lamellae have significant resistance towards grooving by plastic deformation. The lamellae are stacked together enabling less material detachment and a smoother topography amongst all of the microstructures studied (Fig. 5.14d and Table 5.2).

The literature suggests that not all abrasive particles are involved in the process of groove formation. Some particles participate in changing the surface roughness, whereas, others result in the material removal [163]. Surface topography of the multiphase microstructures reveal significant differences in their surface profile (Rq, Rt and Rz parameters as explained earlier) and flow of material removal (Fig. 5.10 and Table 5.2). The presence of pits in the pearlitic grooves obstructs the material removal process, thereby leading to the termination of grooves at different points (Figs. 5.10b and 5.12c), as reported elsewhere [39]. As explained earlier, these obstructions could be attributed to the hard cementite lamellae that have significantly reduced the material loss leading to narrow grooves and a relatively smooth topography (Table 5.2). Conversely, very few obstructions in bainitic grooves indicate their continuous material removal process. During the abrasive wear test, the retained austenite in the bainitic microstructure is largely transformed into martensite (i.e. TRIP effect), as the austenite peaks were significantly reduced after wear test (Fig. 5.17). This ultimately results in a hardness increase after the abrasive wear test (Fig. 5.16). This work hardening behaviour of bainite potentially improves the wear resistance in comparison with martensite and tempered martensite.
During the abrasive wear test, the asperities of the particles fracture lead to the accumulation of metallic debris (i.e. a mixture of fractured abrasive and metallic particles) on the wear tracks during abrasion [11, 161]. In the present study, the rest of the wear regime is largely unique for each microstructure except for the running-in period. The distinctive friction curve for each microstructure is largely influenced by the amount and rate of material loss (Fig. 5.9). In a typical friction curve, the fresh abrasive asperities come in contact with the metallic surface during the initial running-in period, explaining the higher friction coefficient. As the wear progresses, the abrasive particles tend to lose their cutting or abrading efficiency leading to the stabilization of wear or the steady state condition [63, 179]. For bainite, there is a rise in the friction coefficient indicating continuous material removal and higher abrasive wear than pearlite (Fig. 5.9). In pearlite, the friction coefficient is almost constant leading to negligible material loss during this steady state period (Fig. 5.9). However, in the case of pearlite, the near constant friction coefficient is due to the fact that the abrading efficiency of the SiC particles reduces progressively with an increase in sliding distance.

The above arguments based on the current results describe the superior abrasion resistance of the multi-phase microstructures. However, a better understanding of the characteristics of single-phase microstructures would substantiate this.
5.4.2 Abrasive wear resistance of single-phase microstructures (martensite and tempered martensite)

The high strain levels produced during sliding abrasion makes the single-phase (mostly brittle) microstructures more vulnerable, as there is very little plastic deformation leading to Hertzian fracture on the abrading surface [180]. This was evident in martensite and tempered martensitic microstructures that exhibited higher abrasive wear rates and similar material removal mechanisms (Figs. 5.8, 5.13a-d). The microstructures tend to attain a steady-state condition during abrasion. As a result, abrasion induces microstructural changes (i.e. hardening) in the sub-surface layers that differ from that of the bulk microstructure. For instance, the sub-surface layers of martensite and tempered martensite were considerably different from that of multi-phase microstructures (Figs. 5.13c and d).

It is notable that plastic deformation of constituents was observed in the sub-surface of the multi-phase microstructures. Heavy deformation and strain levels during sliding abrasion resulted in a highly dislocated region closer to the abrasive surface (Fig. 5.15). Consequently, these layers do not etch and are featureless due to very fine constituents or sub-structures (Figs. 5.15b and d), as reported elsewhere [24, 175, 176]. It must be noted that the ductile ferrite grains in the tempered martensite accommodate high stress leading to a relatively well-defined white featureless layer compared with martensite (Figs. 5.15b and d). However, the thickness of the deformed layers remains similar for the martensite and tempered martensitic conditions that explains their comparable material loss (Figs. 5.8, 5.15c and d). Nevertheless, their hardness increments were different (Fig. 5.16), which can be due to the presence of less dislocations in the tempered martensite in comparison with the martensitic microstructure.

Nevertheless, the single-phase microstructures displayed similar groove features, i.e. wide and shallow (Figs. 5.13a and c). The material removal process is dominated by a ‘cutting’ wear mode that has resulted in the formation of wear debris [181]. The debris, in the form of continuous or discontinuous strips is removed (i.e. delamination) from the grooves through a low-cycle fatigue mechanism [177, 180]. Strips of fragmented material attached to the groove ridges portray the cutting mechanism in both martensite and tempered martensite (Fig. 5.18). However, the
material removal rate in each of these conditions was distinctive during various stages of wear regime, i.e. tempered martensite had a continuous material removal rate throughout the wearing process. This is evident in their friction curves, unlike martensite, where the friction coefficient is almost constant after the running-in period (Fig. 5.9). In other words, the material removal process in martensite is either stabilized or the wear track is filled with wear debris, which is evident through the numerous peaks in their friction curve (Fig. 5.9).

![Figure 5.18: Delamination of fragmented material: a) martensite and tempered martensite.](image)

The surface profiles of their abraded surfaces also revealed significant differences (Fig. 5.11 and Table 5.2). Interestingly, the surface roughness of the former ($R_a = 529$ nm) is smoother than the latter ($R_a = 693$ nm). This can be explained by comparing the surface irregularities of martensite and tempered martensitic microstructures. Martensite has more deep and wide valleys than that of tempered martensite (as indicated by arrows in Figs. 5.11c and d). In other words, there are large amounts of fragmentation at certain regions (Fig. 5.10d and Table 5.2), leading to higher material loss due to the brittle natured martensite laths. Thereby, favouring the cutting mode in active material removal process during abrasion (Fig. 5.10c). Furthermore, $R_a$ represented the mean value of surface irregularities [48], which in turn neglects the irregularities that are not in the range. This presumably explains the similar abrasion resistance of martensite and tempered martensitic microstructures.

The current observations suggest that multi-phase microstructures are better equipped to combat abrasive conditions than the single-phase microstructures. The schematic representation of the microstructural conditions under the action of an
abrasive particle helps to further explain this (Fig. 5.19). In general, brittle single-phase microstructures are prone to higher material displacement due to the homogeneity of their constituents. When an abrasive particle penetrates the microstructure (Fig. 5.19b), they offer very little resistance and are more susceptible to cracks. Thereby, leading to excessive material loss through wider grooves (Figs. 5.19a-c). Conversely, a microstructure matrix with a combination of brittle and ductile phases can offer relatively better resistance (Figs. 5.19d-f). The brittle phases can resist the penetration action of the abrasive grain, meanwhile the ductile phase provides a dampening effect for the load bearing brittle phase (Fig. 5.19e). Moreover, it suppresses the effect of failure to a greater extent leading to narrower grooves than the single-phase microstructures (Fig. 5.19f). In other words, the phenomenon of plastically realigning the microstructural constituents is feasible in multi-phase microstructures. Such syndicate effort of these microstructures makes them an attractive alternative under high-stress abrasive conditions.

Figure 5.19: Schematic representation of single (a–c) and multi-phase microstructures (d-f) under the action of an abrasive particle. Grey and white regions representing brittle and ductile phases.

5.5 Summary

The two-body abrasive wear tests were conducted on four distinct microstructures, namely bainite, pearlite, martensite and tempered martensite having a similar hardness level (330-360 HV$_{0.01N}$). The results revealed that the microstructure had a significant
effect on the specific wear rate and groove characteristics. The following is a summary of the conclusions drawn:

1. The characteristics of microstructural constituents were influential in displaying a unique abrasion behaviour for each microstructure. However, multi-phase microstructures exhibited superior abrasion resistance to single-phase microstructures.

2. The abrasive wear resistance of the microstructures was greatly influenced by its sub-surface deformations, which was a direct measure of its metallurgical structure. The sub-surface analyses were crucial in determining their abrasive response, i.e. realignment of microstructural constituents (pearlite and bainite) and/or formation of white layer (martensite and tempered martensite).

3. The distinct material removal processes of the microstructures were attributed towards their microstructure matrix (phases). In the case of multi-phase microstructures, there was a simultaneous action of ploughing and wedge formation mechanisms leading to narrow and deep wear tracks. In single-phase microstructures, the cutting mode was responsible for wide and shallow wear tracks.

4. A strong correlation exists between the work hardening behaviour and the abrasive wear resistance of the microstructure.
Chapter 6

Impact of microstructural constituents on the two-body abrasive wear behaviour of ultra-high strength bainitic steels

6.1 Introduction

An extensive study on the commonly used steels in abrasive environment has revealed that multi-phase microstructures exhibit superior abrasion resistance. The ability of these multi-phase microstructures to offer high work-hardening behaviour makes them highly suitable for high-stress abrasive conditions [13, 24, 29, 76]. Moreover, this behaviour was largely attributed towards the synergetic action of the individual phases. It is, therefore, expected that the microstructure constituent characteristics such as size, morphology, composition and volume fraction of the phases affect the abrasion behaviour [35, 36]. Microstructures with a combination of hard and soft phases can impart plastic deformation, which is considered to be highly beneficial in abrasion [173, 182, 183]. For example, the presence of martensite and ferrite can improve the abrasion in dual-phase steels. Here, the martensite characteristics (i.e. carbon content and volume fraction) determines the abrasion resistance, as this property gradually
deteriorates beyond a given martensite volume fraction depending on the steel composition. This is due to the brittle nature of the martensite phase offering relatively less fracture toughness and little resistance to the high-stress levels associated with the abrasion [55, 184]. On the other hand, bainitic phase can be a good candidate to replace brittle martensite, as the bainite offers a wide range of mechanical properties in terms of tensile strength, hardness and fracture toughness [37]. Meanwhile, the outcome of Chapter 4, i.e. effect of microstructures with similar hardness levels on the two-body abrasion has shown that bainitic microstructures can display better abrasion properties.

Conventional bainitic steels are often multi-phase (i.e. a combination of ferrite, granular bainite/lower bainite, martensite and retained austenite phases) [37]. Hence, it is difficult to investigate the abrasive wear behaviour of this class of steel due to the collective response of different microstructural phases. In addition, advanced bainitic steels mostly contain retained austenite phase, which may undergo martensitic transformation on abrasion (so called TRIP phenomenon) [37, 124, 139, 185]. However, it is not clear how freshly formed martensite from the retained austenite (i.e. TRIP effect) contributes to the abrasion behaviour. The most recent development in advanced high strength steels led to the design of a new class of TRIP steel consisting of very fine bainitic ferritic lath and retained austenite, known as nanobainitic structured steel. This class of steel contains a relatively high carbon content and alloying elements that offers superior mechanical properties (e.g. yield strength of 2 GPa) [40, 41] and promise in tribological applications [38, 42, 124, 186]. A recent study has shown that the nanobainitic steels have higher abrasive wear resistance compared with other microstructures (fully martensite and pearlite microstructures) under three-body abrasive wear conditions. This behaviour was attributed to the work hardening behaviour of the retained austenite embedded in the microstructure [39]. However, there is still a lack of understanding on how the characteristics of retained austenite influence the abrasive wear behaviour in a composite bainitic ferrite plus retained austenite microstructure.

The ultimate aim of the current chapter was to investigate the role of retained austenite characteristics (size, morphology, volume fraction and carbon content) on the abrasive wear behaviour of nanobainitic steels. Therefore, a wide range of fully bainitic microstructures consisting of bainitic ferrite and retained austenite with distinct characteristics were produced at different isothermal bainitic transformation
temperatures, i.e. 200-350°C in a high carbon high alloyed steel. These microstructures were then subjected to two-body abrasive wear tests. The specific wear resistance of each microstructure was correlated to the topographic analysis of their abraded surfaces and the abrasion induced microstructural changes in their sub-surface regions. Single-wear track analysis was also performed to study the mechanism of material displacement in the microstructures.

6.2 Experimental procedure

6.2.1 Materials

The chemical composition of the steel used in the current study was 0.79 %C, 1.5 %Si, 1.98 %Mn, 0.98 %Cr, 0.24 %Mo, 1.06 %Al and 1.58 %Co (in wt. %). The steel composition ensures that it falls within the nanobainitic steel classification. The as-received billet was initially hot-rolled at a temperature range of 1050-1100°C to reduce the thickness from 40 mm to ~15 mm. The material was then homogenized at a temperature of 1200°C for 24 hr in an argon gas atmosphere. Afterwards, the steel was subjected to a series of heat treatments to achieve fully bainitic microstructures with distinct characteristics. The samples were initially austenitized at 900°C for 30 min followed by austempering in a salt bath furnace at different temperatures of 350°C, 300°C, 250°C and 200°C for a holding time of 1 day, 2 days, 5 days and 10 days, respectively. These heat treatment schedules resulted in a fully bainitic microstructure consisting of bainitic ferrite lath and retained with different characteristics (size and morphology). One of the homogenized specimens was furnace cooled from 900°C to room temperature to obtain a fully pearlitic microstructure.

6.2.2 Characterization techniques

Struers, Dura Scan micro-hardness machine was employed to carry out the hardness measurement at 0.01N with a dwell time of 15 s. An average of ten measurements was taken into account for each heat treatment condition. After the wear tests, microhardness measurements were performed on the worn surface. The tests were carried out meticulously to ensure that the indentation lies approximately in the middle of the wear groove. To reduce the error percentage, a minimum of 15 hardness tests were performed for each microstructure condition. The samples for scanning electron microscopy (SEM) were prepared using standard polishing procedure and lightly etched in a 4 vol. % nital solution. The microstructural characterization was performed using SEM, SUPRA 55VP microscope operated at 20 kV with a SE2 detector.
X-ray diffraction was extensively used to measure the volume fraction of retained austenite in the fully bainitic microstructures, prior and after the wear test at different conditions. To reduce the residual stress and eliminate the phase transition during the sample preparation, the as-received steel samples were subjected to a chemical treatment containing a solution of 80% hydrogen peroxide, 5% hydrofluoric acid and 15% water. The worn out surfaces subjected to wear were cleaned only in ethanol and the XRD characterization was performed without any chemical treatment. X-ray diffraction was employed using a Philips PW 1130 diffractometer with graphite monochromated CuK$_\alpha$ radiation at 40 kV and 30 mA in the 2$\theta$ range of 40-100° at a rate of 0.02° per 7 sec. Direct comparison method was used to compare the integrated intensities of (200)$_\gamma$, (200)$_\alpha$, (220)$_\gamma$ and (220)$_\alpha$, thereby measuring the volume fraction of retained austenite in the microstructure. The carbon content of the retained austenite before and after the wear tests were measured from the lattice parameter of (220)$_\gamma$ peak of diffraction pattern, using Dyson and Holmes equation, $a_\gamma$ (Å) = 3.578 + 0.033*$C_\gamma$ (in wt. %), where $a_\gamma$ – lattice parameter (Å) and $C_\gamma$ – carbon content of austenite (in wt. %) [187].

The microstructural constituents were further characterised by transmission electron microscopy (TEM), using a Philips CM-20 microscope operated at 200 kV. TEM foils were prepared by mechanically grinding 3 mm Ø discs to a thickness of 70 µm, followed by twin-jet polishing using a solution of 5% perchloric acid in methanol at -20°C at an operating voltage of 50 kV. The dislocation density, $\rho$, in the bainitic ferrite was calculated using the formula, $\rho$=2$N_L$/Lt, where, $N_L$, is the number of intersections with dislocations, $L$, is the length of random lines and $t$ is the foil thickness [188].

The topographic analysis of the abraded pin surface was investigated using an Alicona-Infinite Focus, optical profilometer. Scanning the abraded pin surfaces rendered three-dimensional images revealing the depth profile of the abraded surfaces. The images were further analysed using modular software interfaced with a PC. The characteristics of the surface irregularities (peaks and valleys) were characterized based on parameters such as arithmetic and geometric average roughness (R$_a$ and R$_q$, also known as root mean square or RMS). In addition, the height of the irregularities over a defined distance was measured using R$_t$ and R$_z$ [48]. This approach enabled the
surface roughness and the groove characteristics of the fully bainite microstructures to be determined after the abrasive wear tests.

6.2.3 Two-body abrasive wear tests
Abrasive wear behaviour was studied using a CSM high temperature tribometer compliant to ASTM G99 standards (as discussed in Chapter 3, section 3.4.1). Two-body abrasive wear was simulated by subjecting a pin sample (~6 mm Ø and 60 mm long) on an alumina (P320) abrasive disc. The pin tip was chamfered to 45°, as the pin holder was inclined at 45° to the abrasive disc. This angular orientation of the pin ensured a constant mechanics (i.e. constant cross sectional area of the wearing surface) throughout the test. The abrasive wear test was conducted in an unlubricated condition for a sliding distance of 150,000 mm with a constant speed of 20 mm/s and a load of 9 N to minimise any adiabatic heating during the test. The pin samples were ultrasonically cleaned in ethanol and weighed before and after each wear test. At least four tests were performed for each testing condition and an average specific wear rate was calculated based on the weight loss of the pin sample.

6.2.4 Single-track wear tests
To examine the effect of microstructural characteristics on the material removal mechanism, a single wear track test was performed in a controlled test condition. Here, the pin with a polished surface was subjected to a minimal traversal (i.e. sliding distance of 20 mm at a sliding speed of 20 mm/s, which was less than one full travel). Therefore, the wear track was not overrun by more than one particle. The wear track was then characterised using electron microscopy to observe the interaction between the abrasive particles and the microstructural constituents.

6.3 Results
By subjecting, the high carbon-high alloy steel to isothermal bainitic transformation at a temperature range of 200-350°C, resulted in fully bainitic microstructures. The microstructures consisted of bainitic ferrite and retained austenite with distinct characteristics (size, volume fraction, carbon content and morphology). The impact of retained austenite characteristics and the role of TRIP behaviour in the two-body abrasive wear will be discussed in detail below.

6.3.1 Microstructural characterization
The characteristics of the microstructural constituents of the fully bainitic microstructures were largely dependent on the austempering conditions (i.e. isothermal transformation temperature). The bainitic microstructure became finer with a decrease in the transformation temperature (Figs. 6.1 and 6.2). For instance, the lath thickness was reduced from 300±100 nm for FB-350°C to 60±10 nm for FB-200°C. Also, retained austenite thickness was reduced from 70±30 nm for FB-350 to 30±5 nm for FB-200. Furthermore, the volume fraction of bainitic ferrite increased with a decrease in the transformation temperature. The dislocation density of the bainitic ferrite laths gradually increased with a decrease in the transformation temperature, (i.e. $2 \times 10^{15}$ for FB-350 to $4.7 \times 10^{15}$ for FB-200), which was also evident in the microstructural hardness increment (Fig. 6.3a). Similarly, the characteristics of retained austenite (volume fraction and thickness of retained austenite) were altered with the decrease in the transformation temperature (Figs. 6.1 and 6.2). At 350°C, both film and blocky retained austenite morphologies were observed in the microstructure (Figs. 6.1d and e). With a decrease in the transformation temperature, the retained austenite became finer, with mostly film morphology at a temperature of 250°C and below. In general, the volume fraction of retained austenite decreased with a decrease in the transformation temperature (54% for FB-350 to 16% for FB-200). Interestingly, the volume fraction and film thickness of retained austenite was largely similar for the fully bainitic microstructures (i.e. FB) transformed at 200°C (i.e. ~16% and ~30±5 nm) and 250°C (~17% and ~30±10 nm). The current heat treatment resulted in the formation of nano size (<100 nm) bainitic microstructures (i.e. nanobainite) at a transformation temperature less than 250°C. The bainitic microstructures became relatively coarse with an increase in the isothermal transformation temperature (i.e. 300°C and 350°C). It is important to note that our previous investigation revealed the presence of carbides in both bainitic ferrite and retained austenite in this class of steel despite the existence of 1.5 %Si (in wt. %) in the steel composition [189].
Figure 6.1: (a-e) TEM images of the fully bainitic microstructures at different isothermal temperatures: a) FB-200, b) FB-250, c) FB-300, and (d-e) FB-350. f) SEM image of fully pearlitic microstructure. RA and BF represent retained austenite and bainitic ferrite, respectively.

Figure 6.2: SEM images of the fully bainitic microstructures at different isothermal temperatures: a) FB-200, b) FB-250, c) FB-300 and (d) FB-350.
6.3.2 Two-body abrasive wear behaviour of the fully bainitic microstructures

a) Specific wear rate and frictional coefficient characteristics: The specific wear rate of the fully bainitic microstructures with different constituent characteristics (i.e. bainitic ferrite and retained austenite) displayed a unique response towards abrasion (Fig. 6.3b). Both, FB-200°C and FB-250°C displayed superior abrasion among the fully bainitic microstructures when subjected to an alumina abrasive environment. There was a negligible difference in the specific wear rate of FB-200°C \((5.9\pm0.4) \times 10^{-4} \text{ mm}^3/\text{N.m}\) and FB-250°C \((7.1\pm0.6) \times 10^{-4} \text{ mm}^3/\text{N.m}\) conditions. Conversely, a significant amount of material loss was associated with the FB-300°C \((12.1\pm0.8) \times 10^{-4} \text{ mm}^3/\text{N.m}\) and FB-350°C conditions \((15.6\pm0.6) \times 10^{-4} \text{ mm}^3/\text{N.m}\). In general, the wear behaviour was directly related to the hardness of material (Figs. 6.3a and b).
A significant fluctuation was also observed in the friction coefficient curves for all microstructures. The curve consisted of an initial running-in period, where the friction coefficient was high followed by a sudden drop. After the running-in period, there was an increase in the friction coefficient and the behaviour of friction curve changed for different fully bainitic microstructures (Fig. 6.4). For microstructures transformed at 300°C and 350°C, the friction coefficient curve, on average, showed a gradual increase with sliding distance (Figs. 6.4c and d). However, the friction coefficient revealed steady-state behaviour at low transformation temperatures of 200°C and 250°C (Figs. 6.4a and b). These results were in agreement with the specific wear rate of the microstructures, where the FB-350°C condition displayed significantly higher specific wear rate compared with FB-200°C (Fig. 6.3b).
Figure 6.4: Friction coefficient curve of microstructures: a) FB-200, b) FB-250, c) FB-300 and d) FB-350.

b) Surface topography of the abraded microstructures: Post wear analysis conducted on a uniform cross section (~716 µm × ~544 µm) of the abraded surfaces revealed distinctive groove characteristics for each fully bainitic microstructure (Fig. 6.5). The groove features largely characterised the quantum of material removal by their differential colour profile corresponding to a scale bar. The groove characteristics of the fully bainitic microstructures were in accordance with their specific wear rate, i.e. wide and shallow/deep and narrow. There was a transition in the groove features (i.e. wide and shallow to deep and narrow) based on the amount of material loss. In the case of FB-350°C, the grooves were relatively wide and shallow, where heavy material loss was observed (Fig. 6.5d). Moreover, the grooves were not continuous due to the broken ridges (as shown by arrows, Fig. 6.5d). A similar scenario appeared in FB-300°C (Fig. 6.5c), although it was less severe than FB-350°C. FB-200°C and FB-250°C microstructures were characterized with continuous, deep and narrow grooves (Fig. 6.5a and b). It must be noted that the abraded surfaces of FB-250°C also displayed regions of significant material loss through wide and shallow grooves (as shown by arrows, Fig. 6.5b). Thus, the FB-250°C microstructure revealed a combination of both wide and deep grooves.
The surface profile data was consistent with the groove characteristics observed in the topographic images (Table 6.1). The mean and maximum peak to valley height ($R_z$ and $R_t$) was largely similar for FB-350°C and FB-300°C microstructures. Conversely, FB-200°C displayed a relatively higher $R_t$ (15.48 µm) and $R_q$ (1.95 µm) among the fully bainitic microstructures (Table 6.1). Moreover, the surface profile characteristics ($R_a$, $R_q$, $R_z$ and $R_t$) of FB-250°C were intermediate between the FB-300°C and FB-200°C microstructural conditions. A graphical representation of the surface profile was presented to depict the peaks and valleys in the fully bainitic microstructures (Fig. 6.6). In general, wide valleys or grooves (shown by dashed ovals in Fig. 6.6d) accounted for the significant material loss observed in FB-350°C and FB-300°C. On the other hand, FB-200°C and FB-250°C presented deep and narrow valleys (shown by dashed ovals in Figs. 6.6a and b). In summary, the numerical data and graphical representation of the surface profile of the abraded microstructures were consistent.
Table 6.1: Surface profile measurements of the fully bainitic microstructures

<table>
<thead>
<tr>
<th>Microstructures</th>
<th>$R_a$ ($\mu m$)</th>
<th>$R_q$ ($\mu m$)</th>
<th>$R_z$ ($\mu m$)</th>
<th>$R_t$ ($\mu m$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FB-200</td>
<td>1.42</td>
<td>1.95</td>
<td>9</td>
<td>15.48</td>
</tr>
<tr>
<td>FB-250</td>
<td>1.29</td>
<td>1.6</td>
<td>7.8</td>
<td>11.22</td>
</tr>
<tr>
<td>FB-300</td>
<td>1</td>
<td>1.28</td>
<td>6.1</td>
<td>9.65</td>
</tr>
<tr>
<td>FB-350</td>
<td>1.16</td>
<td>1.5</td>
<td>6.5</td>
<td>10.16</td>
</tr>
</tbody>
</table>

Figure 6.6: Surface profile of the abraded surfaces of the fully bainitic microstructures with similar constituents: a) FB-200, b) FB-250, c) FB-300 and d) FB-350.

c) Single-wear track characterization: Single-wear track analysis through SEM investigations presented further evidence to the nature and extent of material removal during abrasion (Fig. 6.7). The influence of the characteristics of microstructural constituents was clearly seen in the groove characteristics transition (i.e. from wide to narrow) as the transformation temperature decreased (Fig. 6.7). In FB-350°C, the grooves were wide, with delamination at their edges, indicating a significant material removal (Fig. 6.7d). The width of the grooves decreased as the microstructural constituents were refined (i.e. lower transformation temperature, Fig. 6.7). Moreover,
delamination was less dominant in the fully bainitic microstructures transformed at temperatures of 200°C and 250°C (Figs. 6.7a and b). In other words, nanobainitic microstructures with fine bainitic ferrite lath and retained austenite film thickness of <100 nm displayed lesser material removal. At this temperature regime, the material was mostly displaced towards the edge of the grooves with a negligible material detachment, leading to a reduced material loss.

Figure 6.7: Single-wear track analysis of the fully bainitic microstructures with similar constituents: a) FB-200, b) FB-250, c) FB-300 and d) FB-350.

d) Sub-surface characterisation of microstructures after abrasive wear tests: Significant morphological (microstructural features) changes were observed in the layer beneath the abraded surface (sub-surface, Fig. 6.8). This layer was in contrast to that of the bulk microstructure for all heat treated conditions (Fig. 6.8). In general, the sub-surface layers revealed severe deformation accompanied by the realignment of bainitic ferrite lath and retained austenite towards the sliding direction (Fig. 6.8). However, the extent of deformation layer (i.e. thickness) was distinct and varied for each fully bainitic microstructure. Especially, in the case of FB-350°C (Figs. 6.8h), the realignment of microstructural constituents was less significant compared with other fully bainitic microstructures (Figs. 6.8b, d and f). The deformation layer was significantly enhanced with a decrease in the transformation temperature (i.e.
microstructure refinement), mostly revealing featureless and white non-etching layers (Figs. 6.8b and d).

Figure 6.8: Sub-surface characteristics of the abraded fully bainitic microstructures subjected to abrasive wear: (a-b) FB-200, (c-d) FB-250, (e-f) FB-300 and (g-h) FB-350.

This was consistent with the hardness and XRD measurement of the worn sub-surface at different heat treatment conditions (Table 6.2 and Fig. 6.9). In general, the hardness of the abraded surface increased after the wear test (Fig. 6.9b). However, the extent of the hardness increment was significantly influenced by the initial microstructure characteristics (bainitic ferrite lath and retained austenite). FB-350°C displayed the highest amount of hardness increment (i.e. 68%) followed by FB-300°C (49%), FB-250°C (33%) and FB-200°C (33%). The XRD measurement also showed that the retained austenite transformation took place in the abraded surface for all
conditions (Fig. 6.9a). However, the amount of retained austenite transformation was significantly higher for the microstructures transformed at higher temperatures (i.e. FB-300°C and FB-350°C, Table 6.2). Meanwhile, it was interesting to find that FB-250°C and FB-200°C (i.e. nanobainitic microstructures) exhibiting a similar amount of retained austenite transformation (i.e. ~2%, Table 6.2) and hardness increment (~33%, Fig. 6.9b).

Table 6.2: Volume fraction of RA transformation (TRIP effect) and carbon content prior and after abrasion.

<table>
<thead>
<tr>
<th>Microstructures</th>
<th>Vol. fraction of RA (in %)</th>
<th>Carbon content of RA (in wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before wear test</td>
<td>After wear test</td>
</tr>
<tr>
<td>FB-200</td>
<td>16</td>
<td>14</td>
</tr>
<tr>
<td>FB-250</td>
<td>17</td>
<td>15</td>
</tr>
<tr>
<td>FB-300</td>
<td>51</td>
<td>19</td>
</tr>
<tr>
<td>FB-350</td>
<td>54</td>
<td>24</td>
</tr>
</tbody>
</table>
Figure 6.9: a) X-ray diffraction analysis and b) micro hardness of the microstructures prior and after abrasion.

6.4 Discussion

In the current study, fully bainitic microstructures consisting of bainitic ferrite and retained distinct microstructure with distinct characteristics (i.e. size, morphology and volume fraction) greatly influences the two-body abrasive wear behaviour of fully bainitic microstructures. This is largely evident in the differential abrasive wear resistance and the subsequent subsurface changes in the fully bainitic microstructures.

6.4.1. Effect of isothermal transformation temperature on bainitic microstructure constituents

In general, an increase in the bainitic transformation temperature leads to relatively coarse microstructural constituents (bainitic ferrite and retained austenite, Figs. 6.1 and 6.2), which in part can contribute to a higher material loss during abrasion (Fig. 6.3b). The characteristics of fully bainitic microstructures are largely dependent on the isothermal bainitic transformation temperatures and their holding times. If the isothermal treatment is ceased before the completion of bainitic transformation at a given isothermal temperature, then the remaining austenite would have transformed to martensite [37]. Therefore, the wear behaviour of fully bainitic microstructures formed after the completion of bainitic transformation at different isothermal holding temperatures was studied in the current investigation. However, the volume fraction
of retained austenite is enhanced with an increase in the transformation temperature due to the negative slope of $T_o$ (i.e. the temperature at which the free energy of bainitic ferrite equals with that of austenite during bainitic transformation) [40-42]. The phase transformation temperature also reveals a significant change in morphology and carbon content of the retained austenite. For instance, the FB-300°C and FB-350°C microstructures consist of both film and blocky retained austenite morphologies; although the film retained austenite is dominant at low transformation temperatures (i.e. nanobainitic microstructures, FB-200°C and FB-250°C). In general, the average carbon content of retained austenite reduces with an increase in the bainitic transformation temperature (Table 6.2).

6.4.2. Impact of retained austenite morphology and TRIP effect on abrasive wear behaviour of fully bainitic microstructures

The fully bainitic microstructures heat-treated at lower transformation temperatures (i.e. nanobainitic microstructures, FB-200°C and FB-250°C) display superior abrasion resistance compared with the bainitic microstructures transformed at a higher temperature range (FB-300°C and FB-350°C). In addition, there is a linear relationship between the hardness and the abrasion resistance of the bainitic microstructures (Fig. 6.3). Despite hardness being one of the key parameters that influence abrasive wear, other factors need further investigation. The distinctive morphologies of retained austenite can influence the abrasive wear behaviour through the formation of new martensite via. the strain-induced transformation phenomenon (i.e. TRIP effect) during abrasion [39]. The TRIP effect is evident in the XRD results, where the amount of retained austenite is significantly reduced after the wear test for all transformation temperatures (Fig. 6.9a and Table 6.2). This diverse behaviour is a result of distinct differences in the characteristics of retained austenite in the initial microstructure. Moreover, the XRD results of the worn surface show that the average carbon content of untransformed retained austenite is relatively higher than that of initial average carbon content of a given bainitic microstructure (Table 6.2). This suggests that the martensitic transformation mainly takes place in the retained austenite with less mechanical stability (e.g. lower carbon content). This is evident in high transformation temperature conditions (i.e. FB-300°C and FB-350°C), where the average carbon content of retained austenite is relatively less compared with the low bainitic transformation temperature (nanobainitic microstructures, Table 6.2). In
addition, the retained austenite has blocky and film morphologies in the high transformation temperatures (FB-300°C and FB-350°C, Figs. 6.1 and 6.2). The coarse blocky retained austenite mostly has a relatively low stability and can transform into martensite at a lower strain level than the thin film morphology that are mostly observed at low transformation temperatures (FB-200°C and FB-250°C, Figs. 6.1 and 6.2). Consequently, the volume fraction of fresh martensite formed during abrasion is reduced significantly with a decrease in the phase transformation temperature (Table 6.2).

The extent of TRIP phenomenon appears to have a significant effect on the wear behaviour of fully bainitic microstructures. The blocky morphology and increased volume fraction of retained austenite are more prone to local strain concentration and enhanced martensitic formation on straining [39, 190]. This greatly contributes to the substantial hardness increment (FB-300°C and FB-350°C, more than 49%, Fig. 6.9b) on the abraded surfaces compared with other low temperature bainitic microstructures (FB-200°C and FB-250°C, ~33%, Fig. 6.9b). The contribution of fresh martensite on the wear behaviour is expected to be influenced by its composition (i.e. carbon equivalent). In general, the toughness of the martensite decreases with an increase in the carbon content, resulting in lower wear resistance [55, 184]. However, the nanobainitic microstructures (FB-200°C and FB-250°C) reveal superior abrasion properties, despite the presence of higher carbon content retained austenite compared with high temperature bainite (FB-300°C and FB-350°C, Fig. 6.3b). In other words, the martensite formed through TRIP phenomenon at 200°C, is expected to be more brittle than that of 350°C. This can be partly explained due to the differences in the characteristics of retained austenite (morphology and size) formed in the fully bainitic microstructures during abrasion, which ultimately restricts the size and morphology of fresh martensite.

Meanwhile, the poor abrasive performance of FB-350 microstructure is mostly attributed to the bainitic ferrite and the retained austenite characteristics. The former is relatively coarse (300±100 nm) with less dislocation density \((2\times10^{15})\) in FB-350 condition compared with bainitic microstructures transformed at lower temperatures. The blocky retained austenite was more prone to martensitic formation during abrasion than the relatively stable film retained austenite. The coarse fresh martensitic regions formed from the blocky retained austenite would be more vulnerable to crack initiation
and propagation, due to its size irregularity compared with the fresh martensite formed from the thin film retained austenite. In addition, the coarse bainitic ferrite with relatively less dislocation density in FB-350 microstructure is vulnerable to abrasion contributing further towards a higher material loss. Consequently, the volume fraction of martensite formed at high bainitic transformation temperatures (~30% at 350°C) is much greater than that of low temperature bainite (~2% at 200°C, Table 6.2). Consequently, a significant material loss is observed in high temperature bainitic microstructures (FB-300°C and FB-350°C, Fig. 6.3b).

The high material loss experienced in FB-300 and FB-350 microstructures is evident through their friction curve characteristics, which registers a continual rise in frictional coefficient after the initial running-in period (Figs. 6.4c and d). This could be attributed towards the continual material removal process during abrasion. During the start of the two-body abrasive wear, i.e. initial running-in period, the metallic pin comes in contact with fresh abrasive particles, as a result there is a rise in the coefficient of friction. As wear test progresses, the abrasive particles tend to lose their cutting efficiency resulting in a more steady loss of material or steady state condition [11, 16, 17, 25, 171]. During this period, it is more likely that the metallic pin has some debris attached to its surface due to its continuous traversal in the same wear track. This attached debris when comes in contact with new or partly damaged particles (or asperities) can result in fluctuations (i.e. localized rise and drop in coefficient of friction). These fluctuations could be ascribed to the slow sliding speed (20 mm/s) of the pin resulting in a greater interaction (more signals) between the wearing surface and the wear debris (Fig. 6.4). In this context, FB-350 microstructure demonstrated a significant amount of fluctuation due to an increased debris accumulation on the wear track.

Continual material loss in FB-300°C and FB-350°C microstructures is evident through the wide (marked by dotted ovals in Figs. 6.6c and d) and shallow grooves. During abrasion, the abrasive particles indent into the abrading metallic surface (i.e. pin), leading to the formation of either wear tracks (grooves) or multiple scratches to affect the surface profile. The surface profile of an abraded surface is usually characterized by peaks (i.e. section of the profile in the positive direction from the mean line) and valleys (i.e. section of the profile in the negative direction from the mean line) [48]. The relatively low peak to valley height ($R_z$ and $R_T$ values, Table 6.1)
confirms that the grooves are not continuous due to the broken ridges. The scenario of broken ridges (as shown by arrows in Fig. 6.5d) is more prevalent in bainitic microstructures formed at high temperatures (e.g. FB-350°C). This could imply that the displaced material (debris) is detached from the grooves (as shown by arrows in Figs. 6.7c and d). The process of debris delamination has led to a significantly high material loss in FB-300°C and FB-350°C microstructures.

A combined study on the topographic analysis and single-wear track analysis explains the mechanism of material removal in FB-200°C and FB-250°C nanobainitic microstructures (Figs. 6.5 and 6.7). The deep continuous grooves with a comparatively narrow width reveal that the majority of the abraded material is displaced towards the groove sides without losing them as wear debris (Figs. 6.7a and b). This process of material displacement to the side/edge of the grooves is known as ploughing/wedge formation. It is important to understand that these processes involve very little or negligible material loss [62, 177, 178]. The constant abrading action affects the surface roughness of the abraded metallic surface (Fig. 6.6 and Table 6.1). The formation of deep and narrow grooves in FB-200°C and FB-250°C nanobainitic microstructures results in an increase in their geometric surface roughness ($R_q$) and peak to valley height ($R_z$ and $R_t$, Table 6.2). The friction coefficient of FB-250 and FB-200 is, on average, fairly constant after the initial running-in period for most part of the wear regime (i.e. sliding distance, Figs. 6.4a and b). The steady state behaviour of the friction curve is an indication of less material loss in these microstructures (Figs. 6.4a and b). Despite FB-200 microstructure revealing superior abrasive resistance their coefficient of friction is relatively high when compared with other microstructures (Fig. 6.4a). This can be explained by the fact that the microstructure matrix of FB-200 consisting of very fine bainitic ferrite and film retained austenite was highly efficient in breaking down the abrasive particles during the initial stage of abrasion (i.e. running in period). Thereby, the particles were mostly reduced to wear debris with very little metallic flakes in it. During subsequent travels, there are very little particles for the metallic surface to involve in the abrading action. As a result, there is a transition in the wear mode, i.e. abrasive to adhesive, where the pin mostly comes in contact with a metallic wear debris and no abrasive particles. This metal-metal contact resulted in an increase in the coefficient of friction. Such scenarios are mostly prevalent in cases where there is almost complete deterioration of abrasive particles. The phenomenon of abrasive particle deterioration has been explained in detail in Chapter 4.
Although, the above discussion provides crucial information on the amount of material loss, the sub-surface analysis sheds more light on the abrasion response of the microstructural constituents. The sub-surface realignment of layers of all fully bainitic microstructures reveals the plastic deformation of its microstructural constituents (bainitic ferrite and retained austenite, Fig. 6.8). In general, the sliding abrasion involves high strain levels at the abrading surface, leading to significant deformation on the sub-surface layers of the microstructure [25, 175, 176]. These layers are marked by microstructural changes, which differs strikingly from the bulk microstructure (Fig. 6.8). However, the extent of microstructural realignment in FB-200°C and FB-250°C nanobainitic microstructures (Figs. 6.8a-d) is significant when compared with FB-350°C and FB-300°C microstructures (Figs. 6.8e-h). The realignment of ferritic laths strongly depends on the stability of the adjacent retained austenite. The thin film retained austenite with high mechanical stability can be considered as a relatively ductile phase in the fully bainitic microstructure as it requires more strain to undergo martensitic transformation (i.e. TRIP effect). As a result, the thin film retained austenite would be expected to offer support to the load bearing adjacent bainitic ferrite laths and reduce the risk of crack or failure (Fig. 6.8) [24, 62]. However, the low stability of retained austenite at high transformation temperatures (FB-350°C and FB-300°C) leads to early onset of martensitic transformation, resulting in a limited realignment of adjacent bainitic ferritic laths (Figs. 6.8f and h).

The current study suggests that the characteristics of retained austenite play a significant role on the abrasion wear behaviour of fully bainitic microstructures formed in a high carbon high alloying content steel at different transformation temperatures. However, it appears that the presence of retained austenite in this type of microstructure is not always beneficial for the two-body abrasive wear. A fully pearlitic microstructure was produced from the same chemical composition through furnace cooling of the steel alloy after austenitization at 900°C. The steel with fully pearlitic structure (Fig. 6.1e) was then subjected to a similar abrasive wear testing environment employed for other bainitic microstructures. Despite a relatively low hardness, the fully pearlitic microstructure (335 HV0.01N, Fig. 6.3a) displayed better abrasion wear resistance (Fig. 6.3b) than FB-350°C (413 HV0.01N). The sub-surface analysis of pearlitic microstructure reveals a thicker deformed layer suggesting a better work-hardening behaviour (Figs. 6.10a and b), despite having a low hardness.
increment (12%) than FB-350°C (~68%, Fig. 6.10b). This suggests that the presence of retained austenite with low stability and the resulting martensite with brittle nature may have a detrimental effect in the current abrasive condition.

Figure 6.10: Sub-surface characteristics of the abraded pearlitic microstructure subjected to two-body abrasive wear.

The current findings are not consistent with earlier work [39], where the presence of retained austenite in this class of bainitic microstructure appears to be beneficial in three-body abrasive condition, offering high work-hardening behaviour. In addition, it reveals superior abrasion resistance to both fully pearlitic and martensitic steels. This could be due to different abrasive environment (i.e. three-body versus two-body abrasion) and/or distinct retained austenite characteristics (i.e. size, morphology and composition) in [139, 191] compared with the current study.

6.5 Summary

The two-body abrasive wear resistance was studied in fully bainitic microstructures formed in a high carbon high alloy steel at different transformation temperatures. The following is a summary of the conclusions drawn:

1. The fully bainitic microstructures largely consisted of bainitic ferritic lath and retained austenite. The bainitic phase transformation significantly influenced the characteristics of microstructural constituents (size, morphology, carbon content and volume fraction). The retained austenite morphology was changed from film to film+blocky with an increase in the transformation temperature.
2. The two-body abrasive wear resistance was greatly enhanced with a decrease in the transformation temperature due to the refinement of microstructure constituents (i.e. greater hardness) and the extent of retained austenite TRIP phenomenon.
3. The distinctive morphologies of retained austenite greatly influenced the TRIP effect, leading a differential abrasive wear resistance. The presence of coarse and blocky retained austenite significantly reduced their mechanical stability, leading to an early onset of TRIP effect. Meanwhile, film retained austenite displayed a relatively high mechanical stability, thereby delaying the TRIP effect.

4. In general, the TRIP effect often resulted in the formation of fresh martensite with different morphologies, depending on the characteristics of retained austenite. The coarse martensite formed from blocky retained austenite is more vulnerable to crack initiation and propagation, due to its irregular morphology, compared with the fresh martensite formed from the thin film retained austenite (nanobainitic microstructures).

5. A comparative study on the abrasion resistance of high temperature bainite (i.e. FB-350) and fully pearlitic microstructure revealed a detrimental effect of the less mechanically stable retained austenite (blocky) on the two-body abrasion.
Chapter 7

Investigation on the abrasive wear behaviour of ferrous microstructures with similar hardness levels using a scratch-test method

7.1 Introduction

The effect of microstructure constituents in the two-body abrasive wear was clearly highlighted in Chapter 5. One of the key findings was that the microstructure undergoes evolution, i.e. changes in morphology and mechanical properties during the process of abrasion. The sub-surface layer of the microstructure often experiences severe deformation leading to an increase in their hardness [12, 13, 24, 163]. This implies that the microstructure is quite dynamic and their abrasive resistance cannot be bulk hardness dependent. Moreover, the abrasive behaviour of multi-phase microstructures is based on a number of factors such as volume fraction, morphology and carbon equivalent of their metallurgical phases [35, 36]. Nevertheless, the process of material removal during abrasion is largely quantified by the characteristics of the abrasive environment.

In the case of two-body abrasive wear, the abrasive particle characteristics, i.e. particle size, geometry and density play a vital role as they are predominantly involved in the material removal process [11, 16-18, 192]. Especially, in the case of the current pin-on-disc experiment (i.e. two-body abrasive wear), the continuous deterioration of the abrasive particles often raises a series of arguments over their abrasive efficiency. Meanwhile, in an actual abrasive conditions (e.g. digging and excavating operations) the material is often subjected to a series of constant abrading action by fresh particles [6]. This eventually necessitates more emphasis on simulating a laboratory abrasive wear test that can impart more control over its parameters and offer better
reproducibility. To tackle this issue, a high strain abrasive scratch testing could be one of the potential laboratory tests that can aid in a better understanding of the abrasion process [181, 193].

Consequently, the current study focussed on the abrasive behaviour of four distinct microstructures with similar hardness levels (as discussed in Chapter 5) when subjected to a high strain abrasive scratch test. During the test, a robust indenter abrades the microstructure surface under the action of a normal load. The grooves made during the scratch test were characterized using electron microscopy and an optical profilometer. Sub-surface layers (i.e. region beneath the grooves) were carefully examined to understand the abrasive response of the microstructures. One of the salient features of the current study is that, microstructures with similar hardness levels would be greatly beneficial in understanding the impact of microstructure constituents on the material removal mechanisms.

7.2 Experimental procedure

Four distinct microstructures namely bainite, pearlite, martensite and tempered martensite produced in Chapter 5 were extensively used in this study. Struers, Dura Scan micro-hardness machine was employed to carry out the hardness measurement on the heat-treated microstructures (i.e. prior to the scratch tests) at 0.01 N with a dwell time of 15 s. After the wear tests, the micro-hardness measurements were carried out along the sub-surface layer at a distance of ~3 µm below the sample edge. Six hardness measurements were taken for each condition and an average was taken into account. Scanning electron microscope (SEM, SUPRA 55 VP FEG operated with SE2 detector at 20 kV) was extensively used for microstructure and scratch characterization. The volume fraction of retained austenite in bainitic microstructure was measured using direct comparison method in an X-ray diffraction technique. Prior to XRD measurement, bainitic samples were chemically treated (as explained in 5.2 in Chapter 5) to reduce the phase transition during sample preparation techniques.

The abrasive resistance and the process of material removal in different microstructures were evaluated using an in-house abrasive scratch test instrument. This instrument was designed to create a controlled scratch on the surface of interest (~45 mm × 55 mm × 7 mm) using a robust indenter. The indenter was conical in shape with a tip radius of ~820 µm and was made of tungsten carbide and cobalt. The scratches were performed on the microstructure surface at a constant sliding speed
(1 mm/s), sliding distance (30 mm). Five different normal loads, namely 200 N, 500 N, 1000 N, 1500 N and 2000 N. However, the bainite and pearlite microstructures were subjected to a load range of 200 N-1500 N. The tip of the indenter was cleaned after each pass to avoid debris attachment. The instrument was interfaced with a PC, which enables ease of control over load and displacement of the indenter. The grooves made during the scratch test and their sub-surface (i.e. layer beneath the scratch) characteristics were studied using scanning electron microscopy. TEM foils of size ~6×8 µm² were prepared from the deformed regions at the sub-surface layers (Fig. 7.1a) through Focused Ion Beam (FIB) technique using FEI Quanta 3D FEG FIB-SEM. This technique involved a series of precise steps namely, identifying the region of interest, platinum deposition, bulk-out, U-cut, lift-out, mounting, thinning and cleaning. The prepared foils were further investigated in a high-performance Transmission Electron Microscope (JEOL JEM-2100F) fitted with a NanoMEGAS ASTAR automated crystal orientation and phase mapping. The microscope operates at 200kV coupled with a Gatan Orius SC1000 fast-rate acquisition high-resolution camera of 11 Mpixel. The data was exported to the HKL Technology/Oxford instruments Channel 5 for post processing.

Alicona optical profilometer was also employed to perform three dimensional scans on the surface profile of the grooves. Extensive scans, i.e. 4 sections (of ~6 mm long) in each scratch (~ 30 mm long) was analysed to ensure repeatability. Subsequent analysis was carried out using a modular software that was interfaced with a PC. The volume of the material removal, \( V_{rem} \), during the scratch test for a given condition was calculated based on the equation, \( V_{rem} = [V_g - (V_1 + V_2)] \), where, \( V_g \) is the volume of the groove (mm³), \( V_1 \) and \( V_2 \) are the volume of the built-up edges (mm³) as shown schematically in Figure 7.1b. In addition, the degree of penetration, \( D_p \), was determined based on the equation, \( D_p = 2d/w \), where, \( d \) and \( w \) are the depth and width of the groove (µm) [27] (Fig. 7.1). The surface roughness of the grooves was quantified based on the characteristics of the peaks and valleys along the sliding direction [48]. Average arithmetic roughness, \( R_a \), of the groove profile was calculated over a defined length (i.e. ~14.5 mm) and an average was reported for all conditions.
Figure 7.1: Schematics showing a) TEM foil region beneath the scratch and b) cross section of a scratch with material displaced to the sides as built-up edges.

7.3 Results

7.3.1 Microstructural characterization

Four distinct microstructures with similar hardness levels were produced through distinct heat treatment schedules. The bainitic microstructure formed in steel A consisted of bainitic ferrite and 11.5% of retained austenite with a hardness of 363±1 HV$_{0.01N}$ (Fig. 7.2c). A fully martensitic microstructure with highly dislocated laths were formed in steel B having a hardness level of 355±3 HV$_{0.01N}$ (Fig. 7.2a). By tempering the fully martensitic phase formed in steel A, the dislocation density of martensitic laths was reduced through the annihilation process (i.e. recovery, Fig. 7.2b) and fine cementite particles were also formed, resulting in a hardness level of 350±2 HV$_{0.01N}$. Steel C was used in the as-received condition with a fully pearlitic microstructure consisting of ferrite and cementite lamellae with a hardness level of 326±2 HV$_{0.01N}$ (Fig. 7.2d).
7.3.2 Scratch tests

The volume of material removal, $V_{rem}$, and the degree of penetration, $D_p$, were studied as a function of the applied normal load during a scratch test (Fig. 7.3). In general, the volume of material removal was directly proportional to the normal load for all microstructures (Fig. 7.3a). Martensite displayed the highest amount of material removal, followed by tempered martensite across different loads. However, bainite and pearlite exhibited a similar amount of material removal at all load conditions. It was interesting to note that at 1500 N, the volume of material removal of bainite and pearlite became comparable to that of tempered martensite. The degree of penetration as a function of normal load mostly revealed a sigmoidal behaviour for all microstructures, except the martensite where there was a sudden increase in the depth of penetration at low normal load followed by a linear rise beyond 500 N (Fig. 7.3b). However, the increase in the degree of penetration was not proportional across other microstructures. In the case of tempered martensite, the degree of penetration increased from $\sim 0.085$ at 500 N to $\sim 0.275$ at 2000 N. Meanwhile, the pearlite microstructure experienced a higher degree of penetration across all loads (i.e. 200 N to 1500 N) compared with the bainite microstructure.
The volume of material removal, $V_{rem}$, was enhanced with an increase in the degree of penetration, $D_p$. Interestingly, all microstructures in general, displayed a similar behaviour up to a $D_p$ of 0.2, beyond which a sharp change in the material removal was observed for the martensitic microstructure for a given degree of penetration, $D_p$ (Fig. 7.4). This clearly indicates that there is a slight correlation between the degree of penetration, $D_p$, and the volume of material being lost during a scratch test independent of initial microstructure (Figs. 7.2-7.4).
7.3.3 Groove profile characterization

The groove profile was characterized by conducting optical profilometer scans on the microstructures subjected to scratch tests at different loads (Fig. 7.5). A comparative groove curve of the scratch tests plotted as a function of the normal load revealing distinct groove characteristics (Figs. 7.5). In general, the material displacement to the sides (built-up edges) was reduced significantly with an increase in the normal load for all microstructures. However, the built-up volume was significantly reduced beyond 1500 N in the case of martensite and tempered martensite (as shown by arrow, Fig. 7.5a). Moreover, there was a non-uniformity in the built-up volume across the scratch edges. For instance, the built-up volume on the left was substantially less than the built-up edge on the right in bainite at 1000 N (as shown by arrows in Fig. 7.5a). This trend was more dominant with an increase in the normal load, i.e. > 500 N. Furthermore, the martensite and tempered martensite mostly revealed wider and deeper grooves compared with bainite and pearlite for a given load condition. On the other hand, the groove profile of pearlite (1000 N and 1500 N) and bainite (1500 N) displayed more peaks and valleys.

Figure 7.4: Volume of material removal, $V_{rem}$ in different microstructures as a function of depth of penetration, $D_p$. 
Figure 7.5: A comparative groove characteristics of different microstructures as a function of the normal load: a) Martensite, b) tempered martensite, c) bainite and d) pearlite.

The average arithmetic surface roughness, $R_a$, was calculated along the groove (Fig. 7.6) for different microstructures across the load regime. Overall, the average surface roughness of the groove profile augmented with an increase in the normal load (Fig. 7.7). In the case of martensite and tempered martensite, they displayed a significantly high $R_a$. It is important to note that there was a steep increase in the average surface roughness, $R_a$, for martensite at 500 N, followed by a gradual rise with the load. However, in the case of tempered martensite, the steep rise was observed up to a load of 1000 N, beyond which the curve began to attain a near steady state. Meanwhile, the bainite and pearlite microstructures revealed a gradual surface roughness increase from $\sim 2.5 \, \mu m$ at 200 N to $\sim 5 \, \mu m$ at 1000 N and then suddenly enhanced to 15 $\mu m$ at 1500 N. The transition surface roughness behaviour (shown by dash circle in Fig. 7.7a) closely correlated to the groove surface profile for a given microstructure. There was a clear difference in the surface profile characteristics of all microstructures with respect to the depth ($\mu m$) (as shown in, Figs. 7.7a-c). Especially, in the case of martensite at 500 N, there was a prominent display of peaks and valleys in comparison with other microstructures. Furthermore, the groove profile characteristics (peaks and valleys) grew wide and deep at these critical loads (i.e. transition surface roughness, Figs. 7.7a-c).
Figure 7.6: Schematic representation of an optical profilometer scan direction.

Figure 7.7: Average surface roughness, $R_a$, of the scratch track in different microstructures as a function load. Comparison of the groove profiles along the
sliding direction at the transition loads: (A) martensite (200 N-500 N), (B) tempered martensite (500 N-1000 N) and (C) bainite (1000 N-1500 N).

7.3.4 Groove morphology and material displacement
The characteristics of the scratch tracks (i.e. grooves) were significantly influenced by the initial microstructure for a given normal load (Figs. 7.8-7.11). In general, the width of the scratches (grooves) in the microstructures increased progressively with a rise in the normal load, i.e. from 200 N to 2000 N. Moreover, the width of the grooves in martensite microstructure was comparatively higher than tempered martensite for a given normal load (Figs. 7.8a-c and 7.9a-c). For instance, the width of the grooves in martensitic microstructure increased progressively from ~350 µm at 200 N to ~830 µm at 2000 N, whereas it was ~225 µm at 300 N to ~660 µm at 2000 N in the case of tempered martensite (Figs. 7.8a-c and 7.9a-c). However, martensite and tempered martensite microstructures displayed similar groove profile characteristics with respect to the crack initiation and propagation. At 1000 N, cracks were observed perpendicular to the groove (as shown by arrows in Figs. 7.8c and 7.9b). The extent of crack propagation was significantly enhanced at 2000 N along with the material displacement (as shown by arrows in Figs. 7.8-7.9).

This scenario was also evident through the topographical analysis (i.e. a differential colour profile) in martensite and tempered martensite microstructures, where the depth profile was measured at different normal loads (Figs. 7.8d-f and 7.9d-f). These three-dimensional images also demonstrated that the amount of material removal was proportional with the applied normal load. This was largely evident with an increase in the scratch depth (Figs. 7.8d-f and 7.9d-f) and width (Figs. 7.8a-c and 7.9a-c). The crack propagation and material displacement were observed through the formation of step in the groove profile (as shown by arrows in Figs. 7.8e-f and 7.9e-f). However, in case tempered martensite, this step formation was significantly less and mostly observed at 2000 N (as shown by arrows in Figs. 7.9e-f) compared with grooves formed in martensite microstructure (Figs. 7.8e-f).
Meanwhile, the groove characteristics of bainite and pearlite displayed distinct differences in their groove characteristics compared with martensite and tempered martensite. For instance, in the case of bainite and pearlite, the phenomenon of crack propagation was observed at a much higher load, i.e. 1500 N, whereas numerous cracks and material displacement scenario were observed at 1000 N in martensite and tempered martensite (Figs. 7.8a-c, 7.9a-c, 7.10a-c and 7.11a-c). However, the groove characteristics remained largely similar for bainite and pearlite microstructures. The width of the grooves increased from ~150 µm at 200 N to ~600 µm at 1500 N
Furthermore, the occurrence of minor-cracks was dominant at 1000 N, followed by extensive crack propagation and material displacement at 1500 N (Figs. 7.10c and 7.11c). Also, it was interesting to note that the groove width of tempered martensite at 2000 N (Fig. 7.9c) was comparable with the groove width of bainite and pearlite at 1500 N (Figs. 7.10c and 7.11c). Nevertheless, the average depth of the tempered martensite groove at 2000 N was relatively higher (~500 µm, Figs. 7.9c) than bainite, and pearlite (~350 µm, Figs. 7.10f and 7.11f.)

Figure 7.10: (a-c) SEM and (d-f) topographical analysis of the scratch tracks in bainite microstructure at different load conditions: (a and d) 200 N, (b and e) 1000 N and (c and f) 1500 N.

Figure 7.11: (a-c) SEM and (d-f) topographical analysis of the scratch tracks in pearlite microstructure at different load conditions: (a and d) 200 N, (b and e) 1000 N and (c and f) 1500 N.
7.3.5 Sub-surface characteristics

Sub-surface (i.e. layer beneath the groove) investigations displayed substantial deformation in comparison with the bulk microstructure (Figs. 7.12-7.14). The thickness of the subsurface layer showed an increased dependence on the normal load subjected during the scratch test and the initial microstructure (Fig. 7.15a). In general, the tempered martensite and bainite displayed the highest and lowest amount of deformation (i.e. average thickness of the deformed layer) amongst all microstructures at their corresponding normal loads (as shown by dashed lines, Figs. 7.12-7.14). Also, in the case of tempered martensite, there was a sudden increase in the sub-surface layer thickness at 1500 N, ~22 µm at 1000 N to ~53 µm at 1500 N (Figs. 7.13d and 7.15a). Conversely, the thickness of the sub-surface layers in martensite, bainite and pearlite increased proportionately with the normal load in the case of martensite (Figs. 7.12-7.15). Moreover, bainite microstructure revealed comparatively lesser deformation at low loads, 200 N and 500 N. (Figs. 7.14a-c and 7.15a). Interestingly, pearlite and martensite exhibited a similar sub-surface layer thickness until 1000 N (Fig. 7.15a). Furthermore, the sub-surface layers of bainite and pearlite at 1500 N, displayed signs of detachment from their bulk microstructure (as shown by arrows, Figs. 7.14c and f). The extent of deformation was reflected in the hardness of the sub-surface layers, which increased substantially in comparison with the bulk microstructure hardness after the scratch tests (Fig. 7.15b). Overall, there was a progressive increase in the sub-surface layer hardness with respect to the normal load. The bainite displayed the highest amount of hardness increment followed by pearlite, tempered martensite and martensite microstructures for a given normal load. In the case of martensite, the amount of hardness increment was significantly less when compared with other microstructures.

Quite severe porosity/cavities (i.e. black circles, Fig. 7.12e) appeared in the sub-surface layer of martensite. TEM characterization of the martensite sub-surface layer revealed rows of elongated fine grains, which were coalesced together (Fig. 7.16). Consequently, it was difficult to measure the size of the grains, as it was hard to differentiate between them. However, these rows of elongated grains were approximately 0.1 µm in thickness. This suggested that the sub-surface layer was subjected to severe plastic deformation. Meanwhile, bainite displayed a highly deformed sub-surface layer that could be hardly resolved, along with the several cracks
propagating perpendicular to the groove direction (Fig. 7.14c). Contrastingly, the sub-
surface layer of pearlite revealed a coalescence of its microstructural constituents (i.e.
an amalgamation of ferrite and cementite regions, Figs. 7.14e and f) making it harder
to resolve. Furthermore, the extensive TEM characterization of pearlite subsurface
layer exhibited complex morphological changes in the microstructure, which consisted
of fragmentation of cementite layers (i.e. region B in Fig. 7.17) and dissolution of
cementite in ferrite with high dislocation density (i.e. region A in Fig. 7.17). In fact,
the sub-surface layer experienced a severe plastic deformation.

Figure 7.12: Sub-surface characteristics of martensite microstructure subjected to
scratch tests at different load conditions: a) 200 N, b) 500 N, c) 1000 N d) 1500 N
e) highly deformed region from 1500 N and f) 2000 N.
Figure 7.13: Sub-surface characteristics of tempered martensite microstructure subjected to scratch tests at different load conditions: a) 200 N, b) 500 N, c) 1000 N, d) 1500 N, e) 2000 N and f) highly deformed region from 2000 N.

Figure 7.14: Sub-surface characteristics of (a-c) bainite and (d-f) pearlite microstructures subjected to scratch tests at different load conditions: (a and d) 200 N, (b and e) 1000 N and (c-f) 1500 N.
Figure 7.15: a) Sub-surface layer thickness and b) hardness of different microstructures subjected to scratch tests as a function of normal load.
Figure 7.16: a) TEM and b) EBSD characterization of the martensite sub-surface layer subjected to a scratch test at 2000 N.

Figure 7.17: TEM characterization of the pearlite sub-surface layer subjected to a scratch test at 1500 N. A: Cementite dissolution in a highly dislocated ferrite, B: cementite fragmentation and C – bulk microstructure.
7.4 Discussion

The current study demonstrates the impact of microstructures in a controlled high-stress abrasive scratch testing. Despite similar hardness levels (330-370 HV\textsubscript{0.01N}), the microstructures reveal a distinct behaviour towards the scratch testing. In other words, the groove characteristics (i.e. $V_{rem}$, $D_p$, $R_a$) and sub-surface deformation layer are significantly influenced by the microstructure (Figs. 7.3, 7.5, 7.7-7.17). For instance, pearlite displayed a similar material removal, $V_{rem}$, compared with bainite, though the pearlite hardness (326 HV\textsubscript{0.01N}), is relatively lower than bainite (363 HV\textsubscript{0.01N}, Fig. 7.3a). Meanwhile, martensite (355 HV\textsubscript{0.01N}) and tempered martensite (350 HV\textsubscript{0.01N}) experienced significant volume of material removal, $V_{rem}$, compared with bainite and pearlite in the current study (Fig. 7.3a). This clearly indicates that the characteristics of microstructures play a vital role in the process of material removal during the abrasive scratch testing.

The current findings demonstrate that the single-phase microstructures (martensite and tempered martensite) exhibit higher material loss compared with multi-phase microstructures i.e. bainite (bainitic ferrite and retained austenite) and pearlite (ferrite and cementite lamellae). This could be attributed to the combined action of brittle and ductile phases leading to a better abrasion response in a multi-phase microstructure. On the other hand, a single-phase microstructure offers very little resistance against the abrasion due to their brittle nature of the phase constituent [24, 25, 28, 29, 76]. This scenario is largely evident through the unique groove characteristics of the microstructures (Figs. 7.8-7.11). In the case of martensite and tempered martensite, the average width of the grooves increases progressively with a rise in the normal load (250 μm at 200 N to 600 μm at 2000 N, Figs. 7.8-7.9). This could be attributed to the basic underlying logic of continual rise in the contact pressure, $P$, (Pressure= Force/Area) of the indenter on the microstructure surface as a result of a progressive increase in the normal load. However, in the current study the normal load, i.e. force is varied, but the contact area remains constant. The same ideology has been explained in [193], where smaller indenter size (50 μm) results in a higher wear rate compared with large indenter size (200 μm). Moreover, with an increase in the normal load and contact pressure, micro-cracks began to appear in the groove at 500 N (Fig. 7.18a), followed by the crack propagation and material displacement at higher loads (2000 N, Figs. 7.8-7.9).
During an abrasive scratch test, the indenter that is being subjected to a normal load traverses along the microstructure surface resulting in a groove. Moreover, the width of the grooves enhances with an increase in the normal load. Subsequently, this leads to an increase in the degree of penetration, $D_p$, in the case of martensite and tempered martensite (Fig. 7.3b). However, the augmentation in the $D_p$ is not uniform across the normal loads for different initial microstructures (as shown by arrows in Fig. 7.4), despite constant indenter size and the sliding speed. The brittle nature of the martensitic laths and cementite particles in tempered martensite could have resulted in this significant rise in the degree of penetration, $D_p$, compared with bainite and pearlite for a given normal load. It is interesting to find that, there is a limited correspondence among the degree of penetration, $D_p$, the volume of material removal, $V_{rem}$, and the normal load among different microstructures (Fig. 7.4). This is due to the fact that the degree of penetration, $D_p$, is a mathematical ratio (i.e. twice the depth of the groove to the width of the groove) [27]. As a result, a similar degree of penetration, $D_p$, can lead to a range of contrastingly different material removals. This can explain the unpredictable trend in the degree of penetration, $D_p$, versus the volume of material removal, $V_{rem}$, for similar normal loads across different microstructures (Fig. 7.4). As
expected, a simple and straightforward relation is observed between the degree of penetration, \( D_p \), and the volume of material removal, \( V_{rem} \), for all microstructures (Fig. 7.4). These results to some extent are in agreement with earlier work [30], where the curve plotted between degree of penetration, \( D_p \), and degree of wear having a same relationship.

The current findings exhibit that the displaced material from the grooves is not uniform, in the case of martensite and tempered martensite. As the indenter traverses on the microstructure surface, the material removed from the groove is displaced to their sides known as built-up edges (Fig. 7.1). At lower loads (200 N and 500 N), there is a significant amount of material built-up on both the sides of the groove, in the case of martensite (Figs. 7.5a and 7.18 d). The built-up material attached to the groove sides is known as ploughing mechanism [177, 178]. This phenomenon becomes less dominant with an increase in the normal load above 500 N (i.e. very little material built-up, Fig. 7.5a). In other words, the material is lost as wear debris, which is known as cutting [177, 178]. The brittle nature of martensitic phase facilitates the material displacement through step like formation (Fig. 7.8c and f), but this behaviour is quite negligible in the case of tempered martensite (Fig. 7.9). Furthermore, there is a sudden increase in the built-up volume in martensite, which could be due to a combination of both metallurgical characteristics and loading conditions (as shown by arrow in Fig. 7.5a and b). The low ductility of martensite is more susceptible to crack initiation and propagation, resulting in an irregular displacement of material to the sides at high loads, i.e. 1500 N, as reported elsewhere [194]. Contrarily, at low loads (200 N and 500 N), the material built-up is fairly similar on both sides of the groove in martensite and tempered martensite. This could be attributed to the comparatively low strain associated at these loads, which can greatly affect the flow of the material [194, 195]. These observations suggest that there could be a possible transition in the material removal mechanism with respect to the normal load and the metallurgical structures.

The transition from ploughing to cutting mechanism across the load regime is evident through the average surface roughness of the groove profile in martensite and tempered martensite microstructures (as shown by dotted boxes, Fig. 7.7). With an increase in the normal load, the volume of material being displaced as built-up edge increases. However, the mechanical properties of the displaced material (deformed and brittle nature) makes them more prone to detach as wear debris. As a result, when there
is a higher material removal, an increase in the average surface roughness, $R_a$, of the groove profile is observed (Fig. 7.7). The augmentation of the surface profile, i.e. peaks and valleys over the transition region (i.e. martensite: 200 N to 500 N and tempered martensite: 500 N to 1000 N) could possibly indicate the correspondence of material removal mechanism with respect to the normal load (Fig. 7.7a-b). Also, the region (i.e. sub-surface layer) beneath the grooves is often subjected to severe deformation during the grooving action, leading to the morphological changes (Figs. 7.12-7.14).

The severity of the abrasion (i.e. high stress) increases with an increase in the normal load, subsequently leading to an increase in the sub-surface layer thickness. Thereby, this results in a substantial rise in the deformation (i.e. fine grain structure, Figs. 7.12e and 7.14f) and sub-surface layer thickness of martensite and tempered martensite compared with the other microstructures (Fig. 7.15a). Furthermore, the presence of less dislocations in the initial tempered martensite microstructure can accommodate more strain than the martensite with high dislocation and brittle nature, (i.e. higher work-hardening capability, as explained in section 5.4.2 in Chapter 5) resulting in a significantly increased sub-surface layer thickness (Figs. 7.13 and 7.15a). Also, an extensive increase in the sub-surface layer hardness would be expected in the tempered martensite. On the other hand, the highly dislocated martensitic laths undergo deformation in their sub-surface layers (Fig. 7.12). However, the low toughness of the martensitic laths results in negligible work-hardening, leading to minor sub-surface hardness increments (Fig. 7.15b). Also, the sub-surface layer displays extensive cavities (i.e. voids, Fig. 7.12e), which often results in the detachment of the martensitic laths. In other words, these regions are vulnerable to crack initiation. During abrasion, the martensitic laths reaches the threshold of deformation and further strain often results in the formation of such cavities. It is important to note that the martensite laths terminate on (1 1 0) planes due to the crystallographic constraints associated with the shear transformation [196]. Interestingly, the (1 1 0) is the slip plane, which is a highly stressed plane during deformation. As a result, it would be expected that the intervariant boundaries become the preferential sites for the void formation during abrasion similar to what was observed in the heat affected zone of HSLA steels subjected to the impact testing [197]. Moreover, the shear force associated with abrasion leads to the coalescence of these voids which results in the easy removal of material.
Pearlite and bainite display better abrasion resistance than martensite and tempered martensite during the abrasive scratch tests, i.e. less volume of material removal, $V_{rem}$ (Fig. 7.3a). At high loads, i.e. 1500 N, both bainite and pearlite display a distinct sub-surface layer. In the case of the former, the sub-surface is highly deformed which could be hardly resolved (Fig. 7.14c), whereas the phenomenon of microstructural realignment (i.e. coalescence of ferrite and cementite regions) is dominant in the latter (Figs. 7.14e and 7.17). They are multi-phase microstructures consisting of brittle and ductile phases, acting in synergy towards resisting the abrasive action [39]. In the case of pearlite, the hard cementite lamellae resist the penetration of the indenter and the ductile ferrite phase favours the realignment of lamellae. Due to the continuous strain-induced deformation during the scratch testing, it results in the formation of a dislocated ferrite along with the fragmentation of cementite into small segments in the sub-surface layer (Region B in Fig. 7.17). In addition, the augmentation in the ferrite dislocation density leads to an increase in its carbon solubility. Thereby, the cementite partly dissolves into the deformed ferrite (Region A in Fig. 7.17) [198-202]. Meanwhile, the undissolved brittle cementite in ferrite are vulnerable regions, leading to formation of voids that act as potential sites for crack initiation and propagation, leading to the detachment of the sub-surface layer (Fig. 7.14f).

On the other hand, the presence of retained austenite in bainite enhances the work-hardening through strain induced martensitic transformation of retained austenite, so called TRansformation Induced Plasticity (TRIP) effect) [37, 124, 139, 185]. This ultimately leads to the formation of a highly dislocated fresh martensitic region, which greatly corresponds to the paramount hardness increment (i.e. bainite-125.3% at 1500 N) in their sub-surface layer hardness (Fig. 7.15b). Also, the less formability of freshly formed martensite creates weak bond (i.e. multiple cracks along the sub-surface layer, Fig. 7.14c) at their interface with the bulk microstructure. Observations from previous work has shown that the intervariant lath boundaries of bainite were (1 1 0) planes similar to the lath martensite. As discussed earlier, there would be very little plasticity at high normal loads (1500 N), leading to fracture initiation [197] and detachment of their sub-surface layer (Figs. 7.14c). Furthermore, the superior abrasive response at a load range of 200 N to 1000 N, (Fig. 7.3a) is validated through the comparatively less sub-surface layer thickness (Fig. 7.15) and groove characteristics (Figs. 7.9-7.10).
A careful examination on the groove characteristics of pearlite and bainite reveals that their groove width is relatively less when compared with other microstructures (Figs. 7.10 and 7.11). This behaviour is evident through their relatively low degree of penetration, $D_p$, across the normal loads (Fig. 7.3b). SEM analysis on the grooves reveal that the micro-cracks appear only at 1000 N, which once again confirms their resistance to the grooving action (Figs. 7.10 and 7.11). These observations explain the superior abrasive performance of bainite and pearlite in the load range of 200 N to 1000 N. In addition, the material built-up on the groove sides (i.e. 200 N and 500 N) suggest the presence of ploughing mechanism, resulting in a lesser material removal (Fig. 7.18b-c). However, a sudden rise in the degree of penetration, $D_p$, is observed at 1500 N, which ultimately leads to the crack propagation and higher material displacement from the grooves. This could be directly related to the sudden increase in the average surface roughness, $R_a$, of groove profile at 1500 N (Fig. 7.7). As explained earlier, at higher loads the material displaced is usually lost as wear debris through the cutting mechanism. Thereby, a higher material loss is experienced at 1500 N in the case of bainite and pearlite. Also, their volume of material removal, $V_{rem}$, and the degree of penetration, $D_p$, are almost similar to the tempered martensite at the corresponding load of 1500 N (Fig. 7.3a). This suggests that the cutting mechanism is more dominant at loads above 1000 N in bainite and pearlite microstructures.

The current observations portray that the characteristics of microstructures play a crucial role in determining their abrasive response when subjected to a controlled scratch testing. In general, the multi-phase microstructures (bainite and pearlite) display better resistance than the single-phase microstructures (martensite and tempered martensite) during a scratch test. In multi-phase microstructures, the syndicate effort of both the phases (brittle and ductile) often becomes the driving force for their superior abrasive performance. Conversely, the homogeneity (could be brittle or ductile) of the single phase microstructures, makes them vulnerable to the abrasive action of the indenter. Nevertheless, the abrasive behaviour of the microstructures shows an increased dependence on the normal load subjected during a scratch test.
7.5 Summary

In the current study, high strain abrasive scratch testing was conducted on four different microstructures, namely bainite, pearlite, martensite and tempered martensite with similar hardness levels. The grooves made during the scratch testing revealed the abrasive behaviour was greatly influenced by the microstructure characteristics and the severity of the abrasive conditions, i.e. normal load. Nonetheless, these single-scratch tests, to some extent, reflect on actual industrial abrasive conditions, though the material is often subjected to a series of multiple/parallel scratches in the industrial scale. However, the ultimate aim was to study the abrasive behaviour of microstructures with similar hardness levels under controlled abrasive scratch tests (i.e. constant indenter tip geometry and sliding speed). The following conclusions can be drawn from this study.

1. Generally, multiphase microstructures, i.e. bainite and pearlite displayed relatively low volume of material removal, $V_{\text{rem}}$, when compared with martensite and tempered martensite consisting of mostly a single metallurgical phase. However, bainite and pearlite had better abrasion resistance at nominally loads less than 1000 N.

2. The groove characteristics (width, $w$, depth, $d$ and average surface roughness, $R_a$) of the microstructures were significantly influenced by the properties of the microstructure constituents and the normal load. However, there was a limited correlation with the degree of penetration, $D_p$, volume of material removal, $V_{\text{rem}}$ and the normal load, $P$.

3. A transition in the material removal mechanism was observed with respect to the normal load subjected during the scratch testing. For instance, the ploughing material removal mechanism was dominant at low loads (e.g. 200 N-500 N), though the cutting mechanism was quite severe at loads higher than 1000 N leading to high $V_{\text{rem}}$.

4. The distinct sub-surface characteristics of the microstructures were a clear indication of their response to the abrasive scratch test. The amount of work-hardening (i.e. increase in sub-surface hardness) had a direct and positive correlation with respect to the abrasive behaviour of the microstructures.
5. There was a significant microstructural changes (i.e. fragmentation of initial microstructure) in the subsurface layer suggesting the presence of severe plastic deformation.
Chapter 8

Conclusions and suggestions for future work

8.1 Introduction

This dissertation has revealed that the two-body abrasive wear was quite a dynamic system, involving simultaneous changes in both the microstructure and the abrasive particle characteristics. This necessitated separate investigation on both the abrasive environment and the microstructure of a material (steel) demonstrating their individual impact on the two-body abrasive wear. The influence of abrasive particle characteristics in the material removal process during abrasion was clearly outlined in the initial study. Followed by this, the significance of microstructure constituents in combating abrasion was observed through their distinctive material removal mechanisms. In ultra-high strength steels (nanobainitic steels), it was demonstrated that the retained austenite characteristics play a significant role on the abrasive wear behaviour through the extent of TRIP phenomenon. This study was further extended by conducting high stress abrasive wear tests, where the microstructure characteristics and the normal load displayed an increased effect on the two-body abrasive wear behaviour.

The subsequent section draws up the conclusions based on the results and discussion reported in Chapters 4 through 7 and offers possible suggestions and recommendations for future work.
8.2 Conclusions

8.2.1 Effect of abrasive particle characteristics on the two-body abrasive wear

The main focus of this study was to understand the impact of abrasive particle characteristics (particle size, type and density) in the two-body abrasive wear test using a pin-on-disc tribometer. The abrasive particle characteristics had a tremendous influence in determining the two-body abrasive wear behaviour of a microstructure. In the current study, a pearlitic microstructure was subjected to different abrasive environments (i.e. abrasive particle type and size) and their abrasive response was recorded. Irrespective of the particle type, the specific wear rate of the microstructure increased proportionally with an increase in the abrasive particle size. On the other hand, the friction coefficient decreased with an augmentation in the particle size. Coarse abrasive particles (SiC-58 $\mu$m and alumina-40 $\mu$m) often resulted in a relatively deeper penetration in the abrading surface than the fine abrasive particles (SiC-15 $\mu$m and alumina-20 $\mu$m). Post-wear electron microscopic examinations on the abrasive papers revealed the mechanisms of abrasive particle deterioration, which were once again influenced by the abrasive particle size. For instance, coarser particles were prone to shelling (i.e. particle detachment from resin) and attrition (i.e. loss of cutting edges and/or morphology), whereas, complete fracture also known as fragmentation was more common in the case of fine particles (SiC-15 $\mu$m and alumina-20 $\mu$m).

However, the abrasive particles were subjected to constant deterioration due to the continuous traversals of the metallic pin on the same wear track. Interrupted abrasive wear tests proved to be a valuable tool in investigating the efficiency of abrasive particles, which was quite subjective to particle density. In the current study, alumina displayed better abrading efficiency than silicon carbide, which was largely attributed to the dense packing nature in the case of the former. Such dense packing nature in alumina has resulted in a better abrading action, leading to a greater threshold level (sliding distance) of particle efficiency.

8.2.2 Impact of microstructure constituent characteristics on the two-body abrasive wear

In this study, two-body abrasive wear resistance of four distinct microstructures (i.e. bainite, pearlite, martensite and tempered martensite) with similar hardness levels (330-360 HV$_{0.01N}$) were investigated. The unique abrasive response of the
microstructures was observed through the distinctive specific wear rate and friction coefficient curve characteristics. In general, multi-phase microstructures (bainite and pearlite) demonstrated superior abrasive resistance when compared with single-phase (martensite and tempered martensite). Despite pearlite having a relatively lower hardness level (326 HV$_{0.01N}$), it displayed the lowest specific wear rate. This was ascribed to their microstructure matrix consisting of a combination of brittle (cementite) and ductile (ferrite phases). A similar rationale could be extended towards bainite microstructure comprising of bainitic ferrite and retained austenite. Single-wear track and topographical analysis further identified the amount and mechanism of material removal in the microstructures. Ploughing and wedge formation mechanisms were more dominant resulting in narrow and wide grooves in the case of multi-phase microstructures. Conversely, single-phase microstructures experienced cutting mode of material removal causing wide and shallow grooves. Schematics summarizing the abrasive response in the aforementioned microstructures was developed in the current study. However, to negate the effect of abrasive environment (i.e. particle deterioration) on the material removal process, high strain abrasive wear tests were further employed.

The high stress scratch testing to some extent reflected the actual industrial abrasive conditions. During the scratch testing, the grooves made in the microstructures were a direct measure of their abrasive wear resistance. In general, the groove characteristics (width, $w$, depth, $d$ and average surface roughness, $R_a$) were greatly determined by the microstructure characteristics and the severity of abrasive conditions (normal load). For example, multi-phase microstructures displayed superior abrasive performance at nominally loads less than 1000 N. Furthermore, a possible transition in the material removal mechanism (i.e. ploughing at 200 N – 500 N to cutting $\geq$ 1000 N) was observed for all microstructures with respect to the normal load. The distinct abrasive response of the microstructures was further evident through the sub-surface layers, which revealed significant morphological changes due to severe plastic deformation. A positive correlation was observed between the work-hardening behaviour and the abrasive resistance of a microstructure. The extent of deformation (i.e. thickness of the deformed layer) and the material fracture (i.e. crack initiation and propagation) was once again determined by the characteristics of the microstructure constituents.
8.2.3 Influence of retained austenite characteristics and work-hardening behaviour in the two-body abrasive wear

The beneficial impact of work-hardening in microstructures during abrasion turned out to be the driving force for investigating the two-body abrasive wear behaviour of nanobainitic steels. This research developed fully bainitic microstructures through isothermal bainitic transformations in a high-carbon high alloy steel. Their microstructure matrix consisted of bainitic ferrite and retained austenite with different characteristics (size, morphology, volume fraction and carbon content). The abrasive wear resistance of the microstructures displayed a direct correspondence with the extent of microstructural refinement. In other words, nanobainitic microstructures, i.e. FB-200°C consisting of fine bainitic ferrite (60±10 nm) and film retained austenite (30±5 nm) demonstrated superior abrasion resistance than other fully bainitic microstructures. The above microstructure matrix displayed very high hardness and a delayed TRIP effect. One of the major highlights of this study was the differential TRIP effect that could be obtained through different morphologies (i.e. film and blocky) of retained austenite. Film retained austenite was mechanically more stable than the blocky retained austenite. In other words, an early onset of TRIP effect in blocky austenite, i.e. FB-350°C resulted in the formation of coarse fresh martensite, which were more vulnerable to fracture during abrasion.

However, an abrasive wear study conducted on a pearlitic microstructure heat treated from the same chemical composition provided a valuable insight on the effect of TRIP behaviour in abrasion. Despite a relatively low hardness (335 HV$_{0.01N}$), the pearlitic microstructure revealed better abrasive wear resistance than FB-350 (413 HV$_{0.01N}$). This observation emphasis that the presence of retained austenite and their TRIP effect not necessarily will have a positive impact on abrasion. In summary, the morphology of retained austenite and microstructural refinement can determine the abrasion resistance in ultra-high strength bainitic steels.

8.3 Suggestions for future work

The entire research conducted a detailed investigation on the effect of microstructures and abrasive environment characteristics in the two-body abrasive wear. Also, the abrasive wear behaviour of ultra-high strength bainitic steels has shown more promise
with potential use of nanobainitic steels in abrasive wear applications. This project further highlighted the following potential areas for future work:

1. Introducing changes in the test modes of the existing high strain scratch tests, i.e. scratch tests on work-hardened surface. Here, multiple scratches with different indenter radius sizes will be performed on the same area using the single-scratch testing approach. This type of study can reflect the industrial abrasive conditions to a greater extent, where the steels are continuously subjected to multiple abrading actions. This can be conducted on a wide range of steels with different work-hardening capabilities such as ultra-high strength bainitic steels, TWIP steels and dual-phase steels.

2. The findings from Chapter 4 highlights the effect of abrasive particle characteristics in the two-body abrasive wear. However, there is a need for an in-depth characterization of the abrasive particles, i.e. abrasive particle angle and morphology. Such studies can aid in correlating the abrading action of the particles with the metallurgical phases in a microstructure.

3. The measurement of shear strain distribution at the deformed sub-surface will be evaluated in future. Also, crystallographic texture analysis of the region beneath the abraded surface can disclose the main restoration mechanism/s taking place due to the frictional heat.
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