A study of micro-particles in the dust and melt at different stages of iron and steelmaking

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To my beloved parents
Abstract

This study focuses on two different types of micro-particles selected from different stages of iron and steelmaking processes. These particles are dust particles generated due to mechanical wear of iron or particles and clusters formed in molten stainless steel alloyed with rare earth metals (REM). Firstly, the influence of three factors on the size distribution of the dust generated from iron ore pellets was investigated. The investigated factors include the characteristics of iron ore pellets, applied load on a pellet bed and partial reduction of iron ore pellets. Secondly, three dimensional investigation of REM clusters extracted by using electrolytic extraction are carried out to evaluate the size distribution of the clusters. Moreover, an extreme value distribution (EVD) analysis has been applied for the observed REM clusters.

A planetary mill was used to investigate the influence of the characteristics of pellets on the dust generation. It was observed that the size of pellets can influence the wear rate under the given experimental conditions. The pellets with larger size ($13.5 < D_{eq} < 15.0$ mm) showed a 10 to 20% higher wear rate as compared to small sized pellets ($9.5 < D_{eq} < 12.5$ mm). Based upon the analysis of the dust generated during the wear experiments, the mechanism of wear of pellets was identified as abrasion and collision wear.

In addition, a pellet bed setup was designed to study the influence of applied load on the dust generation and friction forces in a pellet bed. A varied load of 1 to 3 kg was applied on the pellet bed. An increase of ~67% was observed in the friction and the dust generation in the bed as the applied load increased from 1 to 3 kg. Moreover, it was observed that a higher friction in the pellet bed can lead to an increased amount of airborne particles. The mechanical wear of pellets reduced at 500 °C (P500) and 850 °C (P850) was carried out in a planetary mill. It was found that P500 pellets exhibit a ~16 to 35% higher wear rate than reference unreduced pellets. For the P850 pellets, the mechanical wear is inhibited by a formation of a metallic layer at the outer surface of the pellets. Further, the dust generated due to mechanical wear of reduced pellets contained 3 to 6 times higher amounts of coarse particles ($> 20 \mu m$) as compared to the dust from unreduced pellets. Moreover, considering the industrial aspects, the mechanisms involved in the mechanical wear of pellets at different process steps and the relation between the velocity of off-gases and the size and morphology of the dust particles is discussed.

REM-oxide clusters extracted from 253MA stainless steel grade samples were investigated in three dimensions (3D). A reliable cluster size distribution (CSD) was obtained by improving the observation method and it was used to explicate the formation and growth mechanism of REM-oxide clusters. The circularity factor of clusters was used to divide the clusters into two different groups, which form and grow in accordance to different mechanisms. The results also show that the growth of clusters is governed by different types of collisions depending on the size of the clusters. It has been concluded that for REM-oxide clusters turbulent collisions are the main controlling mode for the growth rate.
The problem of unit areas without any clusters, in an extreme value distribution (EVD) analysis, has been discussed. Moreover, three different size parameters were considered for EVD analyses. The results show that using the maximum length of clusters ($L_c$) results in a better correlation of EVD regression lines by improving $R^2$ value up to 0.9876 as compared to 0.9656 – 0.9774 for other size parameters. Moreover, a comparison of predicted and observed maximum lengths of clusters showed that further work is required for the application of EVD analyses for REM clusters.

**Key words:** Particle size distribution, ironmaking, iron ore pellets, dust generation, mechanical wear, friction, reduction, REM clusters, Electrolytic extraction, cluster size distribution, growth mechanism, collisions, statistical analysis.
Sammanfattning

Studien fokuserar på två olika typer av mikropartiklar som är valda från olika delar av järn- och ståltillverkningsprocessen. Dessa partiklar är dels stoft som genereras på grund av mekanisk nötning av partiklar och dels klusters som bildas i flytande rostfria stål legerate med sällsynta jordartsmetaller (REM). Inledningsvis så undersöks inverkan av tre faktorer på storleksfördelningen hos stoft som bildas vid hantering av järnoxidpellets. De undersökta faktorerna inkluderade karakteristiken hos järnoxidpellets, det applicerade trycket på pelletsbädden och den partiella reduktionen av järnoxidpellets. Därefter så utfördes tredimensionella undersökningar av REM kluster som extraherats med hjälp av elektrolytisk extraction för att bestämma storleksfördelningen hos klustren. Dessutom så utfördes en extremvärdesdistribution (EVD) studie för de studerade klustren.

En planetkvarn användes för att undersöka inverkan av karakeristiken hos pellets på stoftbildningen. Resultaten visade att storleken på pellets kan påverka nötningshastigheten under dessa försöksförhållanden. Pellets som hade en större storlek (13.5 < D<sub>eq</sub> < 15.0 mm) uppvisade en 10 till 20% högre nötningshastighet i jämförelse med mindre pellets (9.5 < D<sub>eq</sub> < 12.5 mm). Baserat på analyserna av stoftet som genererades under nötningsexperimenten så konstaterades att nötningsmekanismerna för dessa pellets var abrasions- och kollisionsnötning.

En pelletsbädd skapades för att möjliggöra studier av inverkan av ett applicerat tryck på stoftbildningen och friktionskrafterna i en pelletsbädd. Ett varierat tryck på mellan 1 till 3 kg applicerades på pelletsbädden. Resultaten visade att en ökning på ~67% av friktionskraften och stoftbildningen ägde rum när det applicerade trycket ökades från 1 till 3kg. Dessutom så visade resultaten att en högre friktionskraft i pelletsbädden kan resultera i en ökad mängd luftburna partiklar. Den mekaniska nötningen av pellets som reducerats vid 500 °C (P500) och 850 °C (P850) studerades också genom användande av en planetkvarn. Resultaten visade att P500 pellets uppvisade en ~ 16 till 35% högre nötningshastighet i jämförelse med oreducerade referenspellets. Resultaten för P850 pellets visade att den mekaniska nötningen motverkades genom bildningen av ett metalliskt skikt på den yttre delen av pelletsen. Resultaten visade också att stoftet som bildats pga mekanisk nötning av reducerade pellets innehöll 3 till 6 gånger mer grova partiklar (>20µm) i jämförelse med stoft som bildats från oreducerade pellets. Slutfinal forsökades hur dessa resultat kan relateras till industriella förhållanden med avseende på demekanismerna som är involverade i den mekaniska nötningen av pellets samt med avseende på relationen mellan hastigheten av de utgående gaserna och storlken och morfologin hos stoftpartiklarna.

Klusters innehållande REM-oxider som extraerats från en 253MA rostfri stålsort undersökes med användande av en tredimensionell teknik. En trovärdig storleksfördelning av klusters (CSD) erhölls genom att förbättra undersökningsmetoden och denna användes för att studera bildningen och tillväxten av REM oxider. Dessutom så användes kategorisfaktorn hos klustren för att delta in klustren i två olika grupper, vilka bildas och tillväxter enligt olika mekanismer. Resultaten visade också att tillväxten av klusters gynnas av olika typer av kollisioner som beror av av storleken på klustren.
För REM-klusters så drogs slutsatsen att turbulenta kollisioner är den huvudsakliga mekanismen som påverkar tillväxten.

Avhandlingen behandlar även problemet om hur det är möjligt att hantera synfält där det inte förekommer kluster vid en extremvärdesdistribution (EVD) analys. Tre olika parametrar undersöktes i EVD analysen. Resultaten visar att om den maximala längden på kluster ($L_C$) används i analysen så erhålls den bästa korrelationen gällande regressionslinjen för en EVD analys. Specifikt så var $R^2$ värdet upp till 0.9876 i jämförelse med de andra storleksparametrarna som har värden i intervallet 0.9656 – 0.9774. Slutligen så visar resultaten från en jämförelse mellan beräknade och observerade maximala klusterlängder att EVD analyser för studier av REM kluster behöver undersökas ytterligare i framtiden.

**Nyckelord:** Storleksfördelning, järnframställning, järnoxidpellets, stoftbildningen, mekanisk nötning, friktion, reduktionen, REM kluster, elektrolytisk extraktion, mekanism, kollision, statistisk analys.
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1. Literature survey, experimental work, major part of the writing.
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4. Literature survey, experimental work, mathematical calculations, major part of the writing.
5. Literature survey, experimental work, mathematical calculations, major part of the writing.

Parts of this work have been presented at the following conferences:

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1. Introduction

The increasing demands on a sustainable production in combination with high performance products are challenging for iron and steel industry, especially due to formation of inevitable products during the different process steps. These products are formed at almost every process step and they can for instance be flue dust, sludge and slag during iron making, non-metallic inclusions in molten steel and mill scale and chips during product processing. Moreover, these products have a wide range of sizes i.e. from micro level to a macro level. Among these, micro sized products can be abstruse due to their small sizes and difficulties to analysing them. Currently, industries are taking several steps toward sustainable development including recycling and reusing the by-products as well as by improving the properties of the final products. However, any other further step towards a sustainable steel production is always welcome.

In the current study two different kinds of micro sized products, namely dust particles generated from iron ore pellets and clusters found in stainless steel alloyed with rare earth metals (REM), which are formed during the different process steps were investigated. The selection of these two type of particles (micro sized products) is based upon the recent attention earned by them in the iron and steel industry in the Nordic countries. Moreover, considering the fact that both these products can be problematic during the production while one of them is a result of degradation and is generated in solid state and the other one is formed and grows in molten state, it is of interest to investigate if specific information about their size distributions can be useful for improvements of the production processes. Therefore, an effort was made to investigate the size distribution of these particles to seek meaningful information which can be relevant to the industrial for minimizing their harmful effects, for instance with respect to the formation/ generation of the micro sized particles.

A considerable amount of dust is generated due to mechanical wear/degradation of iron ore pellets during handling, transportation and iron making process. The dust generation is undesired since it leads to significant material losses as well as due to that it causes environmental issues. The higher amounts of exiting flue dust from a blast furnace (BF) increases the energy consumption needed for the production of hot metal. Moreover, the dust particles generated in a BF can clog the voids present in the burden. This, in turn, influences the transport of the reducing gases in the burden. As a result, this increases the coke consumption and this decreases the blast furnace productivity.1-7)

Additions of rare earth metals (REM) to steel is beneficial as REM can modify the shape of sulfide inclusions and improve the hot workability.8) However, steels deoxidized or alloyed with REM are difficult to produce due to the high tendency of REM-oxides to form cluster and clog the nozzle during casting.9,10) Therefore, it is desired to control the formation and growth such clusters in liquid steel during ladle treatment of stainless steels.
1.1. Iron ore pellets’ dust

Iron ore pellets can undergo a degradation according to different mechanisms to produce dust particles. Firstly, degradation may occur due to attritions and collisions between pellets themselves, between pellets and other charged materials and between pellets and other surfaces such as the walls of loading vessels, storage bins and furnace. Secondly, degradation can occur due to decrease in the mechanical strength at higher temperatures. For instance, the pellets lose their mechanical strength at elevated temperatures due to thermal expansion and vaporization of volatile components. Moreover, during reduction in a blast furnace the strength of pellets decreases due to volume and crystallographic changes associated with reduction. The decrease in the strength of iron ore pellets destabilize their structure and generate conditions favorable for dust generation. The dust particles generated through these mechanisms can be classified as mechanically generated dust.

Dust generation due to mechanical wear/degradation of iron ore pellets happens during the transportation and handling as well in a BF while descending down along with other charged materials. The amount of dust produced during the transportation and handling of iron ore pellets depends on the severity of the transportation and handling steps such as vessel loading systems, number and height of drops, the length of conveyor belts and descend velocities in bins. The generated dust can become airborne during transportation and in the production facility, which is considered hazardous to the human health and which is problematic for the environment.

1.1.1. Dust generation during transportation and handling

Several standard and non-standard tests have been developed in order to evaluate the dust generation tendency of iron ore during handling and transportation. These tests include, a tumble test, a modified tumble test, a shatter/drop test, a dust tower and a vibration test. Among these the tumble test, performed in accordance to the ISO 3271 standard, is most commonly applied. In this test, 15kg of a test sample is placed in a circular rotational drum and it is rotated at 25 rpm for 200 revolutions. Thereafter, the test material is sieved in the fractions of +6.3mm and -0.5mm and the weight percent of -0.5mm material is reported as the dust generation tendency (Abrasion Index, AI). Similar to for the tumble test, most of the other tests report the fraction of material under 0.5mm as the dust generation tendency. However, in a dust tower test, developed by Copeland and Kawatra, the dust generation tendency of iron ore pellets called ‘dustiness’ is measured in terms of dust particles, which can become airborne at a given air flow rate.

In the dust tower test, iron ore pellets are dropped in a tower of 2.7m height which contains inclined slants to replicate the degradation of iron ore pellets caused by a drop during the handling of the pellets. The mass concentration of PM$_{10}$ particles captured by an aerosol monitor are reported as the dustiness. Where, PM$_{10}$ refer to particles with an aerodynamic diameter of up to 10µm, which roughly corresponds to a spherical particle diameter of 4-5µm for hematite particles. The dust tower has been used to investigate the factors affecting the dust generation from iron ore pellets, such as the pellet chemistry, firing temperature, coke breeze addition, abrasion index and compressive strength.
It was found that the pellet chemistry and pellet firing temperature are the main factors influencing the dust generation. These investigations provide an insight about the relationship between the pelletization and dust generation. However, the factors which might influence the dust generation during transport and handling were not considered.

1.1.2. Dust generation in a blast furnace

Over the past couple of decades, the blast furnaces (BF) in the Nordic region has shifted from using a mixture of iron ore sinter and pellets to using 100% pellets as raw materials. This transition is beneficial due to the advantages of pellets in comparison to sinter. These include a more regular particle size distribution, a consistency in the chemical composition and a good reducibility. Moreover, better operating results can be achieved in term of productivity and fuel rate by using 100% pellets in comparison to using a mixture of pellets and sinter. During the descending of the pellets in a BF, a considerable amount of dust is formed which exits the blast furnace with the off-gases. Thereafter, dust from BF gases is separated into dry flue dust (coarse fractions) and sludge (fine fractions) using a two stage gas cleaning system. The flue dust particles consist mainly of 20-40wt% Fe from pellets and 30-50wt% C from coke. The amount of generated dust is related to the choice of the ore feed. A BF operation using 100% pellets produces a higher amount of dust compared to a BF using a mixture of pellet and sinter. For instance, a 100% pellets operation produces 13 to 18 kg dry flue dust per ton of hot metal. This is significantly higher in comparison to an average of 6 kg per ton of hot metal for European BFs operating with mainly sinter. Based upon an investigation of off-gas dust, it has been concluded that the Fe oxide particles in flue dust are mechanically formed due to wear of the pellets.

The maximum wear / degradation of iron ore occurs in the upper part of BF where iron ore encounters low temperature zones (400 – 600 °C) and start to reduce. This phenomenon is associated with the stress concentrations caused by the volume expansion during the reduction of hematite to magnetite. A higher mass percent of particles <0.5mm is produced, 0.7 – 3 times higher, due to reduction degradation of iron pellets as compared to lump ore and sinter when reduced at 550 °C. This can be of concern for the BFs operating with 100% pellet as Fe charge.

Generally, the standard ISO 4696-1 is used to determine the wear / degradation of reduced iron ore, also known as reduction disintegration index (RDI). This is done by isothermally reducing the ore at 500 °C for 60 min using a gas mixture of 20% CO, 20% CO₂, 58% N₂ and 2% H₂. This is followed by a tumbling of the partially reduced material in a tumble drum. Finally, the weight percent of the fractions > 6.3 mm, < 3.15mm and < 0.5mm are reported.

As mentioned above, in the standard tests to evaluate the wear / degradation of iron ore pellets and most of the non-standard tests, dust is defined as the fraction of materials less than 0.5mm in size. Using the dust tower, J. A. Halt and S. K. Kawarta has shown that the abrasion index and ‘dustiness’ has a poor correlation for iron ore pellets. However, the dustiness has been defined as the PM₁₀ level (i.e. 4-5μm for hematite particles). In case of dust emissions from a blast furnace, not only PM₁₀ particles exit the BF but depending on the off gas velocities, coarse particles can also end up in the flue dust. The off-gas velocity at the top of the blast furnace can vary from ~1 m/s towards the wall.
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Moreover, the particle size distribution (PSD) of the flue dust (containing 20-30% Fe and 40-50% C) obtained by P. Sikström and L. S. Ökvist contained more than 75 wt% in the size ranges less than 250μm. The characterization of flue dust from an Egyptian iron and steel company showed that iron particles present in it had sizes of less than 210μm. Furthermore, in the case of the BF process operating by using 100% iron ore pellet as a ferrous burden, the dominant fraction of the Fe containing particles in BF flue dust is less than 63 μm. This contradiction between the defined sizes of dust and the observed amounts in industrial processes suggests that there is need to investigate the PSD of dust generated due to mechanical wear of iron ore pellets.

There also exists insufficient data regarding the influence of handling parameters on the generation of particles which can be airborne and which can be found in BF flue gases. Apart from this, information regarding the friction between pellets can be of great use for researchers working with simulation and modeling of the burden descend in a BF. Gustafsson et al. has reported an average friction coefficient of 0.67 and 0.72 for a sliding contact between pellet to pellet and pellet to steel plate, respectively. However, there is need to obtain information regarding the friction in a pellet bed as well as on the influence of different process parameters on the friction values.

1.2. Clusters in REM alloyed stainless steel

The use of REM in the steel industry has increased several folds due to their remarkable effects. REMs are used for the modification of the morphology of inclusions and suppressing their detrimental effects by forming stable globular particle compounds. The elongated manganese sulphide inclusion particles can be eliminated by addition of REM to form compounds of REM and sulphur. Additions of REM can enhance the properties of steel such as the toughness, fatigue resistance, corrosion resistance and hot ductility. Other beneficial effects of REM additions include reduction in the degree of undercooling, refining of the as-cast structure and limiting solidification segregation in steels. However, due to high tendency of REM-oxide particles to agglomerate and form clusters in liquid steel leads to nozzle clogging during casting of the steel. It was reported that the main part of the nozzle accretion is caused by agglomeration of Ce inclusion particles and clusters in the nozzle. Moreover, REM can react with alumina in the refractory of nozzle and initiate clogging, which continues by agglomeration of REM-oxide particles on the nozzle wall. Therefore, the formation and growth of inclusion particles and clusters in liquid steel during ladle treatment of stainless steels has always been under considerations to minimize their harmful effects.

1.2.1. Formation and growth

It has been shown that after an initial nucleation of inclusion particles, their growth is controlled by diffusion of molecules and thereafter collisions play an important role in the growth of inclusion particles. However, due to high tendency of REM-oxide particles to agglomerate and form clusters in liquid steel leads to nozzle clogging during casting of the steel. It was reported that the main part of the nozzle accretion is caused by agglomeration of Ce inclusion particles and clusters in the nozzle. Moreover, REM can react with alumina in the refractory of nozzle and initiate clogging, which continues by agglomeration of REM-oxide particles on the nozzle wall. Therefore, the formation and growth of inclusion particles and clusters in liquid steel during ladle treatment of stainless steels has always been under considerations to minimize their harmful effects.
than 10 μm. 48-50 These conclusions have been drawn on the basis of mathematical modeling done for spherical aluminum oxide inclusions. 48) The irregular shapes and higher density of REM-oxide clusters (6500~7200 kg/m³) in comparison to the spherical Al₂O₃ inclusions suggest that they might have a different behavior and growth rate than the Al₂O₃ inclusions.

REM inclusions have been investigated by using a Confocal Laser Scanning Microscope (CSLM). 49) Appelberg et al. 45) reported that a strong attraction force exists between REM particles, which leads to an agglomeration of inclusions. In addition, Bi et al. 51) discussed that the number of large sized REM clusters increased with an increased holding time as small the sized clusters merged to form large ones. However, the mechanism of the clusters growth was not considered in detail.

1.2.2. Extreme value distribution

From an industrial point of view, it is important to estimate the size of the largest defect (inclusion particle or cluster) to assess of the quality of steel. The probable maximum size (PMS) of inclusions can be determined by using a linear regression formula obtained for an extreme value distribution (EVD) of inclusions. Several researchers have applied this method for predicting the maximum size of inclusions in various steel grades. 52-59) Based upon recommendations of Murakami’s method 54), the square root of the projected area of effective inclusion (i.e. \( \sqrt{area_{max}} \)) is used as a size parameter for EVD analysis.

The actual size of inclusions and clusters cannot accurately be determined on a cross section of a sample, especially not the actual size of clusters. Hideaki et al. 60), compared the sizes of REM clusters observed in two dimensions (2D) on a polished surface and three dimensions (3D) after an electrolytic extraction. They reported that what appears to be large clusters on a polish surface actually are small sized clusters located close to each other during the filling of samples. Moreover, Kanbe et al. 61) has also shown that the apparent size of clusters in 2D could be larger than the real size observed in 3D. Also, several studies have been performed to determine the relationship between the apparent and actual size of inclusions for EVD analyses carried out as 2D observations. 62-63) However, there is a lack of work performed on EVD determinations for clusters, especially based on 3D observations.

Kanbe et al. 61) has applied EVD analyses on 2D and 3D observations of clusters in a Fe – 10 mass % Ni alloy. It has been shown that an EVD analysis can be applied for the estimation of the largest size of clusters in metal samples. However, they have used a size parameter \( \sqrt{area_{max}} \), which was determined as the square root of the product of the maximum length and width of a cluster. Whereas, according to Murakami’s method 54) the size parameter \( \sqrt{area_{max}} \) to be used is the projected area of the defect. Therefore, the use of the length and width of clusters to calculate the size parameter \( \sqrt{area_{max}} \) might not be a good representation of the size of clusters, as it significantly can overestimate the size. Moreover, in accordance to the practice described in the ASTM E2283-03 standard 64) the maximum length of a defect shall be measured for EVD analysis. Therefore, it is required to determine a suitable size parameter of clusters to be used in an EVD analysis.

Based upon Murakami’s recommendation 54) Kanbe et al. 61) have observed 40 unit areas to perform an EVD analysis of clusters. Among these 40 observations, some observed fields didn’t
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contain any cluster. However, the authors did not properly mentioned how these fields (without clusters) were considered while performing calculations in an EVD analysis.

1.3. Objectives and overview

The main approach adopted in the current study is to investigate size distribution of different particles from the iron and steel industry for attaining meaningful information from an industrial prospective. For this purpose, two kinds of micro-particles of importance for the industrial operations have been selected i.e. dust particles generated from iron ore pellets and REM clusters formed in the REM alloyed molten steel. The overview of the current thesis is presented in Fig. 1.1.

*S = Supplement

**Fig. 1.1.** The overview of the current thesis

The first three supplements are related to the dust generation from iron ore pellets. Where Supplement 1, based upon dust generated from iron ore pellets in a laboratory setup, describes the mechanisms involved in the mechanical wear of iron ore pellets. Moreover, the influence of the mechanisms of the dust generation in different process steps are discussed. The influence of applied external loads on the size distribution of dust generated in a pellet bed during transportation and handling of the pellets and in a blast furnace is presented in Supplement 2. It also discusses the influence of the morphology of dust particles on their flow in air / gas stream. Supplement 3 deals with dust particles generated due to wear of partially reduced pellets, which is related to the dust generation in a blast furnace.

In Supplement 4, the characteristics and size distribution of REM clusters in stainless steel are investigated by using electrolytic extraction method. Based upon the size distribution of REM cluster, the mechanism of formation and growth of the clusters has been elucidated. Supplement 5 considers the application of extreme value distribution (EVD) analysis for REM clusters. Moreover, it discusses and resolves issues encountered for EVD analyses of clusters.
Overall, the specific objectives of the current study are to

- mechanically generate dust from iron ore pellets in laboratory experiments which has similar size distributions to industrial dust.
- investigate the influence of the pellet characteristics on the mechanical wear.
- study the influence of the applied load and friction on size the distribution of dust generated in a pellet bed.
- examine the mechanical wear of partially reduced iron ore pellets.
- consider the size distribution of the experimentally generated dusts for evaluation of mechanism involved in mechanical dust generation of iron ore pellets.
- investigate the characteristics of REM clusters by using a three dimensional method.
- use the size distribution of REM clusters for elucidation of their formation and growth mechanisms.
- resolve the issue of observed fields without cluster during the application of an extreme value distribution analysis for REM clusters.
- determine appropriate size parameter to be adopted for EVD analyses of clusters.
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2. Experimental

2.1. Investigation of dust particles

The commercial iron ore pellets were used for the generation and investigations of dust particles. Moreover, the dust generated during transportation and handling of industrial pellets, denoted as ‘industrial dust’, was analyzed for PSD.

2.1.1. Investigated factors

The influence of the following factors on the generated dust from iron ore pellets was investigated.

a) Characteristics of pellets (weight, size, circularity factor, density and hardness).

b) Applied external load on a pellet bed.

c) Partial reduction of iron ore pellets.

a. Characteristics of pellets

The size and circularity factor (CF) of pellets on images were determined by using the “Image J” software. The size of each pellet is presented an equivalent diameter ($D_{eq}$) of a circle, which has the same area as the measured area of the pellet ($A_{pel}$) on a two dimensional image, as given below.

$$D_{eq} = \sqrt{\frac{4A_{pel}}{\pi}} \quad (2-1)$$

The circularity factor is defined as follows:

$$CF = \frac{4\pi A_{pel}}{P_{pel}^2} \quad (2-2)$$

where $P_{pel}$ is the perimeter of pellet on an image.

Based upon Archimedes principle the densities of 30 pellets having different sizes were determined using water or 99.99% pure ethanol. In addition, a digital Vickers hardness tester was used to determine the micro hardness (HV) of the pellets in accordance to the ISO6507-1 standard. The HV values for pellets were measured at different points on a central section of a polished surface by testing from the surface to centre of the pellets. For each point, three measurements were done. This was done for 3 pellets to obtain average HV values.

b. Applied external load

A varied load of 1 – 3 kg was applied on a close packed pellet bed to study the effect of an applied external load on the dust generation in the bed. The pellet bed was subjected to mechanical wear, whose procedure is described in section 2.1.2.2.

c. Partial reduction of pellets

A schematic illustration of the furnace used for the reduction of iron ore pellets is presented in Fig. 2.1. Prior to heating the pellets in the furnace, it was flushed with Ar gas for 30 min followed by 30 min of CO flushing at 0.1 L/min. Thereafter, the pellets were heated to the desired temperature at
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a constant heating rate of 5 °C/min and held for a certain time under a CO atmosphere. The reducing gas was supplied at a flow rate of 0.5 L/min during the entire holding time. The holding time was 120 and 60 min for the reduction temperature 500 and 850 °C, respectively. After reduction, the pellets were cooled down in the furnace which was supplied with CO at a flow rate of 0.1 L/min. At the end before taking out the pellets, the furnace was again flushed with Ar gas for 30 min.

![Fig. 2.1. Setup for reduction of iron ore pellets](image)

The reduction degree ($RD$) of iron ore pellets was calculated according to the following equation.

$$RD = \frac{W_{br} - W_{ar}}{W_{br} \times \%O / 100} \times 100\%$$

where $W_{br}$ and $W_{ar}$ are the weight of the pellets before and after reduction, respectively. The parameter $\%O$ is the weight percentage of oxygen present in pellets as iron oxides. This weight percentage of oxygen in the used pellets is 28.68 % (95.25% Fe$_2$O$_3$ and 0.5% FeO).

### 2.1.2. Dust generation

As mentioned earlier, the dust generated during the transportation and handling of iron ore pellets is due to the mechanical wear / degradation of the pellets. Moreover, the dust particles found in BF off-gases are also mechanically generated. Therefore, laboratory experiments, using two different experimental setups, were performed to simulate the dust generation due the mechanical wear of pellets. The adopted experimental setups are explained in following section.

#### 2.1.2.1. Dust generation / Mechanical wear in a planetary mill

A certain amount of pellets (60-62g), in order to maintain similar experimental conditions, placed in a metallic container (inside diameter = 50 mm, depth = 30 mm) was rotated for a given time in a planetary mill (FRITSCH, Pulverisette) at a rotation speed of 400 RPM. The wear trials were carried out by using two methods: i) Method A - the generated dust was kept in the metallic container during the whole rotational time and ii) Method B - the dust generated during each time interval ($\Delta t$) was removed from the container to investigate its characteristics. A Schematic illustration of the experimental equipment and the main operations of the experimental trials are shown in Fig. 2.2. The weight of pellets was measured before ($W_t$) and after ($W_{t+\Delta t}$) the rotation and for each time
interval. Thereafter, the wear rate (WR) of pellets during a time interval was calculated according to Eq. (2-4):

\[
WR = \frac{W_t - W_{t+\Delta t}}{W_t} \cdot 100\%
\]  (2-4)

Fig. 2.2. Schematic illustrations of (a) experimental equipment and (b) main operations during different experimental trials. \(W_P\) and \(W_d\) represent the weights of pellets and dust at different intervals, respectively.

2.1.2.2. Dust generation in a closed pack bed of pellets (pellet bed setup)

**Figure 2.3** presents a schematic illustration of the equipment used for the dust generation in a closed pack bed of pellets. The pellets lie in the container on the bottom plate, which rotates by a motor. The rotation of pellets on the bottom plate makes the upper loading plate, which contains holes, and loading rod to rotate. The friction force, which is generated amongst the pellet bed, is transferred through the loading rod and measured by the load cell. The inside surfaces of bottom and upper metal plates are covered by a soft natural rubber to control an additional dust generation due to the friction between pellets and metal plates. The equipment also consists of an air inlet and outlet. The outlet can be connected to a particle counter for continuous measurement of the particle size distribution (PSD) of dust particles in the air flow. In addition, the dust particles which did not exit the container with the air flow were collected at the bottom of the container.
Fig. 2.3. Schematic illustration of the pellet bed test equipment.

About 700 g of pellets were placed in the container and rotated at 240 RPM for time interval (Δt) of 10 minutes. The container was supplied with an inlet flow rate of compressed air of 8 L/min. A volumetric flow rate of 8 L/min at the inlet corresponds to a vertical air velocity of 0.013 m/s in the container. Also, the applied load varied from 1 to 3 kg. A S2M force load cell with a measuring range from 0-20 N was used for the friction measurements. The signals from the force load cell were acquired at a frequency of 2 Hz. Moreover, the pellets were cleaned by compressed air to remove dust from the surface of the pellets before the experiments. The weight of the pellets was measured before ($W_t$) and after ($W_{t+\Delta t}$) the rotation and the wear rate ($WR$) of the pellets was calculated according to Eq. (2-4).

2.1.3. Evaluation of the size distribution of dust particles

Three different methods were adopted for analysis of dust generated during the wear experiments, which are explained in the following section.

2.1.3.1. SEM Method

For evaluation of the particle size distribution (PSD) of the dust generated due to the mechanical wear of pellets, a weighed amount of dust dispersed in ~20 ml methanol was filtered through a PTFE film filter with an open pore size of 0.1 μm. Thereafter, a scanning electron microscope (SEM) was used to investigate the dust particles on the surface of a film filter at magnifications of 300 to 1000. Also, ImageJ was used for the measurement of each dust particles on the SEM images. The maximum length ($L_{max}$), the circularity factor ($CF$) and the area of particle ($A_p$) were determined for each particle. Moreover, the equivalent diameter ($d_{eq}$) of each particle was calculated according to Eq. (2-1) using the area of particle, $A_p$. 

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The PSD of the dust particles was determined as the number of particles in the respective size range per unit weight \( (N_W) \) of dust. This was calculated as follows:

\[
N_W = \frac{n_p \cdot A_f}{A_{obs} \cdot W_d}
\]

where \( n_p \) is the number of investigated particles in the given size range, \( A_f \) is the total area of PTFE filter (~1200 mm\(^2\)), \( A_{obs} \) is the observed area on PTFE filter and \( W_d \) is the total weight of dust dispersed on the film filter. Fig. 2.4 shows typical commercial pellets and dust particles observed on film filters.

2.1.3.2. Laser diffraction (LD) Method

In this method CILAS 1064 laser diffraction particle size analyser (liquid mode) with a particle size measurement range of 0.04 to 500 \( \mu \)m was used. Here, the sizes of particles are determined based upon diffraction patterns in accordance to the Fraunhofer theory of diffraction. It is to be noted that the results obtained based upon the Fraunhofer approximation are not very accurate for submicron particles.\(^{66}\) The volume fractions of particles in different size intervals are reported by a laser diffraction particle size analyser, where the measured size is the equivalent diameter \( (d_{eq}) \) of particles in random orientations. The obtained volume fractions were converted to \( N_W \) by calculating the weight and number of particles in each size interval.

2.1.3.3. Particle analyzer, Dekati ELPI+

During the dust generation in a pellet bed a particle analyzer, Dekati ELPI+ (Electrical Low Pressure Impactor), was connected at the outlet. This analyzer can measure the particle concentration in the size range of a 6 nm to 10 \( \mu \)m aerodynamic diameter \( (d_a) \). In the ELPI+ analyzer the concentration (number per unit volume of air, \( N_V \)) of particles is measured by charging the particles in a corona charger. Thereafter, the total charge collected on an impactor stage in a specific size class is converted into \( N_V \). Finally, the concentration of the particles in the fixed 14 size intervals of \( d_a \) is reported. The aerodynamic diameter can be translated to equivalent diameter of spherical particles by using the following equation for the Stokes diameter \( (d_s) \):\(^{67}\)

\[
d_a = d_s \left( \frac{\rho_p}{\rho_d} \right)^{1/2}
\]
where \( \rho_a \) is the density of a water droplet (1000 kg/m\(^3\)) \(^{68}\), \( \rho_p \) is the density of particles (5200 kg/m\(^3\) for hematite). \(^{69}\) In this case, the size range of the dust particles measured by the ELPI+ analyzer corresponds to values between 0.003 and 4.47 \( \mu \)m of the Stokes diameter for spherical particles.

Before starting the experimental measurements, the air in the container was analyzed to obtain the background level of the particle concentrations in injected air.

The LD method and particle analyzer were only used for the evaluation of dust generated during experiments conducted using the pellet bed setup.

### 2.2. Investigation of REM clusters

The investigations were carried out on the REM clusters found in steel samples taken during a pilot trial (350 kg) from liquid 253MA stainless steel alloyed with rare earth metals. The 253MA stainless steel mainly contains (in wt\%): 17-19\% Cr, 7-9\% Ni, 1.3-1.4\% Si and 1.3-1.4\% Mn. The pilot trial was carried out in an induction furnace under an Ar atmosphere. Double thickness lollipop samples (LP4/12 having 4 and 12 mm thickness) were taken from liquid steel at 3 (sample S1), 6 (S2) and 9 (S3) minutes of holding time after an addition of 1.49 kg of misch-metal. The misch-metal contained about \(-50\%\ Ce, -35\%\ La and -15\%\) of the other REM elements.

#### 2.2.1. Evaluation of the size distribution of clusters

The REM clusters extracted from the steel samples by electrolytic extraction (EE) were analyzed to obtain their size distribution. For EE a 2\%TEA electrolyte (2 v/v\% triethanol amine – 1 w/v\% tetramethylammonium chloride - methanol), 40–60 mA current and 3.6–3.8 V voltage were used. The weight of the steel dissolved during the extractions, \( W_{\text{dis}} \), varied between 0.11 and 0.19 g. The extracted clusters, collected on a polycarbonate (PC) film filter with an open pore size of 0.4 \( \mu \)m, were investigated in three dimensions (3D) by using an SEM at magnifications of 300–5000 times. The following two observation methods were used to obtain the size distribution of clusters:

- **Method 1**: All clusters were observed on an \( A_f \) unit area \((A_f=3 \text{ mm}^2)\) of a film filter at a magnification of 500 times. The area observed by Method 1 was 15 mm\(^2\) for each sample.

- **Method 2**: Only large size clusters (larger than 5 \( \mu \)m for samples S1 and S2 and larger than 10 \( \mu \)m for sample S3) were observed on an \( A_f \) unit area \((A_f=15 \text{ mm}^2)\) of a film filter at a magnification of 300 times. The area observed by Method 2 varied from 15 to 60 mm\(^2\), due to different number of clusters present in the different samples.

The \( N_V \) value of clusters in different size intervals was calculated as follows:

\[
N_V = n_c \cdot \frac{A_f}{A_{\text{obs}}} \cdot \frac{\rho_m}{W_{\text{dis}}} \quad (2-7)
\]

where \( n_c \) is the number of clusters in the given size interval. The parameters \( A_f \) and \( A_{\text{obs}} \) are the total area of the film filter (~1200 mm\(^2\)) and the area of the filter observed by SEM. The parameter \( \rho_m \) is the density of the steel (~0.0078 g/mm\(^3\)).

The maximum length \( (L_c) \), width \( (W_c) \), area \( (A_c) \) and circularity factor \( (CF) \) of each cluster on the SEM images were measured by using the “ImageJ” software. **Fig. 2.5** illustrates the measurement of maximum length, width and area of cluster on converted SEM image by using ImageJ.
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2.2.2. Extreme value distribution

Extreme value distribution (EVD) analyses were performed according to ASTM E2283-03 \(^{64}\) and Murakami’s method. \(^{54}\) The size of the largest cluster on each unit area \((A_0)\) was measured for a total observed area of 15 mm\(^2\) of a film filter. All the measured cluster sizes were sorted in an ascending order and given a ranking. Thereafter, the reduced variate of each size data, \(y_i\), was determined by using Eq. (2-8) and plotted against a size parameter.

\[
y = -\ln \left( -\ln \left( \frac{i}{n+1} \right) \right) \quad (1 \leq i \leq n)
\]

(2-8)

where \(n\) is the number of investigated unit areas on a film filter.

A regression line of EVD data can be calculated by the maximum-likelihood (ML) method according to the ASTM E2283-03 standard. \(^{64}\) The obtained EVD regression line is applied to extrapolate the predicted maximum size (PMS) of the largest inclusion in a reference area of steel sample, \(A_{\text{ref}}\). The PMS in the reference area in steel sample can be determined from the reduced variate, \(y\), which is calculated for \(A_{\text{ref}}\) from Eqs. (2-9) and (2-10).

\[
y = -\ln \left( -\ln \left( \frac{T-1}{T} \right) \right)
\]

(2-9)

\[
T = \frac{A_{\text{ref}}}{A_0}
\]

(2-10)

where \(T\) is the return period for the expected area.

In this study to determine appropriate size parameter for EVD analysis of clusters three different size parameters were investigated, i.e. \(L_c\), \(\sqrt{area_{\text{max}}}\) and \(\sqrt{area_{ij}}\). Where the size parameter \(\sqrt{area_{\text{max}}}\) and \(\sqrt{area_{ij}}\) for a cluster were calculated according to Eq. (2-6) and (2-7), respectively.

\[
\sqrt{area_{\text{max}}} = \sqrt{L_c \times W_c}
\]

(2-11)

\[
\sqrt{area_{ij}} = \sqrt{A_c}
\]

(2-12)
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3. Results and discussion

3.1. Iron ore pellets’ dust

3.1.1. Characteristics of iron ore pellets

It has been reported that the large sized iron ore pellets experience a higher wear during the Tumbler tests. Therefore, the pellets investigated in this study were classified into three groups according their weight ($W$) and size ($D_{eq}$), as shown in Fig. 3.1. Group A, B and C represent small sized pellets ($W < 3.0 \, g$, $D_{eq} < 12.5 \, mm$), medium sized pellets ($3.0 \leq W \leq 3.5 \, g$, $12 \leq D_{eq} \leq 14 \, mm$) and large sized pellets ($W > 3.5 \, g$, $D_{eq} > 13.5 \, mm$), respectively. In this study, the characteristics of pellets from Groups A and C were evaluated and compared. Whereas, the pellets Group B were not considered due to a significant overlapping of their characteristics with Groups A and C. The weight of pellets being a precise and easy factor was selected for the classification of pellets. Moreover, $D_{eq}$ value can be affected by the positioning of pellet. The average values of the circularity factor ($CF$) for the pellets of Group A (0.85±0.02) was observed to be slightly higher compared to those of Group C (0.81±0.02) indicating that the pellets from Group C are irregular in shape compared to those from Group A.

![Fig. 3.1. Relationship between the size ($D_{eq}$) and weight ($W$) of pellets and a classification into different groups.](image)

The pellets from both groups (Groups A and C) exhibit similar values of the density as well of the micro-hardness, as can be seen in Fig. 3.2. However, it was found that the HV values tends to decrease when moving from the surface to centre of the pellets, i.e. the outer layer is 2.6-3.3 times harder than the center, as seen in Fig. 3.2(b). This phenomenon can be justified by the presence of hematite in
the outer layer of pellet and due to a gradual increase in the percentage of magnetite, which is relatively softer than hematite, towards the center of pellet. 171)

3.1.2. Influence of characteristics of pellets on dust generation

The wear trials using the planetary mill were conducted to investigate the influence of the characteristics of pellets on the generated dust. As mentioned above, the pellets exhibit very similar characteristics in term of CF, HV and density value. Also, they were classified in different groups on basis of weight and size. Before investigating the classified groups, evaluation of the experimental setup was carried out.

3.1.2.1. Evaluation of experimental setup

The experimental setup was evaluated by adopting two different test methods, two rotation intervals and comparing the PSD of the generated dust to the industrial dust.

Test conditions

A time interval of 5 min and pellets from Group A were selected to compare the wear rate / dust generation by using Method A and Method B (without and with removing the generated dust from the container). For Method A, a drastic decrease (~2.5 times) in the wear rate of pellets was observed with an increasing rotational time, whereas a slight increase in the wear rate occurred by using Method B, see Fig. 3.3. Moreover, selecting a shorter time interval (i.e. \( \Delta t = 2 \) min) resulted in a higher wear rate of pellets from Group C obtained by Method B, as shown in Fig.3.3(b). These results can be explained by the so called “cushioning effect” of dust, which has been reported in several previous studies carried out to study the degradation of iron ore. 172) The cushioning effect means that the presence of generated dust in the container can reduce the wear rate of the pellets. Therefore, in order to minimise the cushioning effect of dust, a time interval of 2 min and Method B were selected for the further investigations.
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Fig. 3.3. Comparison of wear rates obtained using (a) different test methods for Group A pellets and (b) different time intervals for Method B tests of Group C pellets.

**PSD of generated dust**

**Figure 3.4** presents a comparison of the PSD values of industrial dust and experimentally generated dust. The experimental dust used for comparison were collected after 4 min of the wear (Method B) of pellets from Group A and C. It can be seen that the experimental and industrial dusts have similar PSD values, indicating that the conditions adopted in the current experimental setup can simulate the industrial dust generation process.

**Fig. 3.4.** Comparison of the particle size distributions in experimental and industrial dusts.

### 3.1.2.2. Wear Rate

**Fig. 3.5** depicts that *WR* values (obtained under same test conditions; Method B, $\Delta t = 2$ min) of both Group A and C follow a similar tendency i.e. a significant increase in the values during the first 12 to 14 minutes of rotation. Thereafter, they become almost constant. However, the wear rate of pellets from Group C (2.4-3.7 wt%/min) was significantly higher than those from Group A (2.1-3.1 wt%/min). Similar results were obtained for the trials performed at $\Delta t = 5$ min.
Fig. 3.5. Changes of wear rates for different groups of pellets depending on the time of the wear test.

Umadevi et al. \(^{70}\) reported a similar tendency i.e. in a tumbler test (ISO 3271 standard), pellets sieved in the size fraction of -16+12.5 mm exhibit a wear rate of 0.60 wt%/min. This value is higher than 0.42 wt%/min for pellets in fraction -12.5+10 mm. The aforementioned wear rates have been recalculated from the abrasion indices reported by Umadevi et al. \(^{70}\) The pellet fractions -16+12.5 mm and -12.5+10 mm roughly correspond to the size ranges of the pellets of Groups C and A in the present study, respectively. However, the wear rates observed in the present study are significantly higher (\(WR_C = 2.43\) and \(WR_A = 2.14\) wt%/min after 4 minutes of a rotation test) compared to those obtained by Umadevi et al. A higher rotation rate in planetary mill (400 RPM) in comparison to 25 RPM of ISO 3271 and a larger “cushioning effect” of dust in Umadevi’s \(^{70}\) trials are the reason for the significant differences in the wear rates.

Moreover, a higher collision energy of larger sized coke pieces has been suggested to be the reason of their higher degradation rate in a rotating drum as compared to the degradation rate of small sized pieces. \(^{73}\)

The difference in the wear rate of different sized pellets can be understood by considering the following possible wear mechanisms during the wear tests: i) wear due to sliding of the pellets over other pellets or surfaces of the metallic box (sliding/abrasion wear) and ii) wear due to collisions of the pellets with other pellets or the metallic box (collision/impact wear). The contribution of sliding wear is dependent on the total surface area of pellets, whereas the number of collisions and collision energy count for the collision wear. It can be assumed that abrasion/sliding wear should produce fine dust particles in comparison to those generated by impact/collisions, where collision energy can be one of the factors controlling the size of dust particles. As the initial weight of pellets in each trial was kept almost constant, the number of Group A pellets (23-25 pellets/charge) in a trial was higher than...
those for Group C (14-15 pellets/charge). Moreover, the total surface area of small pellets is significantly larger in these trials than those of large size pellets. Therefore, it is safe to assume that the abrasion is dominant for the pellets of Group A whereas Group C pellets undergo wear with higher contribution of collisions.

As shown in Fig. 3.5, the WR values increase during the wear tests, this can be attributed to the fact that the harder materials have a higher wear resistance and the pellets from both groups exhibit a hardness gradient i.e. the hardness of pellets tends to decrease from surface to core. However, when the pellets have worn till the softer core (after 12-14 min of rotation), the wear rate becomes almost constant.

3.1.2.3. Evaluation of generated dust

In order to quantify/validate the assumption regarding different mechanism of wear for different sized pellets, the PSDs of dust generated during wear was investigated and the investigated dust particles were divided into three size ranges, i.e. fine particles (0-10 μm), medium sized particles (10-20 μm) and coarse particles (> 20 μm). Figure 3.6 shows the typical PSDs of dusts collected after 4, 14 and 24 minutes of wear of pellets from both Group A and Group C. As can be seen in Fig. 3.6 (a) and (b), a higher (30-50%) number of fine particles are observed for dust from Group A as compared to Group C and the coarse particles are in abundance in the dust from Group C i.e. ~ 3 times higher than the values for the Group A dust. This comparison is in favour of the assumption that different sized pellets wore according to different mechanisms i.e. dominant roles of abrasion and collisions in the wear of Group A and Group C pellets, respectively. However, it is to be mentioned that this phenomenon occurred due to the selected experimental conditions i.e. fixed weight per charge of pellets.

![Fig. 3.6](image)

**Fig. 3.6.** Size distributions of dust particles generated during wear tests carried out during 4, 14 and 24 minutes.

3.1.3. Influence of external loading on dust generation in a pellet bed

An iron ore pellet bed containing ~700 g of pellets was subjected to varied applied load (1 – 3 kg) to investigate the influence of external loading on the dust generation in the bed.

3.1.3.1. Evaluation of experimental setup

As it was observed in wear experiments conducted by using a planetary mill that Group C pellets undergo a ~10 – 20 % higher wear than pellets of Group A. Therefore, the effect of size of pellets on the wear in the pellet bed setup was investigated. For this pellets from Group A, C and Mixed (without
separation of pellets into groups depending on their size and weight) were selected. A rotation of 240 RPM was induced into the pellet bed subject to a 3 kg applied load for a duration of 10 min. The obtained results of wear rates and friction force generated in bed are presented in Fig. 3.7 (a) and (b), respectively. It can be seen that similar values of the WR (0.66 ± 0.04 wt%/min) and friction force (3.81 ± 0.018 N) are obtained for all groups of pellets, indicating that wear and friction force are not influenced by the classification of pellets in groups. This suggests in the pellet bed setup the different sized pellets undergo a similar wear mechanism, where the sliding/abrasion is dominant since the pellet bed is closed packed. Therefore, the Mixed pellets (without separation into groups) were used for the later experiments. Moreover, Fig. 3.7 indicates that a good repeatability of the experimental results can be achieved by using the pellet bed setup.

![Fig. 3.7. (a) Measured wear rate after 10 min of rotation and (b) the friction forces for different pellets.](image)

3.1.3.2. Wear Rate

The effect of different applied loads (from 1 to 3 kg) on the wear rate and friction forces in a pellet bed was investigated by using Mixed pellets and conducting 8 trials in total; out of which 4 for 3 kg load and 2 trails for 1 and 2 kg loads each. The obtained results are presented in Fig. 3.8. As can be seen in Fig. 3.8 (a), both the average wear rate and friction forces increase linearly with an increased applied load having a correlation coefficient (R) of 0.999 and 0.998, respectively. Increasing the applied load from 1 to 3 kg resulted in ~67% increase in both WR and friction values.
3.1.3.3. Evaluation of dust

The dust generated during the wear experiments in the pellet bed setup is categorized as follows:

i) \( D_0 \) is the total dust generated during an experiment.

ii) \( D_1 \) is the “heavy” dust which stays at the bottom of container after an experiment. This dust was collected from bottom and analyzed by SEM and LD method.

iii) \( D_2 \) is the “light” dust which exits the pellet bed with the air flow during experiment. In addition, \( D_2 \) is further categorized as the amount of dust measured by a particle analyzer (\( D_{2PA} \)) and the remaining \( D_2 \) which is deposited on the walls and on the upper surface of the upper loading plate in chamber (\( D_{2L} \)).

Similar to the dust generated in planetary mill, the dust generated in pellet bed setup was classified in three size ranges, i.e. fine, medium and coarse for SEM Method. When defined for LD method, the size ranges of fine, medium and coarse particles approximately correspond to \( d_{eq} \leq 5 \mu m \), \( 5 \mu m < d_{eq} \leq 10 \mu m \) and \( d_{eq} > 10 \mu m \), respectively.

Comparison of LD and SEM methods

Before discussing the influence of applied load on PSD of generated dust, a comparison of LD and SEM methods is presented in Fig. 3.9. \( D_1 \) dust generated during a trial with 3 kg applied load is used for this comparison. It is to be noted that the \( L_{max} \) value measured by SEM method is recalculated to \( d_{eq} \) to enable a comparison of the results to the LD method. It can be seen that both PSD values show a similar tendency, but that the \( N_W \) values obtained from the SEM method (\( N_{W,SEM} \)) are significantly larger than those from the LD method (\( N_{W,LD} \)). For small sized particles (\( d_{eq} \leq 5 \mu m \)), \( N_{W,SEM} \) and \( N_{W,LD} \) values are very similar (on average \( N_{W,SEM} / N_{W,LD} \) ratio is 1.09±0.4) whereas this \( N_{W,SEM} / N_{W,LD} \) ratio is 2.71±1.7 for particles with \( d_{eq} > 5 \mu m \). Li et al.\(^{75} \) has also reported a similar discrepancy between PSD values in the size range of 45 to 90 \( \mu m \) obtained by using an image analysis (IA) and a laser diffraction (LD) analysis. This difference can arise due to the use of different principles of size measurements in both these techniques. In the SEM method, the equivalent size of observed particles (\( d_{eq} \)) is determined from their maximum projection area as they tend to lie on a filter surface with a
particle plane having the maximum area. Whereas in the LD method, the equivalent size is
determined according to the random orientation of the particles meaning that the size of some plate-
like particles can be underestimated depending on their orientation. Li _et al._\textsuperscript{75} proposed that the PSD obtained by IA (PSD$_{IA}$) can be translated to the PSD of LD (PSD$_{LD}$) according to the following
relationship:

\[
\text{PSD}_{LD} = \sqrt{3} \cdot \text{PSD}_{IA}
\]

(3-1)

where \( S \) is the shape factor called “sphericity”. This shape factor is equal to the circularity factor \((CF)\) used in the current study.

**Fig. 3.9.** Particle size distributions of D$_1$ dusts, generated under 3 kg applied load, obtained by the
SEM and LD methods as well as the circularity factor of particles in different size intervals.

**Fig. 3.9** also presents the PSD$_{(SEM \cdot \sqrt{CF})}$ obtained after conversion according to Eq. (3-1) along with the \( CF \) value for each size class. A decrease in the discrepancy between PSD$_{(SEM \cdot \sqrt{CF})}$ and PSD$_{LD}$ can be observed, though it still exists especially for \( d_{eq} > 5 \mu m \).

Based upon this comparison it was decided to use both SEM and LD methods for evaluating
the dust generated under different applied load.

**Effect of applied load on D$_1$ dust**

**Fig. 3.10** presents the analyses of D$_1$ dusts obtained (by using LD method) under varied applied
loads. It can be seen that the \( N_W \) values of fine particles for 3 kg are \(-13\%\) higher than that for 1 kg.
The medium sized particles doesn’t show any considerable difference, whereas the \( N_W \) value of coarse particles is \(-6\%\) higher for lower applied loads. The increase in the \( N_W \) values of fine particles for higher applied loads can be attributed to higher friction forces between the pellets. Hence, this
promotes a higher sliding/abrasion wear in the pellet bed. This was further investigated by comparing
the PSD values of D$_1$ dust determined by using SEM method to the PSD values of dust from Group A
and Group C pellets from planetary mill. Since the conditions of experiments are different in both
experimental setups, the dust generated after 4 min of rotation in a planetary mill and 10 min in pellet
bed setup were selected for a comparison. The selection was done considering the same extent of wear
\((-6-8\%\) from initial weight of pellets) in both experimental setups.
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Fig. 3.10. Variation in $N_W$ values for (a) small, (b) medium and (c) large sized particles in D$_1$ dust due to varied applied loads.

A comparison of the PSDs (SEM method) of D$_1$ dusts produced at 1 and 3 kg applied loads to the PSD values of Group A and Group C is presented in Fig 3.11 (a) and (b), respectively. It can be seen in both figures that the $N_W$ values of coarse particles ($L_{\text{max}}>20 \mu m$) are ~40 to 70% higher for Group C pellets as compared to the other PSD values. Whereas, the number of fine particles ($L_{\text{max}} \leq 10 \mu m$) is ~13-40% higher for a 3 kg load as compared to the other PSD values. It should be noticed that some amount of fine particles in the pellet bed setup has been removed along with injected air and analyzed by the particle counter. Therefore, the number of fine particles for the trials using a 3 kg load is considerably higher than that of other PSD values. The higher $N_W$ values of fine particles and lower values of coarse particles in the pellet bed setup in comparison to those in the planetary mill suggest that abrasion is the dominating mechanism of dust generation in the pellet bed setup, due to the presence of an externally applied load. Moreover, an increase in $N_W$ values of fine particles with increasing applied load indicate that higher applied loads lead to increased contributions of an abrasion wear. It is important to mention that the applied loads in the pellet bed experiments (1-3 kg, which corresponds to 0.98-2.94 daN) are much lower compared to the crushing strength of the investigated pellets (220 daN). This very high applied loads might crush the pellets and generate the coarse particles found in the dust.
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**Fig. 3.11.** Comparison of PSDs of D$_1$ dust generated under (a) 1 kg and (b) 3 kg of applied load and PSDs of dust (obtained using planetary mill) of Group A and Group C pellets obtained by using the SEM method.

**Effect of applied load on D$_{2PA}$ dust**

The background air before conducting the experiments was analyzed in order to eliminate its influence on the PSD values of the generated dust obtained by ELPI+ particle analyzer. **Fig. 3.12** shows the PSD values of the background air and dust analyzed at the 1st and 10th minute of the experiment using a 3 kg applied load. It can be seen that the $N_V$ values of particles up to 0.1 μm are similar for all the presented PSD values. However, the background air doesn’t contain any considerable amount (~ 0.1% of total $N_V$) of particles >0.1 μm. This indicates that the most particles up to a size of 0.1 μm correspond to particles present in the background air. Therefore, they were not considered in the evaluation of the PSD values of the generated dust during the experiments.

**Fig. 3.12.** A Comparison of background air level and particle size distributions of D$_{2PA}$ dust obtained by ELPI+

The influence of varied applied loads on the PSD for D$_{2PA}$ dust can be seen in **Fig 3.13**, where the analyses of dust generated during the 10th minute ($i = 10$) of experiments. Here the results are
reported in terms of $N_W$ values, which have been recalculated from $N_V$ values measured by the particle analyzer. It can be seen in Fig. 3.13 that the total $N_W(10)$ values obtained for 2 and 3 kg applied loads are almost 27% higher than that for a 1 kg load. This indicates that a larger number of fine particles is generated under a higher applied load, as was also observed by LD and SEM methods for D$_1$ dust. Hence, supporting the conclusion that the application of higher applied load adds to the abrasive wear of pellets.

![Fig. 3.13. Effect of the applied load on the total $N_W$ value of D$_{2PA}$ dust measured by ELIP+ during 10th minutes of rotation (i=10).](image)

The amount of measured D$_{2PA}$ dust was calculated to be ~3.7-5.3 wt% of the total weight of the D$_0$ dust and 12-17 wt% of D$_0$ dust in the size range of 1-4.5 μm (assuming D$_{2L}$ ~ 0) for varied applied loads. These low values can be attributed to the resistance of the pellet bed and the upper loading plate, a low air velocity and a plate-like morphology of dust particles. Assuming that the pellet bed contains pellets having an average diameter of 12.25 mm and weight 3g, it was found that free area (holes) on a horizontal section for the dust particles to exit the bed is about 11% of the total horizontal area. Moreover, this value of the free area may vary due to movements of pellets in the bed during an experiment. Further, the free area of holes in the upper loading plate is only ~9.5% of the total area of this plate. Hence, the resistance faced by dust particles to be captured by the particle analyzer was very high. The influence of air velocity and morphology of dust particles on the uplift of the particles is discussed in the section 3.1.5.2.

### 3.1.4. Influence of partial reduction of pellets on the dust generation

A planetary mill was used for a dust generation from partially reduced pellets. As has been shown earlier, the test conditions in planetary mill can result in different wear rates for different sized pellets. Therefore, pellets from Group A were selected for these investigations. Moreover, after the reduction the pellets from the bottom zone of the sample holder were used for further investigations, since at different zones of the furnace a varied reduction degree of iron ore is achieved. For the investigated pellets the achieved reduction degrees were $RD_{500}$$\sim$16% and $RD_{850}$$\sim$50% for the pellets reduced at 500 and 850 °C, respectively.
3.1.4.1. Characteristics of reduced pellets

The measured density and hardness of pellets reduced at 500 °C (P500) and 850 °C (P850) are presented in Table 3.1. Moreover, these values for the reference pellets (unreduced pellets, denoted as P25) are also given. A decrease of ~9.5% in the densities of P500 and an increase of up to 16.3% for P850 pellets, in comparison to P25 pellets, was observed. The change in the density values is associated with the phase transformations and structural changes, which take place during reduction of iron ore. During reduction at 500 °C the hematite (Fe₂O₃) is reduced partially or completely to magnetite (Fe₃O₄) having a lower density (i.e. 5000 kg/m³) as compared to hematite(5200 kg/m³).

Upon a further reduction at 850 °C, magnetite reduces to high density FeO and (partially up to) metallic Fe phases, 5700 kg/m³ for FeO and 7800 kg/m³ for Fe. Hence, a decrease in the density of pellets is observed during reduction at 500 °C which is followed by an increase upon further reduction at 850 °C. Moreover, the reduction of hematite particles to magnetite and to FeO and Fe is accompanied by up ~26% volume expansion, which also influences the density of a pellet.

Table 3.1. Density and hardness values of reference and reduced pellets.

<table>
<thead>
<tr>
<th>Pellets</th>
<th>Density (g/cm³)</th>
<th>Hardness, HV</th>
<th>Center (5-6mm from surface)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P25</td>
<td>3.83±0.04</td>
<td>369 ±36</td>
<td>115±13</td>
</tr>
<tr>
<td>P500</td>
<td>3.47±0.12</td>
<td>186±11</td>
<td>109±13</td>
</tr>
<tr>
<td>P850</td>
<td>4.10±0.07</td>
<td>121±11</td>
<td>100±28</td>
</tr>
</tbody>
</table>

A decrease in the hardness (HV) of pellets was observed after reduction at temperatures of 500 and 850 °C. Especially, the HV values of the outer surface layers (0-1.5 mm) of the P500 and P850 pellets drastically decreased (up to 50-67%) compared to that of the reference P25 pellets. This behavior can be clarified based upon the fact that the pure high-density magnetite and FeO have almost 50% less hardness than hematite. It should be pointed out that a sharp decrease (47-55%) in the HV values of P25 and P500 pellets was observed for 0 up to 2 mm along the depth from the surface to center, thereafter, the HV values decrease slightly. However, the hardness values of P850 pellets in all zones are almost similar.

3.1.4.2. Wear Rate

The obtained result for the wear of the pellets reduced at 500 and 850 °C are shown as a function of the rotation time in Fig. 3.14 and compared to those of reference pellets (which are previously presented in Fig. 3.6). An increase of 16 to 35% in the wear rate can be observed for P 500 pellets as compared to P25 pellets. The WR values of P25 and P500 pellets follow the same tendency i.e. the wear rate increases during the first 12-14 min of rotation. Thereafter the values become almost constant. However, the WR values of the pellets reduced at 850 °C (P850), which are significantly lower than P25 pellets, remain almost constant up to 20 min and tends to slightly increase towards the end of rotation time.
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Fig. 3.14. Comparison of the wear rate of reference (P25) and reduced pellets at 500 °C (P500) and 850° C (P850).

Similar variations in the wear rate of iron ore due to reduction have been reported by several researchers. \( \text{6,7,24-33} \) Especially, a significant increase in the disintegration of iron ore in the temperature range of 400-600 °C is well known. This takes place due to a 15 to 25% volume expansion of hematite to magnetite, which is associated with the structural transformation of a hexagonal hematite to a cubic magnetite. \( \text{6,7,12,24-33,78} \) This expansion leads to internal stresses around the reduction region, which are relieved through the formation of cracks that result in the disintegration of iron ore.

In addition, the carbon deposition also contributes to a disintegration of iron ore during a reduction in the temperature range of 400-600 °C. \( \text{24, 78}\) The rearrangement of oxygen ions during the reduction of hematite to magnetite can also cause unequal volume changes in different directions of produced magnetite grains, leading to crack formation. \( \text{78} \)

Vyver et al. \( \text{7}\) reported that the degradation of iron ore decreases significantly for the temperatures > 750 °C. Similarly, a decrease (~19%) in the degradation of lump ore was observed by an increase in reduction temperature from 550 °C to 650 °C and corresponding to a 25% reduction degree. \( \text{24} \) Li et al. \( \text{27}\) has reported similar tendency for iron ore pellets. For an increasing temperature from 550 to 650 °C, a decrease of ~30% in the degradation of iron ore pellets occurred. In the aforementioned studies \( \text{7,24,27}\) it is reasoned that the decrease in degradation at higher reduction temperatures due to the increase in plasticity of iron ore. This mean that the ability of iron ore to withstand the volume expansion and breakage due to stress concentration increases. This in turn, leads to a decreased disintegration.

Fig. 3.15 presents the surfaces (outer layers) of reference and reduced pellets on polished cross sections at different magnifications. The composition of the observed phases labeled on Fig. 3.15 are presented in Table 3.2. It can be seen in Fig. 3.15 (a) and (b) that the reference pellets contain hematite, which has been reduced to magnetite during a reduction at 500 °C. The formed magnetite grains contains small sized pores, see Fig. 3.15 (c) and (d). The formation of this porous magnetite
has been reported and justified by the removal of oxygen from the surface of magnetite. The presence of inter-grain pores can promote the propagation of the cracks during the disintegration/wear of pellets. Another possible reason of a higher wear rate can be low HV value of pellets reduced at 500 °C, as shown in Table 3.1.

Fig. 3.15. Microstructures of (a, b) reference P25 pellet and pellets reduced at (c, d) 500 °C (P500) and (e, f) 850 °C (P850).
Table 3.2. Compositions of the phases marked on Fig. 3.15.

<table>
<thead>
<tr>
<th>Pellets</th>
<th>Phase</th>
<th>Content (wt%)</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>P25</td>
<td>1</td>
<td>69.6 – 70.3</td>
<td>FeO</td>
</tr>
<tr>
<td>P500</td>
<td>2</td>
<td>74.1 – 75.6</td>
<td>Fe₂O₃</td>
</tr>
<tr>
<td>P850</td>
<td>3</td>
<td>72.1 – 74.9</td>
<td>Fe₂O₄</td>
</tr>
<tr>
<td>P850</td>
<td>4</td>
<td>100</td>
<td>FeO</td>
</tr>
</tbody>
</table>

On the contrary, despite having lowest hardness (133-100HV), the P850 pellets have very low wear rates. This phenomenon is caused by the microstructure attained during reduction at 850 °C. For the P850 pellets, a relatively compact microstructure was observed at the outer surface as can be seen in Fig. 3.15 (e) and (f). Moreover, a metallic iron phase is formed and sintered around the particles at the surface resulting in a significant decrease in the porosity. The presence of this sintered metallic iron layer is the main reason for the significantly lower wear rate of the P850 pellets in comparison to the other pellets. Huang et al. [12] also reported an increase in the strength of iron ore pellets with an increased degree of the reduction due to the sintering of newly formed metallic iron. Moreover, the surfaces of the P25 and P500 pellets contain loosely bonded particles. Hence, they have a much higher wear rate compared to the P850 pellets.

3.1.4.3. Evaluation of generated dust

The SEM method was used to obtain the PSD values of the dusts generated due to wear of the pellets reduced at 500 and 850 °C. Fig. 3.16 represents the variation in the $N_W$ values of different sized particles observed in the dust collected after 4 and 14 min of rotation for partially reduced as well for reference pellets (previously shown in Fig. 3.6). It can be seen in Fig. 3.16 (a) that the $N_W$ values of fine particles for the P500 and P850 pellets are 10 to 56 % lower than the $N_W$ value of the P25 pellets. However, the medium sized particles doesn’t exhibit a clear tendency (Fig. 3.16 (b)). On contrary, the $N_W$ values of coarse particles are significantly higher (3-6 times) for reduced pellets, as can be seen in Fig. 3.16 (c).

![Fig. 3.16. Variation in $N_W$ values for (a) fine, (b) medium and (c) coarse particles depending on the reduction temperature and sampling time.](image)

Since the pellets from Group A were selected for partial reduction and a fixed weight of charge was used for wear in the planetary mill, the variation in the $N_W$ values due to different wear...
mechanisms can be ruled out. It can be assumed that this variation in $N_W$ values is related to the significantly higher hardness of P25 pellets compared to P500 and P850 pellets. Therefore, the reduced pellets can generate more coarse dust particles compared to reference pellets. A similar tendency was observed in Fig. 3.6, i.e. a 60 to 80% increase in the $N_W$ values of coarse particles for pellets of Group A and C due to removal of hard outer layer with increased rotation time from 4 min (HV > 300) to 14 min (HV ~200-300). Further, the $N_W$ values of coarse particles (for Group A pellets) increases up to ~4.5 times for the softer core i.e. after 24 min (HV < 200) rotation time.

3.1.5. Industrial aspects

3.1.5.1. Transportation and handling of iron ore pellets

Based upon the investigations of the dust generated during the laboratory wear experiments of iron ore pellets, the wear mechanisms and their influence on the PSD values of the dust has been determined. This knowledge can be useful for the iron making industry with respect to a minimization of the dust generation at different stages of the process. For instance, Fig. 3.17 presents a schematic illustration which identifies the dominant mechanisms of wear at different stages of the iron making process. It indicates that during loading, unloading and charging of the pellets, collisions can have dominating role in dust generation as compared to sliding. Whereas, the sliding wear is dominant during transport of pellets through vehicles and conveyor belts and descending of burden in the blast furnace. Moreover, the presence of an external load (in form of stacks of pellets) during these operation, which enhances the dust generation as well the abrasive wear, is an important factor to be considered. Further, the dust generated on conveyor belts contains larger amounts of fine particles is charged into the BF together with the pellets and can easily end up in BF flue gases.

![Fig. 3.17. Schematic illustration of different dominant wear mechanisms during different stages of the iron making process. $WR_{slid}$ and $WR_{col}$ represent the values of wear rates due to sliding and collisions of iron ore pellets, respectively.](image)

Although it is not easy to quantify the dust generation and contribution of each mechanism at the different stages of a process, the dust generation can be minimized by optimizing some parameters such as the height of drops, the length and speed of conveyor belts and off gas velocities etc. Further
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studies to estimate dust generation during each process step and optimization of process parameters is required.

3.1.5.2. Dust particles in blast furnace

The dust generated on conveyor belts and during charging the burden follows the pellets into the BF and can negatively influence the BF process or easily end up in BF flue gases. These dusts correspond to the unreduced pellets (25 °C), which have a lower wear rate compared to the pellets partially reduced to magnetite (500 °C). Whereas, the dust generated due to mechanical wear of partially reduced pellets also needs to be considered. Fig. 3.18(a) presents a schematic illustration of a blast furnace indicating the temperatures investigated and the corresponding wear rates and PSD values obtained for the wear of partially reduced pellets. The investigated reduction temperatures correspond to the shaft/ stack of a BF, where the charged material encounters the ascending reducing gases and acquires a temperature up to 800 – 900 °C. As illustrated in Fig. 3.18(b), in the shaft during the descend of charged material the wear rate of pellets tends to increase and reaches a maximum value around the temperature range of 500 °C, where hematite has been reduced to magnetite. Thereafter, as a further reduction taking place the wear rate of pellets tends to decrease. When the pellets reach the thermal reserve zone, having a temperature range of 800 – 1000 °C, the wear rate of pellets should be very low i.e. ~89% lower than that at 500 °C.

Leimalm et al. 21) mentioned that the Fe containing particles observed in off-gas dust of an experimental BF were unreduced hematite particles. Whereas, samples taken from the upper part of shaft (temperature ~ 650-750 °C) contained magnetite, FeO and Fe particles along with some hematite particles. This means that only the dust from unreduced pellets, either charged along with pellets or generated in the upper part of BF before being reduced, ends up in the off gases. Whereas the dust from the reduced pellets, which contains higher percentages of coarse particles as compared to dust of unreduced pellets (Fig. 3.18(c)), stays within the BF.
The size of particles which can exit the BF with the off-gases depends on the gas flow rate. A critical diameter of particles which can be removed by the off-gas at the given flow rate can be estimated by using the following relation:

$$U_t = \left[ \frac{4d_p(\rho_p-\rho_g)g}{3\rho_g c_D} \right]^{1/2}$$  \hspace{1cm} (3-2)

where $U_t$ is the terminal velocity of the gas flow (m/s), $d_p$ is the diameter of a particle (m), $\rho_p$ is the apparent density of particles (5200 kg/m$^3$, for hematite), $\rho_g$ is the density of off-gas from the BF (1.25 kg/m$^3$), $g$ is the acceleration of the gravity and $C_D$ is the coefficient of the drag force. The value of $C_D$ was determined depending on the value of the Reynolds number of particle ($Re_p$) as follows: $C_D = 24/Re_p$ for $0.4 < Re_p < 500$ and $C_D = 0.43$ for $500 < Re_p < 200000$.

Assuming that the velocity of dust particles ($U_p$) is same as that of the off-gas at the top of BF ($U_p = U_t$), the $Re_p$ value can be calculated as follows:

$$Re_p = \frac{U_t \rho_g d_p}{\mu_g}$$  \hspace{1cm} (3-3)

where $\mu_g$ is the gas viscosity (1.8×10$^{-5}$ kg/m·s). It was found that the $Re_p$ values varied from 0.4 to 500 for the gas velocities of 0.3 to 9.6 m/s and particle size in the range $21 < d_p \leq 752$ μm. Therefore, the value of $C_D = 10/(Re_p^{1/2})$ was used in Eq.(3-2). Based on Eq.(3-2) and Eq.(3-3), the critical diameter of dust particles, which can be removed with off-gas from the BF, can be calculated as follows:

$$d_p = U_t \left[ \frac{2(\rho_p-\rho_g)g}{15\mu_g} \cdot \frac{\rho_g}{\mu_g} \right]^{2/3}$$  \hspace{1cm} (3-4)

**Fig. 3.19** shows a relationship between the off-gas velocity and the critical diameter of dust particles obtained by using Eq. (3-4). At the upper part of BF, the off-gas velocities can vary from ~1 m/s towards the wall to ~7 m/s at the centre of a radial section. Also, it can be seen in Fig. 10 that at the off-gas velocities of 1 m/s and 7 m/s the dust particles having the size smaller than ~78 and ~550 μm can be removed from the BF. This indicates that higher off gas velocities will result in substantially higher amounts of off-gas dust in a BF. The relationship between the critical diameter of dust particles and the velocity of off-gas in the top part of the BF can be described at the given conditions using the following linear function:

$$d_p = 7.87 \cdot 10^{-5} \cdot U_t$$  \hspace{1cm} (3-5)
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Fig. 3.19. Relationship between the off-gas velocity and the critical size of dust particles which can be removed from the top of a blast furnace along with the off-gases.

The analysis of dust generated during the laboratory wear experiments of unreduced and partially reduced pellets showed that most of the dust particles have sizes smaller than the critical diameter (78 μm) for off-gas velocity of 1 m/s. This suggests that the most dust particles generated due to mechanical wear of pellets can be easily be transported out from the furnace with BF off-gases. However, the morphology of dust particles and their orientation in a gas stream can significantly influence their flow. As mentioned earlier, the generated dust particles have mostly a flake or a plate shape. During an uplifting of the particles, the area of particles perpendicular to air flow (projected area) is of more importance as it defines the drag force which shall act on the particles to be lifted.

For a particle of a known mass \( m \) and projected area \( A_p \), Eq. (3-2) can be rewritten as follows:

\[
U_t = \left[ \frac{2mg(\rho_p - \rho_a)}{\rho_a \rho_p \rho_g C_D A_p} \right]^{1/2} \tag{3-6}
\]

From the above equation it is apparent that a lower gas velocity is required for a particle having a larger \( A_p \) value among those having a constant weight/volume. This suggests that the orientation of dust particles in the air flow can significantly influence their uplifting tendency. Fig. 3.20 shows the variations of the required gas velocity for the uplifting of a fine \( (d_{eq} \leq 5 \mu m) \) and a coarse \( (d_{eq} > 10 \mu m) \) observed plate-like dust particles, which have different orientations (projected area) in the gas flow. The gas velocities are calculated for the experimental conditions of the pellet bed setup i.e. \( U_t = 0.013 \) m/s and \( \rho_g = 1.29 \text{ kg/m}^3 \) for air. Moreover, the \( Re_p \) values were obtained assuming that the velocity of dust particles is same as that of the air. The dimensions (length, width and thickness) and different projected areas depending on the orientations \( (A_{p1}, A_{p2} \text{ and } A_{p3}) \) and measured by using ImageJ \( (A_{p(IJ)}) \) are given in Table 3.3. It is noteworthy that the required gas velocity for uplifting of a plate-like particle can vary by 22 to 30\%, depending on the dimensions of this dust particle and its orientation in the air flow. Especially, this variation is higher for coarse particle. It should be noted that the real thickness \( (z) \) of the large size dust particles can be significantly lower than the assumed thickness i.e. half of its width. In this case, the required air velocity for the uplifting of such particles
should be significantly larger. Hence, the influence of the orientation and the morphology of particles is of great importance for flow of particles, especially for the large sized particles.

![Fig. 3.20.](image1)

![Fig. 3.20.](image2)

**Fig. 3.20.** Variation in the required gas velocity for the uplifting of a) a small sized and b) a large sized particle having different orientations and projected area ($A_p$) in the gas flow.

<table>
<thead>
<tr>
<th>Dust particle</th>
<th>Length, $x$ (µm)</th>
<th>Width, $y$ (µm)</th>
<th>Thickness, $z=y/2$ (µm)</th>
<th>$A_{p1} = x \cdot y$ (µm$^2$)</th>
<th>$A_{p2} = x \cdot z$ (µm$^2$)</th>
<th>$A_{p3} = y \cdot z$ (µm$^2$)</th>
<th>$A_{p(IJ)}$ (µm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Small</td>
<td>2.3</td>
<td>1.6</td>
<td>0.8</td>
<td>3.7</td>
<td>1.8</td>
<td>1.3</td>
<td>3.2</td>
</tr>
<tr>
<td>Large</td>
<td>32.6</td>
<td>23.5</td>
<td>11.8</td>
<td>766.1</td>
<td>383.1</td>
<td>276.1</td>
<td>506.2</td>
</tr>
</tbody>
</table>

### 3.2. Clusters in REM alloyed stainless steel

Firstly, it is to be mentioned that the agglomerations consisted of three and more inclusions have been considered as a cluster. Some typical small and large clusters, which were observed on a surface of film filter after electrolytic extraction, are shown in **Fig. 3.21.** For different samples around 400 to 1100 extracted clusters were observed on a surface of film filter to determine the characteristics such as the number, size and morphology of the clusters.
3.2.1. Obtaining a reliable cluster size distribution (CSD)

The measured maximum lengths ($L_C$) of clusters were plotted against $N_V$, with a constant size step ($\Delta L_C = 1 \mu m$) to obtain cluster size distributions (CSD) for each sample. A typical cluster size distribution (CSD) obtained by observing 147 clusters on 3 mm$^2$ on film filter of the sample S3 is shown in Fig. 3.22(a). In addition, Fig. 3.22(b) presents the same CSD in terms of $\log(Wt_C)$, where $Wt_C$ is the weight of the steel sample containing only one cluster in the given size range. The value of $Wt_C$ can be calculated using $N_V$ value as follows:

$$Wt_C = \frac{\delta m}{N_V}$$

Fig. 3.22. Typical size distribution of clusters (a) and the weight of steel sample ($Wt_C$) containing only one cluster in the given size range (b) in sample S3.

In other words, the $Wt_C$ value represents the weight of steel sample which must be dissolved to find one cluster in a given size range. As can be seen in Fig. 3.22(b), the CSD in terms of $\log(Wt_C)$ can be well described by a linear function ($R = 0.961$) for the size range from 4 to 10 $\mu m$. In this case, the small sized clusters ($<4 \mu m$) are not considered for determination of this linear function, hereafter
called “CSD function line”. However, the data points for clusters larger than 10 µm are very scattered and do not correspond to the obtained CSD function line. The possible reasons for this big deviation can be: 1) less number of investigated large size clusters (>10 µm) due to a limited observed area of the film filter, 2) difference in the composition of these clusters and 3) different formation and growth mechanism of clusters in different size ranges.

The CSD function line was selected as a decisive way for investigating reliability of a CSD due to its ease in determining whether a data point follow the linear function or not. Moreover, the above mentioned possible reasons for deviation observed in Fig. 3.22(b) were systematically investigated.

3.2.1.1. Increasing the observed area

Based on the results obtained by an estimation of largest size of inclusions in rolled steels \(^6\) \(^1\), it was found that the linear distribution of SEV (statistics of extreme value) can be significantly improved with an increased number of measurements, especially in the size ranges of large size inclusions. Therefore, the observed area on a filter was increased from 3 mm\(^2\) \((A_1)\) to 6 \((2A_1)\), 9 \((3A_1)\), 12 \((4A_1)\) and 15 \((5A_1)\) mm\(^2\) according to Method 1 (observing all clusters) to increase the number of observed clusters. The variation in the obtained CSD function lines for increased observed area is shown in Fig. 3.23(a) for the S3 sample. It was found that the linearity of the CSD function lines improves with increasing the observed area 3 \((A_1)\) to 9 mm\(^2\) \((3A_1)\). However, there was no significant change in the slope of the function line by increasing the observed area from 9 \((3A_1)\) to 15 mm\(^2\) \((5A_1)\). The \(5A_1\) function line was used as a reference CSD function line in order to determine an appropriate number of clusters to be investigated in a size step for obtaining a reliable CSD. The deviation of \(\log(W_{tC(i\cdot A_1)})\) value for \(i\)-th CSD function line was calculated in percentage as follows:

\[
\Delta \log(W_{tC(i\cdot A_1)}) = 100\% \cdot \frac{|\log(W_{tC(i\cdot A_1)}) - \log(W_{tC(5A_1)})|}{\log(W_{tC(5A_1)})}
\]  

(a)

(b)

Fig. 3.23. (a) CSD function lines for different observed areas for sample S3 and (b) the effect of the cluster number per size step on the deviation from the \(5A_1\) reference function line.

It was observed that the deviation of the \(\log(W_{tC(i\cdot A_1)})\) values decreases drastically with an increased number of observed cluster per size step and for 5 or more observed clusters the deviation...
was less than 3%, see Fig. 3.23(b). The error bars represent the arithmetic standard deviation of obtained values in the respective size step. Based upon these results it was concluded that (for REM clusters in current study) a reliable CSD function line can be attained by having at least 5 clusters per size step.

![Graph showing CSD function lines](image)

**Fig. 3.24.** CSD function lines of different samples obtained by observation of clusters on a 15 mm² of film filter (Method 1).

**Fig. 3.24** shows the CSD values (in term of \( \log(Wt_C) \)) for all three samples obtained by observation of clusters on 15 mm² of filters (5A). On the same figure the CSD function lines, obtained by considering the data points with a minimum of 5 measured clusters per size step, are also presented. It can be seen that several data points containing less than 5 clusters (open marks) significantly deviate from the obtained CSD function lines. In order to improve the CSD representing larger sized clusters, Method 2 was adopted to increase the number of those clusters.

As expected an increase of the observed area from 15 mm² to 30 mm² resulted in a decrease in the deviation of the \( \log(Wt_C) \) values for larger clusters (≥ 15 µm) from the 5A, reference function line, as shown in **Fig. 3.25(a)** for S3 sample. Interestingly, no significant decrease in this deviation was observed for larger size clusters by further increasing of the observed area from 30 to 75 mm², despite having measured more than 5 clusters in these size ranges (from 5 to 23). **Fig. 3.25(b)** shows the CSD function lines for all three samples obtained at increased observed areas (Method 2) and measuring at least 5 clusters in each size step. **Table 3.4** summarizes the total observed area and the number of measured clusters on the film filter for each sample. It was found that the data points representing larger size clusters (open marks) for samples S2 and S3 didn’t follow function line (closed marks). Instead, they tend to lie on new function lines (FL2). However, this bilinear behavior was not observed for sample S1 due to the absence of large sized clusters. The equations of the CSD function lines for small (FL1) and large (FL2) sized clusters and their correlation coefficient (R) are given in **Table 3.5**.
A study of micro-particles in the dust and melt at different stages of iron and steelmaking

Fig. 3.25.  (a) Relationships between the observed area and deviations of the $\log(Wt_C)$ values for larger size clusters from a $5A$, reference function line and (b) CSD function lines for different samples obtained at increased observed areas using Method 2.

Table 3.4. Observed area and total number of measured cluster in different samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Observed area (mm²)</th>
<th>Total number of measured clusters</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>15</td>
<td>399</td>
</tr>
<tr>
<td>S2</td>
<td>15</td>
<td>669</td>
</tr>
<tr>
<td>S3</td>
<td>15</td>
<td>1087</td>
</tr>
</tbody>
</table>

Table 3.5. Equations of obtained CSD functions lines for different samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>CSD function line 1 (FL1)</th>
<th>R</th>
<th>CSD function line 2 (FL2)</th>
<th>R</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>$y = 0.352 \cdot x - 5.986$</td>
<td>0.999</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>S2</td>
<td>$y = 0.273 \cdot x - 5.936$</td>
<td>0.989</td>
<td>$y = 0.196 \cdot x - 5.394$</td>
<td>0.982</td>
</tr>
<tr>
<td>S3</td>
<td>$y = 0.154 \cdot x - 5.504$</td>
<td>0.996</td>
<td>$y = 0.073 \cdot x - 4.446$</td>
<td>0.982</td>
</tr>
</tbody>
</table>

The bilinear representation of cluster distributions in samples S2 and S3 indicates the presence of two different types of clusters. According to Bretta and Murakami (58) and Kanbe et al. (61) has reported that the inclusions having different compositions show a bilinear representation on the probability plot for SEV analysis. Therefore, the composition of inclusions in the clusters in all size ranges was analyzed and the compositions were found to be very similar for all the analyzed clusters. For instance, the clusters corresponding to the FL1 line contain on average 59.4% La and 33.1% Ce and the clusters corresponding to the FL2 line contain on average 58.8% La and 32.8% Ce. The remaining parts of the compositions for inclusions in clusters were Nd and Pr in both cases. Therefore, it can safely be assumed that the observed clusters have the same source and formation mechanisms.
Based upon the results reported by Zhou et al. (88), i.e. inclusions having the same composition but different 3D structure show different distributions, the morphology of clusters was investigated. It was found that the morphology of inclusions in the investigated clusters was very similar. However, the circularity factor (CF) of clusters, representation the cluster morphology, significantly decreases with an increased length of the clusters. Moreover, it was observed (for all samples) that the average CF values for clusters on FL1 lines are significantly higher (on average ~0.28-0.33) than those for large size clusters on FL2 lines (on average ~ 0.1). This indicates that the clusters representing CSD FL1 and FL2 lines might have different growth mechanisms, hence, different morphologies. Therefore, the value of CF=0.15 was selected as a decisive value for quantitative separation of observed clusters into two different groups, small size clusters (CF≥ 0.15 ) and large sized clusters (CF< 0.15 ) which correspond to the FL1 and FL2 lines, respectively.

3.2.2. Mechanism of formation and growth of clusters

The interesting finding of bilinear representation required for CSD function lines of REM clusters was used to elucidate the formation and growth mechanism of clusters in the liquid steel, which can be divided into the following steps: Step 1- formation of small clusters due to collision of separate inclusions; Step 2 – growth of cluster by collision with separate inclusions (Mechanism 1) and Step 3 – growth of cluster by collision with other clusters (Mechanism 2).

The formation and growth of clusters is dependent on the number of collisions, where the collision rate can be calculated according to following relationship: (89)

$$\frac{dn_{ij}}{dt} = \beta_{ij} n_i n_j$$

where $\beta_{ij}$ is the collision volume ($m^3/s$), $n_i$ and $n_j$ are the number densities ($m^{-3}$) of inclusions/clusters of certain size ranges and $t$ is time (s). Three main types of collisions occur during interaction of inclusions/clusters, i.e. i) Brownian collisions ($\beta_{ij}^B$) due to random movement of inclusions in molten steel, ii) Stokes’ collisions ($\beta_{ij}^S$) due to flotation of inclusions/clusters having lower density than liquid steel and iii) turbulent collisions ($\beta_{ij}^T$), which take place due to movement of inclusions/clusters along with molten steel flow. The collision volume for each type can be expressed as follows: (42,43)

$$\beta_{ij}^B = \frac{2kT (r_i+r_j)^2}{3\mu r_i r_j}$$

$$\beta_{ij}^S = \frac{2g(\rho_i-\rho_{ox})}{9\mu} (r_i + r_j)^3 |r_i - r_j|$$

$$\beta_{ij}^T = 1.3\alpha \sqrt{\frac{\pi \rho_f \varepsilon}{\mu}} (r_i + r_j)^3$$

where $k$ is Boltzman constant (J/K), $T$ is temperature (K), $\mu$ is the dynamic viscosity of steel (kg/ms), $g$ is gravitational acceleration (m/s$^2$), $\rho_f$ and $\rho_{ox}$ are the densities of the steel and oxide particle respectively (kg/m$^3$), $\alpha$ is the collision efficiency, $\varepsilon$ is the turbulent energy dissipation (m$^2$/s$^3$) and $r_i$ and $r_j$ are the radii of the two colliding inclusions/clusters.

All three types of collisions contribute to the total number of collisions that occur, which have different contributions to the total value. To estimate the magnitude of these types of collisions, the
\[ \beta_{ij} \]

\( \beta_{ij} \) values of each type of collisions were calculated for an inclusion having a diameter of 1 μm \( r_i = 0.5 \) μm that collides with other inclusions, by using the parameters given in Table 3.6. The obtained results are presented in Fig. 3.26. It can be seen that the magnitude of \( \beta_{ij}^{h} \) and \( \beta_{ij}^{s} \) are significantly lower than that of \( \beta_{ij}^{t} \) over all studied ranges of \( r_j \). The Stokes’ collisions have no significant influence for small inclusions (up to 1μm) whereas the Brownian collisions have some effect on the collision volume of small size particles. For large size inclusions/clusters \( (2r_j = 2-50 \) μm), the turbulent collisions have much higher contribution \( (24-590 \) times) to the total volume of collisions as compared to the Stokes’ collisions leading to significant impact on the growth rate of clusters.

### Table 3.6. Data used in the calculations of the collision volumes.

<table>
<thead>
<tr>
<th>( \alpha )</th>
<th>( \varepsilon ) (m²/s³)</th>
<th>( \mu ) (kg/m·s)</th>
<th>( \rho_f ) (kg/m³)</th>
<th>( \rho_{ox} ) (kg/m³)</th>
<th>( T ) (K)</th>
<th>( k ) (J/K)</th>
<th>( g ) (m/s²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3</td>
<td>0.01</td>
<td>0.005</td>
<td>7000</td>
<td>6900</td>
<td>1923</td>
<td>1.38×10⁻²³</td>
<td>9.81</td>
</tr>
</tbody>
</table>

Fig. 3.26. Collision volume for an inclusion with a diameter of 1μm \( r_i = 0.5\mu m \) that collides with other inclusions of different sizes.

Based upon the above mentioned results the suggested mechanism of formation and growth of REM-oxide clusters can be correlated to the experimental results as follows. During Step 1 clusters are formed in the liquid steel due to Brownian and turbulent collisions. This is followed by growth of clusters (Step 2) in accordance to Mechanism 1, which is governed by turbulent collisions. The resulting small sized clusters \( (\text{CF} \geq 0.15) \) correspond to the FL1 line. Whereas, the large sized clusters \( (\text{CF} < 0.15) \) grow during the Step 3, where Mechanism 2 due to turbulent collisions and partially by Stokes’ collisions takes place. These large sized clusters are represented by the FL2 line.

### 3.2.2.1. CSD and growth rate of clusters

The obtained CSD values for different samples were used to investigate the growth rate of clusters, which is proportional to the rate of the number of collisions (“collision rate”). Moreover, the collision rate depends on the collisions volume \( (\beta_{ij}) \) and numbers of clusters \( (n_i \) and \( n_j) \).
Calculating collision volume of clusters

It is of importance that the equations of collision volumes are derived considering spherical particles whereas clusters have irregular shape. This means that the use of the equivalent diameter \((d_{eq} = \sqrt{4A_c/\pi})\) of clusters can significantly underestimate the calculated collision volume. This is due to that the irregular cluster has a significantly larger surface area in comparison to its equivalent spherical cluster. For instance, it was found that for a cluster having \(L_C = 25.2 \, \mu m\) and \(d_{eq} = 11.8 \, \mu m\), the \(\beta_{ij}\) values calculated using \(L_C\) were 2–3 times larger than those using \(d_{eq}\). Under the assumption that a cluster in liquid steel can interact with inclusions/clusters within its rotational region, whose maximum diameter equals to the maximum length of this cluster, \(L_C\) of clusters was used to calculate the maximum possible collision volume of inclusions/clusters in the liquid steel.

Growth rate for different samples

The CSD values in different samples obtained by Method 1 + Method 2 are shown in Fig. 3.27(a). The total collision rate \((= \sum \frac{dn_{ij}}{dt})\) of clusters corresponding to the FL1 and FL2 lines were calculated by using Eq. (3-9) and shown in Fig. 3.27(b). The values of \(r_i\) and \(r_j\) for different samples were determined from the obtained cluster size distributions and the results are given in Fig. 3.27(b). The \(r_i\) values correspond to the CSD peak values and \(r_j\) values refer to the cluster sizes observed for each sample. Moreover, the \(n_i\) and \(n_j\) values correspond to the \(N_V\) values for clusters of \(i\) and \(j\) size ranges. An increase in the total collisions rate of clusters with increasing holding time is observed for both the FL1 and FL2 lines. Moreover, this increase is substantially higher for the clusters on FL2 line as compared to the FL1 line. For instance, it can be seen that the total rate of collisions for clusters on FL1 is almost 2 times higher for sample S3 (9 min of holding) than that of sample S2 (6 min of holding), whereas the corresponding value for clusters on FL2 is more than 5 times larger. Therefore, it can be concluded that after a 6 minutes holding time the large size clusters represented by the FL2 have a much higher growth rate than the small clusters which are described by the FL1 line.

![Fig. 3.27](image-url)

**Fig. 3.27.** (a) Cluster size distribution (CSD) of all the three samples obtained at increased observed areas by using Method 2, and (b) the total rate of collisions for all three samples calculated for selective values of \(r_i\).
3.2.3. Extreme value distribution of REM clusters

For EVD analysis, the data obtained for REM clusters by observing a 15 mm$^2$ area on film filter (Method 1) were used. The data of Method 2 were not selected, since small sized clusters were not observed by using this method. The typical cluster size distributions (CSDs) obtained by observing clusters on 15 mm$^2$ on film filter for all three samples are shown in Fig. 3.28 in terms of $N_V$ with a constant size step ($\Delta L_C=1 \mu m$).

![Fig. 3.28](image)

**Fig. 3.28.** CSD of all samples obtained by observation of clusters at 15mm$^2$ area on a film filter.

3.2.3.1. Problem of blank fields

Due to a low number of clusters in comparison to abundantly found single inclusions in the steel melt, for EVD analysis fields without any clusters can be expected. A similar problem was met in the current study. For instance, for sample S3, out of 330 observed fields, 290 contained clusters whereas the remaining 40 fields didn’t have any clusters (hereafter referred as ‘blank fields’ in this study). In order to resolve this problem, 4 different cases (enlisted in Table 3.7) were considered in the EVD analysis of sample S3. According to ASTM E2283-03, $L_C$ was used as a size parameter in the EVD analysis. For Case 1, only the fields containing clusters are used to calculate the reduce variate ($y$), i.e. $n = 290$ in Eq. 2-8. Whereas, in Case 2 all the observed fields, i.e. containing clusters and blank fields ($290 + 40$), are used in the analysis. The blank fields are represented by zeros indicating that cluster with $L_C = 0$ was observed in that field and used in ranking the measured cluster sizes in range of 1 – 330. The correlation coefficient of EVD regression lines can be improved by increasing the number of inclusions on a unit area by increasing the unit area. Therefore, for Case 3 and 4, the unit area for a field was increased from $A_0$ to $2A_0$ and $4A_0$. 
Table 3.7. Parameters used for EVD analysis of sample S3 for four different cases.

<table>
<thead>
<tr>
<th>Case</th>
<th>Unit area, $A_O$ (mm²)</th>
<th>Total observed unit areas</th>
<th>Number of used unit areas, $n$</th>
<th>Number of maximum sized clusters</th>
<th>Number of blank fields</th>
<th>Range of used ranks ($i$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.045</td>
<td>330</td>
<td>290</td>
<td>290</td>
<td>40</td>
<td>1 – 290</td>
</tr>
<tr>
<td>2</td>
<td>0.045</td>
<td>330</td>
<td>330</td>
<td>290</td>
<td>40</td>
<td>1 – 330</td>
</tr>
<tr>
<td>3</td>
<td>0.090</td>
<td>165</td>
<td>165</td>
<td>165</td>
<td>–</td>
<td>1 – 165</td>
</tr>
<tr>
<td>4</td>
<td>0.180</td>
<td>82</td>
<td>82</td>
<td>82</td>
<td>–</td>
<td>1 – 82</td>
</tr>
</tbody>
</table>

The EVDs obtained for sample S3 in accordance to all 4 cases is presented in Fig. 3.29. It can be seen that for Case 1 clusters with $L_C > 15 \mu m$ deviate from the linear distribution, whereas, this deviation decreases when blank fields are taken into consideration (Case 2) and the $R^2$ value significantly improves from 0.9404 to 0.9744, as can be seen in Table 3.8. However, the change in the slope of Case 2 regression line is responsible for this decreased deviation. Where the presence of blank fields as zeros caused the change in slope. Case 3 exhibits almost similar $R^2$ value to that of Case 2. However, it is interesting to note that the blank fields are eliminated by increasing the unit area of observed fields from $A_O$ to $2A_O$. Moreover, when the unit area is further increased to $4A_O$ in Case 4 the linearity of the distribution is improved ($R^2 = 0.9876$).

Fig. 3.29. Comparison of EVDs obtained for three different cases applied for sample S3.
Table 3.8. Regression line equations and coefficient of determination, $R^2$, for EVDs obtained in accordance to four different cases.

<table>
<thead>
<tr>
<th>Case number</th>
<th>Regression line function</th>
<th>$R^2$ value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Case 1</td>
<td>$y = 0.2725x - 1.9053$</td>
<td>0.9404</td>
</tr>
<tr>
<td>Case 2</td>
<td>$y = 0.2162x - 1.1924$</td>
<td>0.9744</td>
</tr>
<tr>
<td>Case 3</td>
<td>$y = 0.2248x - 2.0235$</td>
<td>0.9738</td>
</tr>
<tr>
<td>Case 4</td>
<td>$y = 0.1909x - 2.3127$</td>
<td>0.9876</td>
</tr>
</tbody>
</table>

These results indicate that though an improvement in $R^2$ value can be attained by using Case 2, the presence of blank fields as zero can significantly affect the slope of regression line for the same cluster length data. Moreover, increasing the unit area yields a better linearity of the regression line by eliminating the blank fields. Therefore, for EVD analysis of clusters it is important to select an appropriate unit area which can eliminate the blank fields.

3.2.3.2. Size parameter for EVD of clusters

After determining that Case 4 is the most suitable method with respect to blank fields, it was applied for EVD analysis of S3 to determine the appropriate shape factor to be used for studies of clusters. Fig. 3.30 depicts the EVDs obtained for three different size parameters, and the corresponding equations and $R^2$ values for these parameters are enlisted in Table 3.9. Among the investigated size parameters, $L_C$ gives the highest $R^2$ value (0.9876) followed by $\sqrt{\text{area}_{ij}}$ (0.9774) and $\sqrt{\text{area}_{max}}$ (0.9656). Moreover, for $\sqrt{\text{area}_{max}}$ several data larger than ~20µm deviate from a linear function. A variation in the slopes of the regression lines obtained for all these size parameters can be noted, which is obviously due to different definitions of these parameters. The size parameter $\sqrt{\text{area}_{ij}}$ being the smallest among all results in maximum slope of regression line and vice versa is true for $L_C$. According to these results the size parameters $L_C$ and $\sqrt{\text{area}_{ij}}$ can be preferred over the $\sqrt{\text{area}_{max}}$ values for an EVD analysis of clusters. However, the selection of the size parameter is solely dependent on the choice of information required to determine a specific property of a steel product. For instance, the $\sqrt{\text{area}_{ij}}$ value might be the appropriate choice for estimating the volume fractions of clusters in a steel melt.
Fig. 3.30. Comparison of EVDs of sample S3 obtained for different size parameters.

Table 3.9. Regression line equations and coefficient of determination, $R^2$, for EVDs obtained using different size parameter and $A_0$ as unit area.

<table>
<thead>
<tr>
<th>Size parameter</th>
<th>Regression line function</th>
<th>$R^2$ value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sqrt{area_{ij}}$</td>
<td>$y = 0.4020x - 2.3994$</td>
<td>0.9774</td>
</tr>
<tr>
<td>$\sqrt{area_{max}}$</td>
<td>$y = 0.2322x - 2.1638$</td>
<td>0.9656</td>
</tr>
<tr>
<td>$L_C$</td>
<td>$y = 0.1909x - 2.3127$</td>
<td>0.9876</td>
</tr>
</tbody>
</table>

3.2.3.3. Validation of predicted maximum sizes

The EVDs and predicted maximum sizes (PMS) of clusters in different volumes of steel were obtained for all three samples (S1, S2 and S3) using Case 4 and $L_C$ as a size parameter. The obtained results are presented in Fig. 3.31. In order to validate the PMS values by EVD, the data obtained by using Method 2 for a cluster observation were used to find the largest clusters on increased observed area (30 – 75 mm$^2$). In Fig. 3.31(b) the observed largest sizes of clusters are plotted against the dissolved weights of steel samples which correspond to the increased observed areas on film filters. For sample S1, a significant difference is found in the predicted and observed $L_C$ for dissolved weights i.e. ~1.3 and 1.6 times larger predicted lengths of clusters for 0.0012 and 0.0024 g dissolved weight, respectively. However, the predicted and observed $L_C$ for sample S2 are relatively in good agreement. The predicted $L_C$ for S2 in dissolved weights of 0.0014 and 0.0054 g are 16.85 and 20.09 µm, respectively. Whereas, the observed $L_C$ in the corresponding dissolved weights are 18.19 and 21.96 µm. For S3 ~10% difference in the predicted (35.016 µm) and observed $L_C$ (38.5 µm) in 0.0024 g of steel can be seen. However, for 0.012 g of dissolved weight a higher deviation between can be seen for the predicted and observed $L_C$, which are 43.44 µm and 56.2 µm, respectively.
Fig. 3.31. (a) The obtained EVDs and (b) predicted and observed maximum lengths clusters in different dissolved weights of steel for all three samples.

Based upon the presented results for validation of EVDs it is difficult to conclude that the predictions of $L_C$ are in good agreement with observed $L_C$ because the deviations are higher than 50%, especially for sample S1. Therefore, there is need to conduct further work for the validation of EVDs.
4. Concluding discussion

The current study intended at the utilization of the size distribution of different micro-sized particles from the iron and steel industry for attaining valuable knowledge for the industry. The dust particles generated due to mechanical wear of iron ore pellets and REM clusters formed in the REM alloyed molten steel were selected for the investigations.

Two different experimental setups were adopted to simulate the mechanical wear of iron ore pellets. These included a planetary mill (Supplement 1 & 3) and a pellet bed setup (Supplement 2). The influence of characteristics of iron ore pellets (Supplement 1), applied external loading on a pellet bed (Supplement 2) and partial reduction (Supplement 3) on the mechanically generated dust was investigated. The size distributions of the generated dust was used to explicate the mechanisms involved in the dust generation in the adopted experimental setups. In general it can be inferred that the size distribution and the amount of dust generated due to the mechanical wear of iron ore pellets is dependent on the mechanism of wear. Also, it is clear that the mechanism of wear is controlled by the processing parameters. For instance, during the transportation and handling of iron ore pellets the load on a pellet bed, the speed of conveyor belt and the heights of drops can be deciding factors for the mechanism of wear and thereby for the amount and the size distribution of the generated dust. Moreover, in a blast furnace, these controlling parameters can be the degree of reduction, burden descending speed and the flow rate of flue gases.

Similar to the dust particles, the size distribution of clusters extracted from REM alloyed stainless steel was used to elucidate the formation and growth mechanism of the clusters (Supplement 4). The classification of REM clusters in two groups according to their circularity factor (CF) helped to understand different growth rate of different sized clusters. For instance, it has been calculated that larger clusters (CF < 0.15) can grow up to ~5 times faster as compared to small clusters (CF ≥ 0.15) during a holding time of 3 minutes (after 6 minutes of REM addition). Such information regarding the growth of clusters can be essential during a ladle treatment for process control. In addition, methodological work was performed in regards of obtaining a reliable size distribution of clusters in steel. Moreover, an extreme value analysis (EVD), which is a common practice in a steel industry for estimating the largest size of inclusions in the melt, was carried out for REM clusters (Supplement 5). A Methodological work was done for the application of an EVD analysis for three dimensional observations of REM clusters. The obtained results can be useful for EVD analysis of clusters. Particularly, the suggestion to avoid the blank observed fields and the recommended size parameter of clusters has a considerable potential.
In general, it can be concluded that an evaluation of the size distribution of particles is an effective way for determining essential information that can be used for understanding and control of different mechanisms in metallurgical processes.
5. Conclusions

In this study, the influence of characteristics of iron ore pellets, applied external loading on a pellet bed and partial reduction on the dust generated due to the mechanical wear of iron ore pellets has been investigated. Moreover, the clusters found in REM alloyed stainless steel are examined to investigate their characteristics and size distribution. Furthermore, an extreme value distribution analysis is applied for the examined REM clusters. Based upon the obtained results followings can be concluded.

5.1. Iron ore pellets’ dust

1. It has been shown that both a planetary mill and a pellet bed setup can be used to produce dust particles similar to those generated in industrial processes.

2. Among the investigated characteristics of the pellets, the density and hardness of different sized pellets was observed to be similar. Moreover, the outer layer of a pellet is 2.6-3.3 times as hard compared to the center of the pellet.

3. The wear trials conducted using planetary mill showed that the size of pellets can influence the wear rate of pellets. In the planetary mill setup, large sized pellets (Group C, 13.5 < $D_{eq}$ < 15.0 mm) showed a 10-20% higher wear rate compared to the small sized pellets (Group A, 9.5 < $D_{eq}$ < 12.5 mm).

4. In the pellet bed setup, a linear increase in the friction forces and dust generation in the pellet bed was observed with increased applied load. More specifically a ~67% increase is observed for both the wear rate and the friction forces with an increased applied load from 1 to 3 kg.

5. The partial reduction of iron ore pellets at 500 °C resulted in 16 – 35% increase in the wear rate of reduced pellets, whereas a significant decrease (~ 86%) in the wear rate was observed for the pellets reduced at 850 °C as compared that of unreduced pellets. The formation of the porous magnetite during the reduction of hematite and metallic iron layer at the surface of the pellets are the main reasons of the aforementioned increase and decrease in the wear rate, respectively.

6. Based upon the analyses of the generated dust, sliding/abrasion and impact/collisions are identified as the mechanisms involved in the mechanical wear of the pellets. Specifically, abrasions contribute to generation of fine ($d_{eq} \leq 5 \mu m$, $L_{max} \leq 10 \mu m$) dust particles and the coarse ($d_{eq} > 10 \mu m$, $L_{max} > 20 \mu m$) dust particles are generated due to collisions.

7. A higher friction in the pellet bed under higher applied load results in an increased contribution of abrasion mechanism and produces a higher number of small sized dust particles ($d_{eq} \leq 5 \mu m$). Moreover, a significantly higher number (3 – 6 times higher) of coarse
particles (>20 µm) is generated by the mechanical wear of reduced pellets than for unreduced pellets.

8. The air velocity required to uplift dust particles can significantly be influenced by their morphology and orientation in the air flow.

9. The particle size distributions (PSDs) of dust obtained by laser diffraction (LD method) and image analysis (SEM method) can be well correlated by considering the circularity factor (CF) for particles having high CF values. Whereas, for large sized dust particles having plate-like shape and lower CF values there is need to introduce some correction factor to obtain a good correlation.

5.2. REM clusters

1. It is established that the growth of REM clusters take place according to two mechanisms. Mechanism 1 involves collisions of individual inclusions and collisions of small sized clusters with individual inclusions. The collisions of clusters with other clusters result in growth of clusters according to Mechanism 2.

2. Turbulent collisions are the dominant collision mode in the growth of REM clusters compared to the Brownian and Stokes’ collisions.

3. The growth rate of REM clusters increases exponentially with an increased size of clusters. This is due to the dominance of Mechanism 2 of growth of clusters for large sized clusters.

4. It is suggested that the collision volume of clusters ($\beta_{ij}$) in the melt should be calculated by considering the shape factor of clusters, since the diameter equivalent ($d_{eq}$) results in underestimation of the collision volume. The maximum length of clusters can be used for estimating the maximum possible $\beta_{ij}$ value for clusters.

5. An appropriate unit area must be selected to avoid blank fields (without any clusters) encountered during EVD analyses of clusters, as the presence of such blank fields can negatively affect the results. Moreover, increasing the observed unit area can significantly improve the correlation coefficient of EVD regression lines. Four times increase in the unit area improved $R^2$ value from 0.9404 to 0.9876.
6. Future work

The author has the following suggestions for the future work:

1. The experimental setups used for the mechanical wear of iron ore pellets had limitations such as the rotation speed for both planetary mill and the pellet bed setup and air flow rate in the pellet bed setup. It is suggested to study the influence of varied rotation speeds and their correlation to the movement of pellets during transportation and handling as well as the descending speed of the burden in a blast furnace. Moreover, the flow rate of the supplied air can be varied to estimate variance in the amount of dust exiting the pellet bed.

2. A bed made of other charged materials, such as coke, can be used for similar studies. It seems obvious that dust generation in a coke bed can be influenced by the presence of a layer of pellets. Therefore, a bed consisting of alternate layers of pellets and coke is suggested to investigate dust generation.

3. If possible, the supplied air should be replaced by a hot reducing gas to replicate the condition in the upper shaft of blast furnace.

4. The pellets used in the current study were obtained directly from the palletizing plant. It is suggested to conduct experiments on the pellets collected from pellet storage in an ironmaking plant. This is because the dust generated in the prior stages could stick on the pellet surface and the pellets can also absorb moisture.

5. Online measurement of airborne dust particles at different process steps can be useful for estimating the dust generation and optimizing the process parameters. For instance, recording the airborne concentrations of dust conveyer belts can help to adjust the speed of belts to minimize the dust generation during transportation on them.

6. There is need of further work on validation of EVD analysis performed for REM clusters.

7. An introduction of an appropriate shape factor is necessary for adopting any kind of equations for irregular shaped particles, which are originally derived for spherical particles. For instance, the collision volumes of clusters can be calculated by considering their circularity factor. However, as it was observed in this study that the circularity factor might vary with length of clusters, there is need to introduce a better shaper factor. Perhaps, the fractal dimension of clusters can be considered for future work.

8. Information from a reliable cluster size distribution can be deployed in a mathematical model to estimate growth and removal rate of clusters during a ladle treatment.
References

A study of micro-particles in the dust and melt at different stages of iron and steelmaking


