Morphology and wear of high chromium and austempered ductile iron balls as grinding media in ball mills

BRN Murthy\textsuperscript{a}\textsuperscript{*} Ravichandra Rangappa\textsuperscript{b}

\textsuperscript{a}Department of Mechanical and Manufacturing Engineering, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal, India -576104
\textsuperscript{b}Department of Mechanical Engineering, Sahyadri College of Engineering & Management, Mangaluru, Karnataka, India 575007

*Correspondence E-mail: murthy.brn@manipal.edu Phone: +91 962086749

ABSTRACT: High chromium balls are recognized as better grinding media in terms of wear rates than forged steel balls, which are conventional grinding media in the milling process of iron ore. In this research work, the wear rate of high chromium balls and austempered ductile iron (ADI) balls as crushing media in a ball mill are compared. ADI are prepared by austenitizing the spheroidal graphite (SG) iron balls at 920 °C for one hour, and step austempering heat treatments were given, which includes the first step austenitizing at 300 °C for 15 min, followed by a second step austenitizing at 400 °C for 60 min. The wear rates were estimated when both balls were used separately by maintaining the same machining conditions and when the balls are mixed. The grinding wear conduct of both materials is evaluated for wear loss in wet grinding conditions. The experimental results reveal that the performance of ADI balls is better than high chromium balls when tested separately and mixed. Results also indicate that the wear rates/revolutions will decrease when the operating period increases.

Keywords: Austempered Ductile Iron; High Chromium Balls; Grinding Media; Ball Milling Process; Wear Rate.

1. Introduction
Austempered ductile iron (ADI) is produced by generating a bainitic structure in the material by conducting an austempering over spheroidal graphite (SG) irons. The outstanding characteristics of ADI are connected to its exclusive ausferrite microstructure, which consists of ferrite and high-carbon stable austenite [1-4]. High strength with high ductility, excellent wear resistance, and high fatigue strengths are the attractive properties of ADI [5]. Due to the incredible combination of these properties, ADI materials have emerged as a new group of ductile iron [6]. ADI matrix is the combination of bainitic ferrite along with the austenite retained. The mechanical characteristics of ADI are relying upon the properties and morphology of micro-ingredients. Morphology of ADI can be altered by altering the composition of the alloy, parameters of heat treatment, such as the temperature of austenitizing, duration of austempering, and stepped austempering [7].

The wear loss of grinding ball in grinding mills is due to the combination of several aspects such as abrasion, corrosion, impact, and erosion. Several years back it was calculated that more than 5, 00,000 tons of iron and steel are spent as grinding media every year worldwide. It has been estimated that nearly 50\% of the total mineral processing cost will be consumed by ball mills alone [8, 9]. The wear loss of grinding media material possesses enormous hassle, leading to substantial increases in the manufacturing cost. This increase is due to the replacement of high-priced material and the loss of production occurring due to premature wear or breakage of balls. Hence, it is crucial to reduce the
processing cost by identifying the effect of various parameters on the wear rates of the ball and take the required steps to minimize the wear rate in balls used in the mills. Iron Research Association (BCIRA) and International Nickel Company (INCO) introduced ductile cast iron to the industry in 1948. This material is referred to as nodular cast iron or spheroidal cast iron or graphite cast iron [10]. Xiao et al. reported the size, material, shape aspect influence on the ball mill performance. They found cast iron ball groups are better than steel balls, and excellent grinding medium improved the efficiency of the mill [11]. Sellamuthu et al. reported that the variation in the austempering temperature and duration would lead to a different microstructure, which has a significant influence on the specific wear rate of grinding media and its performance [12]. Similarly, Siddan worked on the wear behavior of ADI balls with two-stage austempering. They predicted that the temperature of the first-stage austempering has a significant effect over the microstructure formed and, thus, on the wear behavior [13]. Clermont et al., have shown that continuous measurement of the degree of grinding ball fill and pulp position is vital to improving the mill performance [14]. In the present work, the authors have attempted to compare the wear of chromium balls and ADI balls as grinding media in a ball mill.

2. Materials and Methods

2.1 Materials

The granulated iron ore was collected from M/s Kudremukh Iron Ore Company Limited (KIOCL) Mangalore, Karnataka, India. The chemical analysis of the iron ore sample used for the study is illustrated in Table 1. The SG iron balls (25 mm diameter 100 numbers) and Chromium balls (50 mm diameter 50 numbers) free from defects were selected and used for the experiments are procured from M/s Shanthala Spherocast Private Limited, Shimogga, Karnataka, India. The chemical composition of SG iron balls is illustrated in Table 2.

| Elements      | Composition (Wt.%)|
|---------------|------------------|
| Manganese (Mn)| 0.05             |
| Silicon (Si)  | 1.6-1.8          |
| Sulphur (S)   | 0.007            |
| Aluminum (Al) | 1.8              |
| Phosphor (P)  | 0.07             |
| Iron (Fe)     | 65.5             |

Table 2. The chemical composition of SG iron balls.

| Elements            | Composition (Wt.%)|
|---------------------|-------------------|
| Manganese (Mn)      | 0.33              |
| Silicon (Si)        | 2.62              |
| Phosphor (P)        | 0.018             |
| Carbon (C)          | 3.5               |
| Nickel (Ni)         | 1.37              |
| Molybdenum (Mo)     | 0.30              |
| Magnesium (Mg)      | 0.004             |

2.2 Austempering process.

The heat-treatment cycle of SG iron balls is, as shown in Figure 1. Initially, SG iron balls were preheated for 10 minutes to remove surface contamination. Further, the cleaned SG iron balls, using running hot water, were kept in the furnace and heated to the austenitizing temperature of 920 °C and held for one hour. This temperature and duration were selected based on the requirement of entire balls austenitizing. The austempering process was carried out in two stages. The first stage was that the balls were moved
to a salt bath of 300°C temperature for quenching and held for 15 minutes. In the second step austempering, balls were taken out from the primary salt bath and put into the secondary salt bath, which was held at 400°C for 60 min. Then, balls were taken out of the salt bath and placed in the open air to cool them to room temperature. The salt bath utilized for austempering treatment was a blend of sodium nitrate and potassium nitrate in the proportion of 55:45. In order to predict the effect of first step austempering time, the quenching time in first salt bath was held at various levels like 15 min, 30 min, 45 min and 60 min as shown in Table 3. The wear rate obtained for various types of austempering treatment is also depicted in Table 3. From the same table it is evident that type B (30 min first step quenching time) exhibits the lowest wear rate.

![Heat-treatment cycle followed during austempering.](image)

Figure 1. Heat-treatment cycle followed during austempering.

### Table 3: Austempering type and wear rates

| Type | Austenitising | First step austempering | Second step austempering | Wear rate cm$^3$/rev. ($\times 10^{-8}$) |
|------|---------------|-------------------------|--------------------------|----------------------------------------|
|      | Temp. (°C)    | Time (min)              | Austempering Temp. (°C)  | Austempering time (min.)              | Austempering Temp. (°C)  | Austempering time (min.)              |
| A    | 920           | 60                      | 300                      | 15                                     | 400                      | 60                                     | 141.14                                 |
| B    | 920           | 60                      | 300                      | 30                                     | 400                      | 60                                     | 102.30                                 |
| C    | 920           | 60                      | 300                      | 45                                     | 400                      | 60                                     | 168.389                                |
| D    | 920           | 60                      | 300                      | 60                                     | 400                      | 60                                     | 163.436                                |

2.3 Wear tests and characterization.

The ball mill set-up used for the experimental work is shown in Figure 2. It consists of a barrel-shaped shell whose length and diameter was 29.46 cm and 20.32 cm, respectively. The inner side of the shell is lined with neoprene rubber. The ball mill barrel is closed on one side, and a separable cap is given at the opposite end and fixed with the help of bolts and nuts. An opening of a 3 mm diameter is provided at the center of the cap through which air or oxygen could be fed during wet grinding. This arrangement is for controlling air circulation on balls to estimate the influence of air on ball wear.
The grinding test was conducted using both types of balls separately and by mixing. Three groups of grinding media were selected for estimating the wear and specific wear rate through ball-mill experimentation, such as; (a) 25 mm diameter 25 of ADI balls were selected out of 100 ADI balls used in the experiment, (b) 50 mm diameter 20 chromium balls were selected out of 50 chromium balls used in the experiment, and (c) combination of 25 ADI balls and 20 chromium balls were selected out of 100 ADI and 50 chromium balls used in the experiment. The selected balls were stamped and measured the total initial weight in all three cases of experiments up to three decimal places using a high-precision electronic weighing machine.

The experiment was conducted in wet conditions. The aggregate plant charge comprised of 1.5 kg of iron ore sample of mesh size of -10+30 along with 1000 ml of water was used, it adds up to 60 % pulp density. The pH level of the water is maintained at 7. This pH was held constant by the addition of lime to the pure water. The crushing mill was spun at 86 RPM. After every grinding experimentation, the noted balls are picked, washed thoroughly with pure water, and dropped on the plastic plate along with acetone. Balls are then kept in hot air stove for 60 minutes, and the temperature was maintained at 100 °C. Then it is cooled down to room temperature, and balls were preserved in the air sealed desiccators for a period of three to four hours. Once the balls cool down to room temperature, precisely weighed by using an electronic balance. The difference in weight of balls before the experiment and after the experiment was estimated. The rate of wear can be estimated using the following equation:

\[
\text{wear rate (cm}^2) = \frac{\text{Wear loss (gms/ball)}}{\text{Total number of revol} \times \frac{1}{\text{Density of material}}} \quad \ldots\ldots(1)
\]

The slurry got was separated utilizing pressurized filtering arrangement and dried in a stove. Further, the experiments were carried out for different time intervals like 2, 4, 8 and 16 hrs. in all three cases.
3. Results and Discussion

3.1 Microstructural analysis

A two-step austempering was performed in this work to obtain maximum grinding wear resistance. In the first stage austempering, the initiation of bainitic transformation around nodules starts from holding SG balls at a temperature of 300 °C for 15 minutes. Lower bainite formation starts and continues up to a certain level, depending on austempering time [12]. At the end of the first austempering treatment, some portions of austenite, mainly in the intercellular region, do not transform. The untransformed austenite will be converted to upper bainite when held at a higher austempering temperature of 400 °C. The rest of the untransformed region could be transformed into the bainitic structure when it held at 400 °C for 60 minutes in the second stage austempering process. Optical photomicrographs were taken to study the variations after the completion of austempering heat-treatment.

Figure 3. Optical photomicrographs of a) as-cast SG iron ball sample, and b) austempered ball samples austempered at 300° C for 15 min etched with 3 % Nital.
Figures 3 (a) and 3(b) shows the microstructure of as-cast SG iron ball sample and austempered ball samples. There are significant differences in the two cases. Due to austempering in the SG iron, the high amount of carbon in the ductile iron produces the spheroidal graphite nodules implanted in the iron matrix. In contrast, when the SG iron balls are quenched and held at the austempering temperature, the acicular ferrite will be formed accompanying rejection of carbon into austenite. This particular microstructure is also identified as “ausferrite” which is the blend of acicular ferrite and high carbon austenite [11]. In this experimental work, since the first stage austempering temperature is 300 °C, the obtained microstructure is a lower ausferrite microstructure. This particular microstructure comprises of spheroidal graphite along with a matrix of ausferrite needles as seen in the micrographs.

3.2 XRD analysis
The phase proportions of ADI balls were assessed by the X-ray diffraction method using the XRD pattern generated by the samples. The XRD patterns taken for as-cast SG iron balls are presented in Figure 4(a), and the pattern taken for austempered balls is illustrated in Figure 4 (b). It is evident from the figures that, in the sample with as-cast conditions, there is a large amount of ferrite is present, and there is no sign of retained austenite. Since the ferrite is a soft phase, the hardness of the sample is low. When the two-stage austempering heat treatment has been conducted, the quantity of ferrite decreases, and the volume percentage of retained austenite increases. This decreased ferrite is transformed into other phases such as bainite and martensite (Fig 4 b). This phase transformation enhances the hardness of the austempered balls, which was more significant than the SG iron.

![XRD spectra of samples](image)

Figure 4. XRD spectra of samples of a) as-cast SG iron ball b) austempered ball.
3.3 Wear study
The wear loss of the grinding media from the ball-mill experiments in various operating conditions is presented in Tables 3. The comparative wear loss and wear rate of chromium balls and austempered balls versus duration of milling when they are operated separately is plotted in Figure 5 (a) and 5 (b), respectively. Similarly, the comparative wear rate of chromium balls and austempered balls versus milling duration when operated under mixed conditions is presented in Figure 5 (c). It is found that wear loss and wear rate of austempered balls is lower than chromium balls. Further, as the number of operating hours increase, there is an increment in the total wear loss of the balls. When austempered and high chromium balls operated separately, the wear rate of austempered balls is lower than the high chromium balls. This decrease may be due to the formation of lower bainitic structure ausferrite, which is a mixture of acicular ferrite and high carbon austenite. The wear loss of balls in the ball mill while crushing is dependent on factors such as hardness, micro-constituents, pH value of the water, and phase structure. When SG iron balls austempered at 300 °C for 15 minutes, followed by 400 °C for 60 minutes, we obtain a structure containing materials with a 76.5 % ferrite, 23.5 % retained austenite and 1.77 % carbon in the retained austenite. Meanwhile, as the operating hours prolong, there is a decrement in the wear rate. In the initial stages of the grinding operation, the ore particles have sharper edges, which generates the chipping action when the balls fall on them. As the grinding action proceeds, these sharp edges become blunt, and thus, there is a reduction in the wear rate as the operating duration increases. When the milling operation is performed by mixing the balls, there is a considerable reduction in the wear rate of both high chromium balls and ADI balls (Table 4). This decrease might be because each type of ball’s mutual contribution during the initial stages of the grinding process at which the wear rate was high. The exact reason for this phenomenon needs to be investigated by carrying out further investigation.

Table 4. Wear loss and wear rate of high chromium and austempered balls.

| Experimental condition | Grinding duration (Hrs.) | 1   | 2   | 4   | 8   | 16  |
|------------------------|-------------------------|-----|-----|-----|-----|-----|
|                        |                         |     |     |     |     |     |
| ADI balls (Operated separately) | Total Wt. loss (g) | 0.49 | 0.94 | 1.768 | 1.942 | 2.728 |
|                        | Wt. loss/ ball (g)     | 0.0306 | 0.0587 | 0.073 | 0.121 | 0.170 |
|                        | Wear rate x10^4 cc/revolution | 82.43 | 79.06 | 49.52 | 40.83 | 28.68 |
| Chromium balls (Operated separately) | Total Wt. loss (g) | 1.8173 | 3.2067 | 6.8012 | 8.2014 | 14.4102 |
|                        | Wt. loss/ ball (g)     | 0.1125 | 0.20 | 0.425 | 0.5125 | 0.90 |
|                        | Wear rate x10^4 cc/revolution | 272.5 | 242.2 | 171.5 | 155.1 | 36.2 |
| Chromium and ADI balls (Mixed) | Wear rate x10^4 cc/revolution | 71.673 | 61.708 | 40.781 | 31.709 | 20.579 |
|                        | ADI Balls Wear rate x10^4 cc/revolution | 222.217 | 189.120 | 138.836 | 126.193 | 29.919 |
|                        | chromium               |     |     |     |     |     |
Figure 5. A plot of a) wear loss versus milling duration b) wear rate versus milling duration of high chromium balls and austempered balls when are separately used in ball-milling c) wear rate versus milling duration of high chromium balls and austempered balls when are used in mix mode in ball-milling.
4. Conclusions

We can draw the following conclusions from the microstructural analysis of as-cast SG iron balls and austempered balls, wear analysis, and X-RD studies.

- Austempering has a significant effect on the microstructure and the wear behavior of grinding balls in the ball mill.
- The performance of austempered balls better than the high chromium balls.
- The use of austempered SG iron balls instead of high chromium balls minimizes the material consumption in ball-milling operations. Thus, reduces the operation cost.
- The wear rate can be further minimized in ball milling operation by mixing the austempered balls and high chromium balls.
- In the initial stages of the grinding balls, wear is more while milling of selected iron ore samples in this study.
- Wear loss of grinding media is directly proportional to milling times, but the wear rate is inversely proportional to the time factor.

5. References

[1]. Bosnjak, B., Radulovic, B. 2004. Effect of austenitising temperature on the austempering kinetics of Ni-Mo alloyed ductile iron. Materials and Technology, 38, 307-312.
[2]. Yang, J., Putatunda, S. K. 2004. Improvement in strength and toughness of austempered ductile cast iron by a novel two-step austempering process. Materials & design, 25, 219-230.
[3]. Myszka, D. 2007. Austenite-martensite transformation in austempered ductile iron. Archives of Metallurgy and Materials, 52, 475-480.
[4]. Eric, O., Jovanovic, M., 2004. Microstructure and mechanical properties of CuNi-Mo austempered ductile iron. Journal of Mining and Metallurgy, 40B, 11-19.
[5]. Nasir, T., Northwood, D. O., Han, J., Zou, Q., Barber, G., Sun, X., Seaton, P. (2011). Heat treatment–microstructure–mechanical/tribological property relationships in austempered ductile iron. WIT Transactions on Engineering Sciences, 71, 159-170.
[6]. Hsu C.-H., Lin K.-T. A 2001 Study on microstructure and toughness of copper alloyed and austempered ductile irons. Material Science and Engineering, 528, 5706–5712.
[7]. Zhang J., Zhang N., Zhang M., Lu L., Zeng D., Song Q. (2014). Microstructure and mechanical properties of Austempered ductile iron with different strength grades. Materials Letter, 119, 47–50.
[8]. Prasad Rao P., Putatunda S. K. 2001 Investigations on the fracture toughness of austempered ductile iron alloyed with chromium. Materials Science and Engineering, A, 349, 136-149.
[9]. Jain S.K. 2001. Mineral Processing, 2nd edition, CBS Publisher and distributor, New Delhi, 102-105.
[10]. Richard W. Heine, Carl. R. Loper, Jr., Philip C. Rosenthal (1967). Principle of Metal Casting Mc Graw Hill Book Company, New York, II edition, 614- 641.
[11].Qingfei XiaoBoLi Huaibin Kang 2014, The Effect of Fine Grinding Medium Feature on Grinding Results, AASRI Procedia, Volume 7, 120-125.
[12].Prabhukumar., Sellamuthu, D. G. Harris Samuel, D. Dinakaran, V. P. Premkumar, Zushu Li 2018, Austempered Ductile Iron (ADI): Influence of Austempering Temperature on Microstructure, Mechanical and Wear Properties and Energy Consumption, Metals, 8, 53-64.
[13].Siddan J. B. 2014, Wear behavior of stepped austempered ductile iron balls in grinding iron ore, International Journal of Research in Engineering and Technology, 03, 475-480.
[14].Clermont, b., De Haas, b., Hancotte, O. 2008, Real time mill management tools stabilizing your milling process. Third International Platinum Conference Platinum in Transformation, The Southern African Institute of Mining and Metallurgy