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Investigation on grinding wear behaviour of austempered ductile iron as media material during comminution of iron ore in ball mills

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Abstract
An attempt has been made to assess the grinding wear behaviour of austempered ductile iron (ADI) as media material in comminution of Kudremukh haematite iron ore in a ball mill. Spheroidal graphite (S.G) iron balls were austenitised at 900ºC for one hour and austempered at 280ºC and 380ºC for different time durations. These materials were characterized by measuring hardness, carrying out X-ray diffraction analysis, studying microstructures using scanning electron microscope (SEM). Grinding wear behaviour of ADI was assessed during wet grinding at different pH of the mineral slurry. The wear resistance of ADI was compared with that of forged En 31 steel balls under similar grinding conditions. It was found that ADI balls austempered at 280ºC for 30 minutes which contains lower bainite registered superior wear resistance. It was also noted that the wear resistance of ADI was more at higher pH range of the slurry.

1. Introduction

Grinding is the most important, critical and common process involved in mineral processing, which is mainly aimed at liberation of one or more minerals present in the ore. Grinding of ore is a cost and energy intensive process, which is done in mills and wear of grinding media accounts for the good portion of the grinding cost [1-2]. Wear of any grinding media is affected by many parameters and option for changing the parameters is limited [3]. The widely used grinding media materials in ball mills are cast steel, forged steel, high chrome steel and alloy steel. Only alternative left for cost reduction is to change the ball material which would possess superior wear resistance. Materials with combination of properties like toughness and hardness are required in such situations [4].

In the present study considerable effort is devoted to develop a material with improved wear resistance for grinding media material. One such material which possesses combination of properties like toughness and hardness is austempered ductile iron (ADI) [5]. ADI is a type of S.G. iron which is subjected to isothermal heat treatment to produce essentially an ausferrite structure in the material. This material is known to possess high strength with toughness, wear resistance, fracture toughness and fatigue strength. The reasons for good properties in ADI are not only due to the surface hardness, but also due to its microstructures which contains ausferrite [6].

2. Materials and methods

2.1 Production of alloyed Spheroidal Graphite (S.G) iron

The S.G. iron balls with required composition and relatively free from defects were produced by sand casting method. Microscopic examination of the S.G. iron balls showed the presence of huge amount of carbides and homogenizing the balls at 900ºC for the duration of 2 hours and overnight furnace cooling has completely eliminated the carbides. These S.G.iron balls free from defects like porosity, cracks and sand inclusions are further subjected to heat treatment.

2.2 Heat treatment processes

Austenitising treatment was carried out on homogenized balls at 900ºC for one hour on each set of 200 balls in a Muffle silicon carbide electrical resistance furnace. Immediately after the austenitising, the balls were given austempering treatment by immersing them in a salt bath containing a mixture of sodium nitrate and potassium nitrate (55:45 by weight), maintained at a temperature of 280ºC for a period of 30 mins, 60 mins or 90 mins on each set of 200 balls. Similarly austempering was carried out at 380ºC for a period of 30 mins, 60 mins or 90 mins on different sets of 200 balls. Then the ball samples were air cooled.

2.3 Metallography

Morphological features of the microstructures were studied using scanning electron microscope (SEM) after etching the samples with 3% nital.

2.4 X-ray Diffraction (XRD) Analysis

Quantitative information on the volume fraction of retained austenite and its carbon content were obtained through X-ray diffraction on Jeol JDX 8P diffractometer. Diffraction studies were carried out using CuK_{α} radiation, and scanning was done over the 2θ range of 4^o at a scan speed of 2^o min^{-1}. The volume fraction of the retained austenite was determined by the direct comparison method using integrated intensities of (110) peak of ferrite and (111) peak of austenite. Assuming that ferrite and austenite were the only matrix phases present, the ratios of integrated intensities of diffraction peaks from these phases are given in equation 1 [16].

$$\frac{I_{\gamma(hkl)}}{I_{\alpha(hkl)}} = \frac{R_{\gamma(hkl)}}{R_{\alpha(hkl)}} . \frac{X_{\gamma}}{X_{\alpha}} \tag{1}$$

where $I_{\gamma(hkl)}$ and $I_{\alpha(hkl)}$ are the integrated intensities of a given (hkl) plane from the γ phase and α phase respectively. X_{γ} and X_{α} are the volume fractions of retained austenite and ferrite respectively. The empirical relationship given in equation 2 [16] is widely accepted for lattice parameter.

$$a_{\gamma} = 0.3548 + 0.0044C_{\gamma} \tag{2}$$

where a_{γ} is the lattice parameter of austenite in nanometres and C_{γ} is its carbon content in wt.%. By finding out $d_{(111)}$ values for (111) the lattice parameter a_{γ} for retained austenite is calculated.

2.5 Hardness

The hardness of as cast S.G.iron, ADI and En 31 forged steel balls were measured using Brinell hardness tester. The applied load was 3000 Kgs, with the hardened steel as indenter material having diameter of 10 mm was used to carry out the hardness test.

2.6 Grinding experiment at different pH of the slurry and Sieve analysis

The grinding experiments were carried out at different pH, i.e., 7.0 and 8.5 keeping other parameters fixed, at a mill speed of 74 rpm. for one hour in a ball mill, which is basically a cylindrical shell having a length of 30cm and diameter of 20cm. The ore samples were crushed to the size of -10 and +30 mesh size using the laboratory jaw crusher. The mill charge consists of a set of 200 ADI balls including 25 marked ones of each 2.5cm diameter, 1500 gms. of iron ore and 1000 ml of water to prepare a feed of required pulp density. After each grinding experiment the 25 marked balls were washed, dried and weighed. The difference in weight, i.e., initial weight before grinding and final weight after grinding gives the weight loss of balls. Similarly the grinding experiments were carried using 200 forged En 31 steel balls as media material. The density of ADI is 7.2 gms/cc and that of forged En 31 steel is 7.6 gms/cc. The wear rate of the grinding media is calculated using equation 3.

$$\begin{matrix}\textit{Wear rate} \\ (cm^3/rev)\end{matrix} = \frac{\begin{matrix}\textit{Weight loss} \\ \textit{in gms/ball}\end{matrix}}{\begin{matrix}\textit{Total no. of rev.} \\ \textit{of the mill/hour}\end{matrix}} \; x \; \frac{1}{\begin{matrix}\textit{Density of} \\ \textit{ball material}\end{matrix}} \tag{3}$$

After each grinding experiment, the slurry was filtered and dried and representative sample of 100 gms was taken and subjected to sieve analysis in a row-top sieve shaker. The sieve numbers used for the sieve analysis were between +75 and -75 and +53 and -53.

3. Results

3.1 Chemical composition of S.G.iron and hematite ore sample

The chemical compositions of S.G.iron and forged En 31 steel are given in Table 1 and iron ore sample is given in Table 2 respectively.

Table 1 : Chemical composition of S.G.iron and forged En 31 steel balls in wt.%

Material	*C*	*Si*	*Mn*	*Cr*	*Mo*	*S*	*P*	*Mg*
S.G.Iron	3.6	2.8	0.4	--	0.3	0.01	0.01	0.04
En31 steel	1.0	0.2	0.5	1.4	--	--	--	--

Table 2 : Chemical composition of the iron ore in wt.%

Elements	Fe_2O_3	SiO_2	*Alumina*	*Sulphur*	*Phosphorous*
Composition Wt.%	94.87	1.7	1.8	0.007	0.07

3.2 Metallography

3.3 X-Ray Diffraction (XRD) analysis

The XRD results of the different ball samples are given in Table 3.

Table 3 : Volume fraction of retained austenite and carbon content of retained austenite by XRD analysis at lower and higher austempering temperature

Sl. no	*Austempering condition after austenitised at 900ºC for one hour*	*Volume fraction of austenite (Xγ) in vol %*	*Carbon content of austenite (Cγ) in wt.%*
1	280ºC for_30 mins	26	1.90
2	280ºC for_60 mins	27	1.75
4	380ºC for 30 mins	46	1.82
5	380ºC for 60 mins	39	1.78
6	380ºC for 90 mins	31	1.01

3.4 Hardness and wear behaviour of ADI as grinding media balls

The hardness value of the homogenized S.G.iron ball is 271 BHN. The hardness and wear rate values of the heat treated S.G.iron balls and En 31 balls are given in the Table 4

Table 4 : Hardness and wear rate of ADI compared with forged En 31 steel balls at different austempering conditions and pH values

Material	Austempering Condition	Wear rate ($x10^{-8}$ cm^3/revolutions) pH 7	Wear rate ($x10^{-8}$ cm^3/revolutions) pH 8.5	Hardness BHN
ADI	280ºC for 30 mins	230	107	444
ADI	280ºC for 60 mins	258	172	415
ADI	280ºC for 90 mins	286	202	388
ADI	380ºC for 30 mins	304	226	363
ADI	380ºC for 60 mins	336	265	341
ADI	380ºC for 90 mins	368	286	321
En31 steel	--	965	554	529

3.5 Grinding efficiency

Grinding efficiency is defined as percentage fraction of ground ore passing through 200 mesh (less than 75µm in size) in the given condition of wet grinding operation. It was found that the operation with ADI balls austempered at 280ºC for 30 minutes registered the higher efficiency of 68% as compared to that with forged En 31 steel balls, which registered lower efficiency of 61%. The results of the sieve analysis are shown in the Table 5.

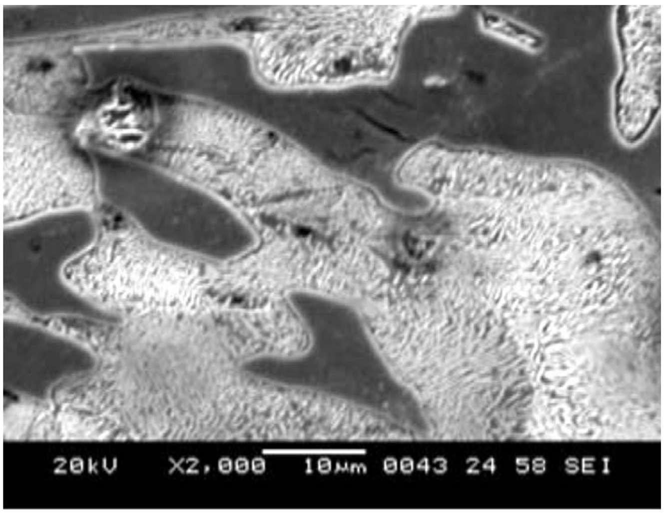

Fig. 1 : SEM of as cast S.G iron

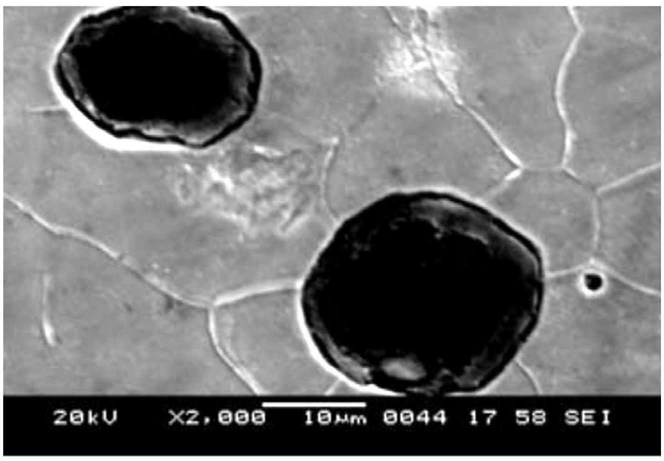

Fig. 2 : SEM of homogenised S.G. iron

Table 5 : Comparison of sieve analysis results of ADI with forged En31 steel

Sieve size range (Microns)	Sieve size range (Mesh)	Weight retained in gms. ADI	Weight retained in gms. En31	Cumulative wt. % passing ADI	Cumulative wt. % passing En31	Cumulative wt. % retained ADI	Cumulative wt. % retained En31
+75	+200	5	12	95	88	5	12
-75+53	-200+300	27	27	68	61	32	39
-53	-300	68	61	00	00	100	100

4. Discussion

4.1 Microstructural evaluation of ADI

Superior wear behaviour and other remarkable properties such as high strength, ductility and toughness are attributed to unique microstructure of ADI, which is ausferrite one and consists of ferrite and austenite. As the ferrite lath, which nucleates predominately around the graphite nodule, tends to grow, the surrounding austenite gets enriched in carbon. The process of enrichment with carbon causes stabilization of the austenite and retains it even after cooling the material to room temperature. Thus, we get austenite in between the ferrite lath as retained austenite and in the mid region between graphite nodules as untransformed austenite as in Figs. 3 to 8. The materials austempered at lower temperature

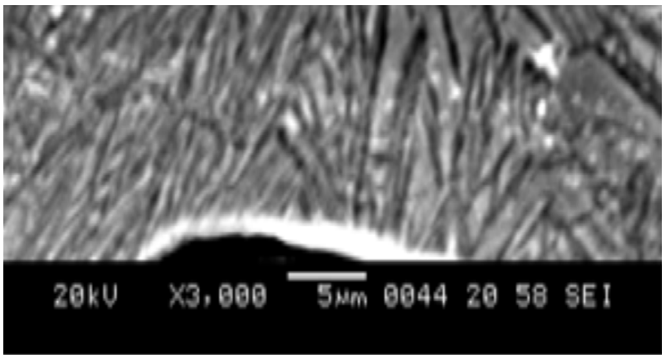

Fig. 3 : SEM of S.G. iron austempered at 280ºC for 30 mins reveals lower fine ausferrite

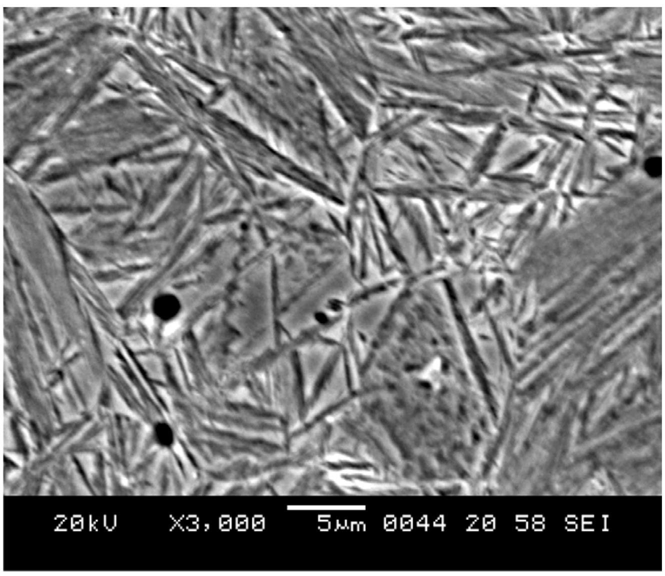

Fig. 4 : SEM of S.G. iron austempered at 280ºC for 60 mins

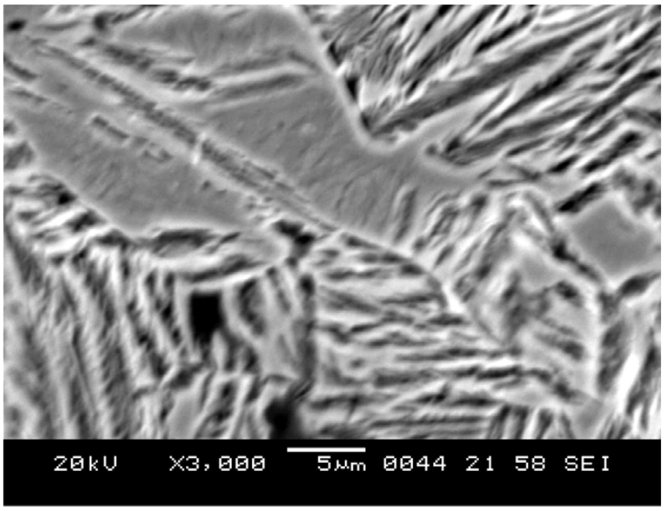

Fig. 5 : SEM of S.G. iron austempered at 280ºC for 90 mins

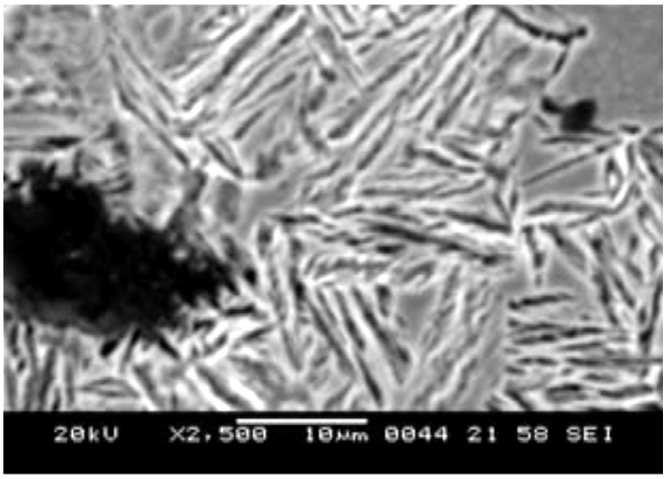

Fig. 6 : SEM of S.G. iron austempered at 380ºC for 30 mins reveals feathery upper ausferrite

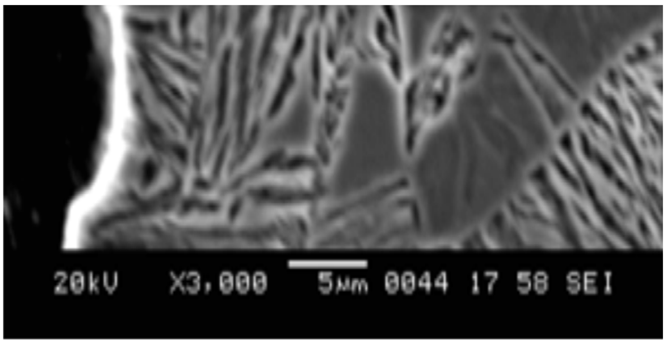

Fig. 7 : SEM of S.G. iron austempered at 380ºC for 60 mins

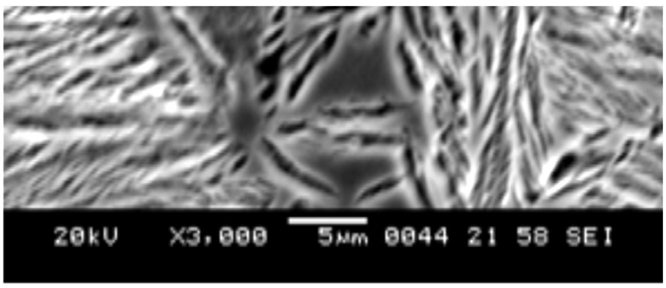

Fig. 8 : SEM of S.G. iron austempered at 380ºC for 90 mins

of 280ºC show acicular ferrite (Figs. 3 to 5) and the samples austempered at higher temperature of 380ºC show feathery type of ferrite (Figs. 6 to 8).

4.2 XRD analysis

It was observed from the XRD values presented in the Table 3 that the fraction of retained austenite was more in the case of materials austempered at 380ºC when compared to those austempered at 280ºC. Among those austempered at 280ºC, the balls austempered for 30 mins registered lowest fraction of retained austenite. As the austempering time increased to 60 mins and 90 mins, the fraction of retained austenite also increased. But the hardness values of the material showed the reverse trend.

The XRD results reveal that the austempering at higher temperature results in higher amount of retained austenite compared to that at lower temperature. Further, samples austempered at 380ºC register decreasing austenite content as duration of austempering increases. However, when samples were austempered at 280ºC the austenite content of the material increases as time of austempering increases, resulting in decreasing carbon content of retained austenite with continued austempering.

4.3 Grinding wear behaviour of ADI balls

Wear behaviour of ADI is controlled by volume fraction of retained austenite, carbon content of the same and morphology of ausferrite. The results brought out in table 4 reveal that the grinding wear resistance of ADI balls is superior to that of forged En-31 steel balls. Among ADI balls, the samples austempered at 280ºC for 30 mins shows the highest wear resistance. During the operation in the ball mill the grinding media material has to face three types of aggressive condition: (i) wear, (ii) impact and (iii) corrosion. It is well known fact that the above three conditions don't act independently, instead they do so collectively.

The microstructure plays an important role in the development of micro cracks. The micro crack may move through the region of retained austenite or through the area of ferritic lath or through the interface between austenite and ferrite. The structure becomes very fine and acicular when they are austempered at lower temperature, which naturally resists the propagation of crack growth. When the samples are austempered at higher temperature the coarse microstructure allow the cracks to grow more easily. Further, it is known that the austenitic region resists the crack growth because of the inherent property of strain induced martensitic transformation. The interface seems to be more favourable phase for the development of crack particularly in those samples where there is a possibility of carbide precipitation.

ADI consists of graphite nodule embedded in ausferrite matrix. Graphite being a solid lubricant comes out of the matrix, stands in between working interfaces and improve the wear resistance. The present investigation reveals that the ADI balls exhibit superior wear resistance in spite of low hardness when compared to forged En-31 steel balls. This brings out the fact that hardness on the surface is not the only criteria in deciding the wear behaviour of grinding media materials, but equally important is microstructure in the subsurface level. It is obvious that crack gets nucleated at the surface, grow into subsurface level causing formation of fragmentation and fall off as debris. In the case of ADI balls, ausferrite matrix in the subsurface level resists the growth of the crack and improves the wear resistance. Srinivasamurthy

Daber and P.Prasad Rao [14] have shown that the retained austenite undergo strain induced martensitic transformation when stressed resulting in increased resistance to crack growth. Such behaviour of retained austenite is influenced by high carbon content which stabilises the retained austenite. In short, in ADI balls, because of ausferrite structure, hard surface is well held in position by tough subsurface materials during grinding operation. It is observed in the present investigation that, the acicular ferrites which are the product of low temperature austempering are known to possess superior wear resistance compared to feathery ferrites which are the product of higher austempering temperature.

At 280ºC for 30 minutes the fracture toughness of ADI is 32mpa√m. The best fracture toughness is obtained when retained austenite content is about 30 volume percent and its carbon content is more than 1.8 weight percent [17]. In the present study the values of retained austenite and carbon content of retained austenite is almost similar to the above mentioned values and hence the toughness and wear resistance of ADI.

In the case of ADI it is the toughness combined with hardness which improves the wear resistance. The hardness values in the table 4 shows that the ADI austempered at 280ºC for 30 minutes possesses 444 BHN as against the forged En31 steel which possesses 529 BHN. Even though the hardness of the En31 steel is more, its wear resistance is less during grinding operation in the ball mill. Based on these results, it is concluded that, it is not only hardness but also toughness which plays an important role in increasing the wear resistance of ADI.

4.4 Grinding efficiency

Sieve analysis results given in Table 5 reveals that the grinding efficiency of the ball mill is superior with ADI balls compared to that with forged En 31 steel balls. It is a known fact that only a part of the energy supplied to the mill is utilized in affecting the comminution of the ore particle and rest of the energy was lost in the form of sound, vibration and heat. When balls are impinging on each other in impact zone causes the size reduction of ore particles. ADI as grinding media materials, due to the presence of free graphite in the matrix are known to dampen the vibration and causes minimum rebounding thus transferring more energy for the process of comminution. This is the probable reasons that ADI balls offering higher grinding efficiency compared to En 31 forged steel balls.

In the present investigation the lower ausferrite microstructure does not reveal the presence of carbides, where as the En31 steel possess large amount of carbides due to the presence of high chromium content. It is a known fact that the presence of carbides in ferrous materials provides easy path for fracture propagation due to the impact or sliding action which normally occurs in ball mill.

5. Conclusions

The following conclusions were drawn from the present study on wear behaviour of ADI as media material in grinding iron ore:

-- The ADI balls properly heat treated found to be a suitable material as grinding media in comminution of iron ore.

-- Among the ADI balls the samples austenitised at 900ºC for one hour and austempered at 280ºC for 30 minutes offer better wear resistance during wet (closed mill) grinding.

-- ADI balls offers better wear resistance when compared to forged En 31 steel balls.

-- The wear behaviour of ADI in grinding of iron ore is not only due to the hardness but also due to its unique microstructure.

-- The wear loss was found to be low in the slurry with higher pH values.

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