Evaluation of Thermally-Assisted Fracture of Particles Using Microscale Fracture Measurements

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Abstract

Thermal pretreatment can significantly improve industrial comminution operations by reducing energy requirements. The effect of pretreatment using conventional and dielectric heating followed by quenching on the microstructure and on the fracture characteristics of selected single-phase and multiphase materials has been investigated using single-particle fracture experiments, crack density measurements and indentation fracture. It was shown that measurements of the crack density from polished sections correlated very well to a parameter related to material integrity, called damage, which makes the latter a very good tool to assess pretreatment. It has been observed that very significant reductions in both fracture energy and material integrity were achieved by heating followed by water quenching.

1. Introduction

Thermal pretreatment was identified early as a potential method to improve comminution and liberation characteristics of ores by weakening the rock and inducing grain-boundary fracture. It takes advantage of the differences in thermal expansion and contraction in single anisotropic grains or in adjacent grains as well as on rapid contraction produced by quenching, resulting in the development of localized stresses and damage.

Thermally assisted comminution has attracted the attention of several researchers since the 1920's, and many studies have been published on a variety of geological materials including quartz, tin ore and iron ores. Comprehensive reviews of the literature can be found elsewhere (Geller and Tervo, 1975; Fitzgibbon and Veasey, 1990). Most recently, interest in thermal treatment has been renewed due to the capabilities of microwave energy in promoting rapid and selective heating of different mineral phases. Improvements in the grindability of iron ores, including taconite, have been reported and the results are encouraging (Walkiewicz et al., 1995).

Although several aspects of the effect of thermal pretreatment on comminution were demonstrated, the techniques used did not provide any direct information on its effect on the ore microstructure. Further, one common method of quantitatively assessing the effect of thermal pretreatment – the Bond Work index method – has several shortcomings (Austin et al., 1984), particularly regarding the assumption that the shape of the size distribution during grinding does not change such that only the 80% passing size is considered. This is a particularly severe assumption as changes in fragmentation pattern have been often observed in thermally treated products (Brown et al., 1958).

An alternative to standard crushing and grinding experiments is single-particle fracture. Single-particle fracture experiments are conducted under controlled, reproducible experimental conditions and provide fundamental information that characterizes particle fracture. A device, called Ultrafast Load Cell (UFLC), developed at the Utah Comminution Center, allows fast and accurate measurement of fracture characteristics of brittle material subjected to impact fracture (King and Bourgeois, 1993a; Tavares and King, 1998). In particular, it provides a method for the measurement of the minimum energy to fracture (also called the particle fracture energy), the damage parameter

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and the single-particle breakage function. Data measured using the UFLC provides a basis for the calculation of the rate of breakage and the breakage function (King and Bourgeois, 1993b), which are used in the modeling and simulation of large-scale comminution machines.

In a recent work it was demonstrated that particle fracture energy measurements with the UFLC were consistent with Bond Work index measurements for a thermally treated quartzite (Pocock et al., 1998). Reductions in particle fracture energy were, however, significantly greater than reductions in Bond Work index. This shows that smaller but significant effects of pretreatment, that would not be detected using standard grindability experiments, can be accurately measured given the high resolution of the UFLC.

In the present work the effect of several variables involved in thermal pretreatment are studied and their effect on the breakage characteristics of materials is investigated through fracture experiments using the UFLC. The relevance of the damage parameter to assess internal material integrity is demonstrated and the effect of thermal pretreatment on the fragmentation pattern is investigated.

2. Experimental

2.1 Materials

Samples used in the experiments consisted of borosilicate glass spheres, quartz (Karlsruhe, Germany), fluor-apatite (Cantley, Québec), specular hematite (Québec), iron ore (Newfoundland), titanium ore (Norway) and copper ore (Utah). Examination of polished sections in an optical microscope indicated that the single-phase materials consisted mostly of single-grained, high-purity particles. The quartz sample was from a riverbed deposit and contained particles exhibiting nearly spherical shapes. Fluor-apatite particles presented a very high concentration of pre-existing micro-cracks in the original material. The iron ore sample consisted mostly of polycrystals of hematite, magnetite and quartz. More details can be found elsewhere (Tavares, 1997). Samples were screened in narrow size intervals using round-mesh precision sieves for thermal treatment and fracture testing.

2.2 Thermal treatment experiments

Conventional heating experiments were undertaken using a batch electric furnace (Thermoline 10500) where samples of about 10 grams were heated at variable temperatures in porcelain crucibles. Low heating rates (about 20°C/min) were used in the tests, ensuring uniform temperature throughout the sample. These heating rates were achieved by manually changing the furnace temperature setting by discrete increments. Samples were immediately withdrawn from the furnace upon reaching the set temperature (within ±5°C).

Dielectric heating tests were only used for samples containing high-loss minerals. Experiments were carried out in an LBM 1.2A batch laboratory microwave oven (by Cober Electronics, Inc.), which operates at 2.45 GHz and allows continuous control of power from 0 to 1.2 kW. The sample volume and position of the mode-stirring fan were carefully selected prior to each pretreatment experiment in order to achieve maximum heating rates. In the present study, microwave heating experiments were conducted at 1200 W for 30 seconds.

Sample temperatures during both microwave and conventional heating experiments were monitored using a type K thermocouple with an ungrounded tip sheathed in stainless steel. Samples were quenched immediately upon withdrawal from the furnace in still air or by immersion in water (or salt solution) at room temperature (15-25°C).

More details on the equipment and procedure used in the thermal treatment experiments can be found elsewhere (Tavares, 1997).

2.3 Microfracture experiments

Apparent crack density measurements were undertaken using an optical image analysis system. It consisted of an optical microscope, a CCD camera and a microcomputer. The digital images were analyzed using VIDAS, a PC image analysis software by Kontron Systems, Inc. The procedure used to measure the apparent crack density consisted of background correction, delineation, segmentation, skeletonization, filtering, feature identification and crack length measurement. Apparent crack densities were then given from the ratio between the total crack length measured and the scanned area.

Indentation fracture experiments were carried out using a Leco M-400 micro-hardness tester. A sharp Vickers indenter, with loads ranging from 50 to 500 g, was used in the experiments. Both the indent dimensions and the lateral extent of cracks associated with the indents were measured using the calibrated eyepiece mounted on the hardness tester within about one minute of the indentation. Indentation tests were used to calculate the Vickers hardness and the surface fracture energy, given by
\[ H_i = 1.854 \frac{F}{d^2} \]  
(1)

and

\[ r_i = \frac{K_f^2 Y}{2(1-\mu^2)}, \quad \text{with} \quad K_f = \chi \frac{F}{\xi^{3/2}} \]  
(2)

where \( F \) is the indentation force, \( d \) is the average diagonal of the plastic indentation, \( Y \) is the modulus of elasticity of the material, \( \mu \) is the Poisson’s ratio, and \( \xi \) is the average distance, measured along the diagonal of the indent, from the center of the indent to the extremity of the crack. \( \chi \) is a term that incorporates factors involving the indenter geometry, friction and free-surface effects (Lawn, 1993; Middlemiss and King, 1994).

Sample preparation for both crack density measurements and indentation fracture tests consisted of mounting several grams of the material in epoxy and polishing using abrasive materials down to 0.3 \( \mu \)m alumina.

Impact-fracture experiments were undertaken using the Ultrafast Load Cell (UFLC). The UFLC is a hybrid between the drop weight apparatus and the split Hopkinson pressure bar (King and Bourgeois, 1993; Tavares and King, 1998). Tests are conducted using individual particles and are used to estimate quantities such as the mass-specific fracture energy and the particle stiffness (Tavares and King, 1998). The size distribution of the progeny was determined using round-mesh precision sieves.

3. Effect of thermal shock pretreatment on particle microstructure

In order to gain insights into the role of thermal pretreatment on the fracture of particulate materials, selected single-phase materials were subject to thermal-shock pretreatment, and their microfracture as well as their single-particle fracture characteristics were examined. Samples of riverbed quartz (4.0-4.75 mm), hematite (2.0-2.8 mm) and glass (3 mm) — an ideal model material — were subject to conventional heating at different preheat temperatures followed by water quenching at room temperature.

Examination of polished sections of quartz under the optical microscope (Fig. 1) shows that pretreatment is able to induce cracks that can be identified and also that the severity of the thermal pretreatment determines the intensity of the cracking. In order to assess quantitatively these effects, the image analysis system was used to estimate the apparent crack density in the polished section. Results given in Figure 2 show that the density of cracks is, indeed, function of
the severity of the pretreatment used. The greater the severity of the shock, the higher the density of crack induced. At the magnification used, however, it appears that regions between the cracks appear intact. This is particularly relevant when thermal pretreatment is used prior to size reduction, because it would mean that as grinding progresses and fragments become smaller than the regions fully surrounded by cracks, they would essentially behave as intact particles and would, therefore, require as much grinding energy for additional size reduction. It is possible, however, that thin – and closed – cracks were present but could not be detected with the optical system and that heat treatment released residual stresses in the material.

Indentation is a very good probe of the material microstructure and is here used to characterize the integrity of regions of the material that were apparently undamaged by pretreatment. Using small indents (with diagonals of 16 μm for quartz, 43 μm for hematite and 55 μm for glass) surface-fracture energies and Vickers hardness were determined. Results summarized in Figure 3 show that the Vickers hardness was little or not affected by thermal pretreatment. Similar results were observed for the surface-fracture energy and are given elsewhere (Tavares, 1997). Therefore, in the scale of the indents, the microstructure did not appear to be significantly affected by pretreatment and that it was largely limited to inducing a macroscopic array of cracks in the particles, as is shown in Figure 1 for quartz. This may suggest that thermal shock essentially only extends preexisting cracks in the material and does not apparently nucleate new ones. Nevertheless, small reductions in the hardness and surface fracture energy of glass and hematite, as well as the smaller fraction of ideal indents in some heat-treated samples (1/5 for glass heated to 600°C and quenched versus about 4/5 for untreated glass, for example) suggest that some relief of residual stresses may have occurred, and/or that closed cracks may still be present in the material.

Measurement of crack density, therefore, gives a good indication on how the material is affected by thermal-shock pretreatment. However, these measurements have several limitations: they are critically dependent on the sensitivity of the optical method of detecting cracks, on surface preparation, illumination and magnification. Further, they represent an invasive measurement and are not a convenient tool to systematically assess pretreatment.

Cracked solids are more compliant (have lower stiffness) than intact solids. This is illustrated in Figure 4, which shows impact-breakage results for quartz before and after thermal pretreatment. By measuring the stiffness of a material before and after pretreatment, the fractional reduction of the cross-sectional area that has been stress-relieved by cracks and that is unable to withstand load can be estimated by (Tavares and King, 1998; Kachanov, 1958)

$$D = 1 - \frac{k_d}{k_u}$$  \hspace{1cm} (3)

where \(k_u\) and \(k_d\) are the stiffness of the particles before and after pretreatment.

For the data in Figure 4 and Equation 3, we can calculate the damage induced in the particle as 0.62, which shows that thermal pretreatment destroyed the integrity of 62% of any cross section of the particle. This is consistent with the intense cracking observed in the micrographs from Figure 1.
Fig. 4 Force-deformation profiles for untreated and heated to 1000°C and water quenched quartz particles (4.0-4.75 mm).

Data from Figure 4 represent only two experiments and the estimated damage value is only valid for illustration purposes. In practice, considerable scatter exists in the fracture characteristics of single particles, requiring the testing of samples of at least 50 particles (Tavares and King, 1998). Damage values were then calculated from average particle stiffness for several materials and are compared to the measured crack densities in Figure 5. Although a unique (material-independent) relationship does not apparently exist, a good correspondence between the measured damage values and the apparent crack density is evident. The significance of this correspondence is that the extent of the microcracking induced by any given pretreatment method could be assessed simply from testing untreated and pretreated samples in the UFLC. This is significant not only when the enormous effort involved in measuring crack densities from polished sections is considered, but also considering the non-absolute character of those measurements, given their invasive nature.

4. Effect of thermal treatment on fracture characteristics

With the use of the damage parameter (Equation 3) and the particle fracture energy, the effect of relevant thermal pretreatment variables such as temperature, quenching medium and particle size on the weakening could be studied in detail.

4.1 Effect of heating temperature

Heating temperature is recognized as the most important variable affecting thermal-shock weakening. It not only controls the magnitude of the thermal stresses induced during rapid cooling but also determines the energy expenditure in heating, which ultimately establishes the feasibility of thermally assisted comminution.

Fracture characteristics of selected single- and multiphase materials heated to a range of temperatures and quenched in water have been measured, and results are summarized in Figures 6 and 7. Residual fracture energy is here defined as the ratio between the mean particle fracture energy of the pretreated particles and mean particle fracture of the untreated particles. Figure 6 shows that heat treatment produces significant reductions in particle fracture energy even at moderate preheat temperatures, result of the superficial cracks induced that offer sites for crack propagation leading to fracture, particularly in the more brittle solids. In some instances, however, little
additional reduction in fracture energy is achieved by increasing the heating temperatures above a certain level, in spite of the greater proportion of crack-initiating sites present, as evident from the higher damage values. A possible explanation is that, in highly cracked solids, fracture is not controlled by crack initiation. A crack that initiates in the particle may arrest in the microstructure and not contribute to failure. Fracture of these materials will only occur when a significant number of pre-existing cracks are activated, thus allowing the propagation of a crack through the entire particle.

Lowering the particle fracture energy as a result of pretreatment only ensures higher breakage rate of the parent in a comminution machine and does not necessarily lead to a reduction in overall comminution energy. Substantial volume cracking, on the other hand, ensures lower energy expenditures not only in breakage of the parent particle but also in the breakage of several generations of progeny fragments. Therefore, substantial energy savings do require a substantial increase in the proportion of damage in addition to the reduction in fracture energy. Figure 7 shows that significant damage occurs only after pretreatment at higher temperatures. The significance of this result is that, in order to achieve substantial gains in comminution greater expenditures in heating must be made. Incidentally, this has been the major limitation for the industrial application of thermally assisted comminution.

4.2 Effect of quenching medium

A promising approach to reducing energy expenditure in thermal pretreatment is the use of more severe quenching media that could allow greater weakening at lower temperatures. Several quenching media exist that allow greater heat transfer and faster cooling than water (Luty, 1993). Brines (salt solutions at high concentrations) have up to 75% higher heat-transfer coefficients and therefore allow significantly higher cooling rates than pure water (Luty, 1993). This is because the duration of the less efficient stage in quenching with liquids that boil — called vapor blanket — is reduced and almost eliminated. The effect of quenching in a 10% sodium chloride solution (in weight) on the fracture energies of quartz and copper ore particles is compared to water quenching, and results are summarized in Figure 8. No significant difference was found between water and salt-solution quenching, so that the application of the corrosive sodium chloride-containing solution as quenching media is not justified. Similarly, no statistically significant increase in the amount of damage was observed with salt quenching compared to water quenching.

In spite of its lower heat-transfer coefficient in comparison to water, air-cooling has the advantage of more effectively allowing partial recovery of the input energy when, for example, vertical shaft furnaces are used. The energy recovered could then be recycled and reduce energy input in thermally assisted comminution. Comparisons between air and water quenching are given in Figure 9. Comparable fracture energy reductions are obtained for copper ore with air and water quenching. The mismatch between the various crystals in the polycrystalline copper ore particles appear to be responsible for the weakening even without a severe thermal shock. Nevertheless, greater reductions in fracture energy are still achieved with water quenching. On the other hand, no statistically significant weakening was observed after heating and...
cooling the monolithic quartz particles in still air. This is true even after heated above its α→β transformation temperature, generally considered as the reason for the greater susceptibility of quartz to thermally assisted comminution (Kanellopoulos and Ball, 1975; Harirham and Venkatachalam, 1977). In the absence of chemical degradation, single-phase materials consisting of single crystals would essentially expand and contract elastically during heating and cooling, respectively, and therefore would not sustain permanent damage.

4.3 Effect of particle size

The effect of particle size has been studied by subjecting narrow size fractions of riverbed quartz to heating and water quenching and testing in the UFLC. Figures 10 and 11 show that the magnitude of the weakening is relatively little affected by particle size in the interval studied (0.25 to 5 mm). The amount of damage induced by thermal pretreatment, on the other hand, generally decreases for smaller particle sizes.

4.4 Effect of heating method

Microwave heating offers an alternative to conventional heating as it allows fast and selective heating of the high-loss phase in the ore. The rapid expansion of the absorbing phase produces stresses that can lead to internal cracking and ultimately lower energy consumption in size reduction.

Both dielectric and conventional heating experiments were undertaken with selected ores containing high-loss minerals, and results are summarized in Table 1. The ores were heated to about 600°C and were subsequently either cooled in air or quenched in water. Moderate heating rates were achieved, with the iron ore sample being heated for 30 s to 612°C, taconite to 588°C, and titanium ore to 566°C during the microwave heating experiments. Significant reductions in particle fracture energy and increases in damage were obtained for the thermal-shock samples.

| Material and size | Heating method | Quenching Medium | Reduction in fracture energy (%) | Damage (%) |
|-------------------|----------------|------------------|-------------------------------|------------|
| Iron ore 4.5-5.35 mm | Conventional | Air | 25 | 29 |
| | Microwave | Air | 36 | 0 |
| | Conventional | Water | 24 | 47 |
| Titanium ore 4.0-4.75 mm | Conventional | Air | 29 | 0 |
| | Microwave | Air | 28 | 51 |
| | Conventional | Water | 50 | 52 |
| Taconite 5.0-5.95 mm | Conventional | Air | 28 | 0 |
| | Microwave | Air | 29 | 0 |
| | Conventional | Water | 56 | 51 |

Fig. 9 Effect of quenching medium on the fracture characteristics of 4.0-5.0 mm copper ore.

Fig. 10 Effect of particle size and heat treatment and water quenching, on the residual fracture energy of quartz.

Fig. 11 Effect of particle size and heat treatment on the damage parameter of quartz.
while limited weakening was observed for air-cooled specimens. Such reductions in fracture energy, however, were comparable to those obtained by heating slowly (at about 20°C/min) in the muffle furnace. Further, no damage was induced by heating taconite and iron ore samples with microwaves, while substantial damage resulted from microwave heating the titanium ore.

Thermomechanical stresses that led to weakening during microwave pretreatment result from the interplay between the thermal expansion of the high-loss mineral and the dissipation of heat by conduction to the neighboring grains. It has been shown (Salsman et al., 1996) that the magnitude of the tensile stresses in the boundary between the high-loss and the gangue mineral would depend critically on the heating rate that can be achieved during heating. The temperature rise depends on the power dissipated in the mineral, which in turn is proportional to the electrical field strength. Therefore, higher powers of the microwave heating system must be used to enhance the weakening and therefore make microwave pretreatment a viable proposition.

5. Effect of Thermal Treatment on Fragmentation and Comminution

Progeny size distributions from breakage of single particles of untreated and heat-treated hematite particles are shown in Figure 12. Similar trends were observed for the others materials tested. Consistently with measurements of internal damage and particle fracture energy (Figures 7 and 8), increases in heating temperature produced greater breakage. However, the fragmentation pattern resulting from breakage of heat-treated particles differed significantly from that found with untreated particles. Heating to higher temperatures produced a significant increase in the fragmentation of coarser sizes with little or no increase in the proportion of fines. This confirms observations from polished sections, which show that as soon as fragments become smaller than the regions fully surrounded by cracks, the effect of thermal pretreatment vanishes.

These were, indeed, confirmed by batch grinding experiments, carried out on a 19-cm ball mill with 1.9-cm, 30-gram steel balls operating at 75% critical speed (Figure 13). Breakage of the heat-treated material results in bimodal size distributions with greater spread of sizes in the product.

Although thermal pretreatment can induce significant cracking and reduce energy expenditure in comminution, the net energy expenditure is usually not favorable (a rough estimation based on thermal capacities shows that heating above 600°C would require well over 100 kW-hr/ton). Therefore, thermal pretreatment cannot be justified on the basis of reduction in grinding energy alone. It could be used in some specialty applications. Excessively competent pebbles only suffer wear inside the mill and do not break. Preconditioning the pebbles using conventional or microwave heating could be used to control their competency, thus allowing an optimum performance of the mill.
Summary and Conclusions

The application of thermal predamage to improve comminution has been studied using a microscale approach involving precise single-particle breakage measurements with the UFLC and microscopic examination. It was shown that pretreatment was essentially responsible to inducing an array of cracks in the solid, with its concentration depending on the severity of the treatment. It was found that a correspondence exists between the crack density and the reduction in material stiffness, given by the damage parameter. Weakening was higher for coarser particles and for severe quenching, with water or salt solution.

Reductions in the measured particle fracture energy indicate weakening of the parent particle due to induced crack-like damage but, in spite of this, substantial reduction in comminution energy can be achieved only if substantial numbers of the progeny fragments are themselves weaker than the undamaged material. When this occurs, substantial damage can be measured in the particle in addition to reductions in particle fracture energy.

The studies showed that limited reductions in fracture energy can occur after thermal-shock pretreatment to temperatures as low as 200°C. However, substantial damage was found to occur at temperatures of 600°C and above. The amount of damage induced was found to decrease with a reduction in particle size. Microwave heating experiments at 1.2 kW showed little or no improvement when compared to thermal pretreatment by conventional heating.

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Marcelo Tavares graduated with First Class Honors in Mining Engineering from the Universidade Federal do Rio Grande do Sul, Brazil. In 1991 he obtained a MSc degree in Metallurgical Engineering at the same university. He joined the Comminution Center in the University of Utah, where he obtained his PhD degree in 1997. He is currently an assistant professor in the Department of Metallurgical and Materials Engineering at the Universidade Federal do Rio de Janeiro, where he is involved in both teaching and research in several topics in mineral processing, particularly comminution and gravity concentration.

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Professor was born in South Africa and received BSc and MSc degrees from the University of the Witwatersrand. He received the PhD degree from the University of Manchester, England in 1963. He has lectured at the universities of Manchester, Natal, Witwatersrand, Utah and at the Camborne School of Mines. He established and led Mintek's Chemical Engineering Research Group at the University of Natal for 7 years and was Head of the Department of Metallurgy and Materials Engineering from 1975 to 1990. He is now professor of Metallurgical Engineering and Director of the Comminution Center at the University of Utah. Professor King has researched and published widely in the field of quantitative modeling of process engineering systems and has contributed particularly to the development of computer simulation techniques for the minerals processing industry. Professor King is an Honorary Life Member of the South African Institute of Mining and Metallurgy and he was President of that institute in 1982 – 1983. He is a member of SME and a member of the Society for Industrial and Applied Mathematics. Since 1999 he has been the Editor-in-Chief of the International Journal of Mineral Processing.