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Temperature of the milling balls in shaker and planetary mills

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Abstract The temperature increase of the milling balls in two laboratory mills, frequently used for mechanical alloying and mechanochemical experiments, was studied using direct calorimetric measurements. The ball temperature remains below 100 °C in a SPEX 8000 shaker mill and it is cooler when flat-ended rather than round-ended vial is used, although the milling intensity, as measured by the mechanical dose rate, follows the opposite trend. Temperatures over 200 °C are typical in planetary mills operating at similar milling intensities. It is suggested that the higher ball temperatures result from more oblique collisions and friction, while the lower temperature but higher intensity of the shaker mill with flat-ended vial is due to the larger proportion of frontal impacts.

Introduction

Mechanochemistry is considered a separate branch of chemistry because mechanical work causes chemical reactions directly and the mechanochemical reactions are often different from those initiated by heat [1]. The ability of mechanical alloying (MA) to make unique metastable phases (e.g., amorphous alloys far from deep eutectics) also relies on carrying out the process in the solid state near ambient temperature, where the limitations of the phase diagram can be circumvented [2]. Any temperature increase during mechanochemical processing or MA can alter the process and the product. Therefore, many high-energy ball mills used to carry out these processes utilize water or forced air-cooling and longer milling runs are often divided into shorter periods separated by cooling breaks to avoid excessive heating.

Nevertheless, temperature effects do play a definite role in ball milling. For example, Cherdyntsev et al. has shown that larger ball size, and the consequent higher mill temperature, promotes alloy formation in a system with negative heat of mixing (Fe–Mn) while slows it down in the Cu–Cr system that has positive heat of mixing [3]. When milling an 80–20 mixture of Pd and Si powders, amorphization was observed at low milling intensity, while crystalline Pd_3Si formed at higher intensity [4]. Similarly, the partial crystallization of a glass-forming Ni–Zr alloy at high milling intensity was attributed to increased mill temperature [5].

The temperature of the milling balls of a water-cooled AGO-2 planetary mill was measured by Kwon et al. recently [6]. Ball temperatures over 600 °C were reached when running the mill for 20 min at the highest intensity setting. Model calculations by Shelekhov et al. for the same type of mill also predicted ball temperatures as high as 500 °C [7]. These are average temperatures; the impulsive temperature increase of the powder compressed between the colliding milling tools may add an additional 50–300 °C to give the highest local temperature [8]. Obviously, the presence of such high temperatures has major implications, yet it receives relatively little attention. One reason may be that a large fraction of the investigations in MA employ shaker mills (such as the SPEX 8000 Mixer Mill) where the problem of temperature increase is assumed to be less serious [9] although—as far as we know—no direct measurement of the ball temperature has ever been carried out to confirm this conjecture.

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In this paper, results are reported on the average ball temperature in a SPEX 8000 Mixer Mill and a Fritsch P-5 planetary mill. It is shown that while the temperature remains well below 100 °C inside the shaker mill under typical operating conditions, temperatures much over 200 °C are easily possible in planetary mills.

Experimental details

The average temperature of the milling balls was deduced from the heat content of the balls, measured with a simple calorimeter using water as the working medium. At the end of a test run, the milling container was removed, opened, and the warm balls were directly emptied into the calorimeter. The entire procedure took less than 15 s when using the SPEX mill and only slightly longer with the planetary mill; heat transfer from the balls during that time could cause at most a 1 °C decrease of the ball temperature. Most tests were carried out without any powder in the milling container in order to avoid the possibility of a chemical reaction between the water in the calorimeter and some activated powder. A few runs were done using inert SiO_2 powder as the milled medium.

The SPEX 8000 Mixer Mill was tested with both flat-ended and round-ended vials, both made of hardened tool steel. Steel balls, 12.7, 9.5, and 6.4 mm in diameter were used. This mill was cooled only by natural air convection. The temperature of the milling vial was measured by a K-type thermocouple attached to the surface of the vial. It was also confirmed by independent calorimetric determination in a few separate experiments. The Fritsch P-5 planetary mill was run with 12.7-mm steel balls at several rotational speeds and using two bowl sizes, 10 and 7.5 cm in internal diameter. The number of balls was 100 and 50 per bowl, respectively. In a few experiments, 4.76-mm balls with the same total mass were used. The mill had built-in forced-air cooling.

Results and discussion

The temperature of the milling balls, as measured after running the SPEX mill for the indicated intervals, is shown in Fig. 1. A flat-ended vial with five 12.7-mm steel balls and no processed powder were used in this experiment. The temperature of the balls increased to about 66 °C. Although this temperature increase may still influence the result of a ball milling experiment, it is an order of magnitude less than the temperatures found for the AGO-2 mill [6]. The temperature of the milling vial also increases, but the temperature of the balls—and consequently the mean temperature of the contents of the vial—is about 25 °C warmer than the vial.

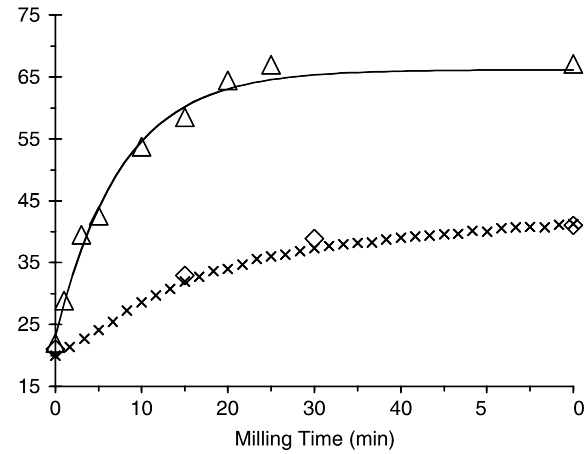

Fig. 1 Ball temperature as a function of milling time when using a SPEX mill and flat-ended vial with five 12.7-mm balls and no powder (Δ). The continuous line represents a fitted exponential. The temperature of the vial as measured by a thermocouple (×) and independent calorimetric determination (◇) is also shown for comparison

The milling time dependence of the ball temperature was fitted to an exponential approach to a steady state temperature. The limiting temperature and the characteristic time are listed in Table 1 for several combinations of ball size and number. Within the investigated selection of parameters, the steady state temperature increases as the total mass of the balls increases. Of course, too many balls impede the motion of each other limiting the dissipated mechanical energy and consequently the maximum temperature, just as the highest milling efficiency is achieved with six or seven 12.7-mm balls and declines if the number of balls is increased further [10]. Therefore, it is unlikely that a ball temperature over 100 °C could be generated by

Table 1 Steady state temperature (T_{max}) and characteristic time of heat-up (τ) for several ball number (N), diameters (D), and total mass m_{tot} combinations

N	D (cm)	m_{tot} (g)	T_{max} (°C)	τ (min)
1	12.7	8	48	16.7
2	12.7	16	52	17.9
3	12.7	24	60	6.0
5	12.7	41	66	7.6
7	12.7	57	74	8.6
5	0.95	17	54	7.4
10	0.95	35	65	6.1
12	0.95	42	68	4.4
8	6.3	8	44	10.8
24	6.3	24	58	4.2
5	12.7	41	78	6.5

A SPEX 8000 mill with flat-ended vial was used, except for the last row that represents data for a round-ended vial using a larger total mass of balls or a mixture of balls with different diameters.

If the other conditions are the same, the ball temperature obtained with round-ended vial is significantly higher than that obtained with flat-ended vial (Fig. 2 and last row of Table 1). This finding is quite surprising, because the milling efficiency, as measured by the ''mechanical dose'' delivered to the processed powder, is about 50% higher when using flat-ended vial. It seems that the mill delivers energy in at least two ways and one contributes more to the mechanical dose delivered to the powder while the other dissipates more energy in the form of heat. In their simulation of a planetary mill, Shelekhov et al. identify ''frontal'' and ''tangential'' contributions to the energy dissipation [7] that can loosely be associated with plastic deformation due to compression versus heating by friction. Although the tangential component also produces deformation and part of the energy delivered by compression is dissipated as heat, it seems that a larger fraction of oblique collisions results in more heating.

Indeed, for a given total mass of the milling balls, the least heating and consequently the slowest temperature increase results when a single ball is bouncing back and forth in a flat-ended vial, making mostly frontal impacts. When the number of balls is larger, many oblique ball-ball and ball-wall collisions occur, making the heat-up faster. If the total ball mass is the same, the final temperature is relatively insensitive to the number and size of the balls.

In a round-ended vial, much friction and heating is caused by balls rolling around the end surfaces and by a large fraction of the collisions being highly oblique. As a result, both the heating rate and the steady state temperature increase. At the same time, the rate of mechanical activation is significantly lower, as shown earlier [11].

Even more rolling and friction take place in a planetary mill. The results presented in Fig. 3 were obtained using a Fritch P-5 mill with two bowl sizes and rotational speeds. The final temperatures are indeed much higher than the temperatures obtained with the SPEX mill. The temperature could be increased further via increasing the speed of the mill. In fact, the highest temperature obtained with the smaller bowl was 237° C at 318 rpm. A decrease of the temperature is expected at very high speeds, as the balls stay attached to the container wall for too long, reducing both heating and the efficiency of milling [12].

These results compare favorably with the computer simulation of Shelekhov et al. who predicted temperatures as high as 500° C for the more energetic AGO-2 planetary mill [7]. According to their work, the form of energy dissipation is a function of the fill fraction, ζ, and the highest temperature is anticipated at relatively low fill factors of about 15–20%, where friction is the main form of energy dissipation. The fill factor in our experiment is about $\zeta = 20\%$ for the larger bowl and $\zeta = 18\%$ for the smaller bowl, therefore, we were operating in the friction dominated regime. The temperatures in this study are somewhat lower than the temperatures (up to 430 °C) obtained by Kwon et al. for step II of the AGO-2 mill (the one closest in intensity to the P-5 mill) [6].

All the above data were obtained without any powder in the milling vial. The addition of a few grams of SiO_2 decreased the final temperature as well as increased the time needed to reach a stationary state (Fig. 2.) This is a complicated phenomenon, as the properties of the charge influence the elasticity of the collisions and the motion of the balls as well as the generation and transfer of heat. A more detailed investigation of how different materials

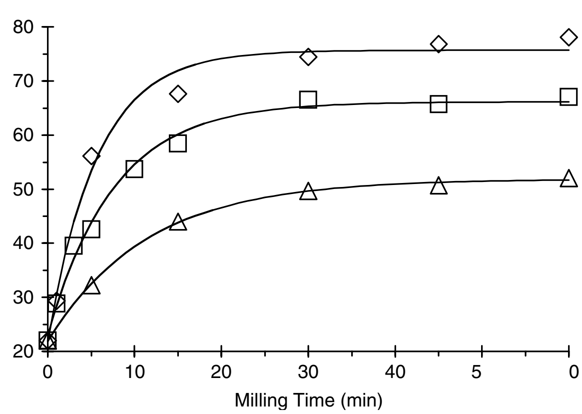

Fig. 2 Comparison of the ball temperature—milling time curves for a SPEX 8000 mill using five 12.7-mm balls and flat-ended vial with no powder (□), a round-ended vial with no powder (◇) and a flat-ended vial that also contains 4 g of SiO_2 powder (Δ)

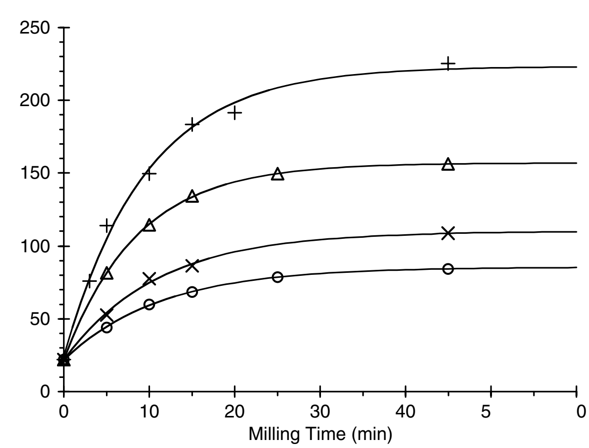

Fig. 3 Ball temperature—milling time curves obtained using a Fritsch P-5 planetary mill. The + and × symbols represent data with a 10-cm diameter milling bowl and 100 balls and 280 rpm (+) and 200 rpm (×) speeds and the Δ and ○ symbols correspond to a 7.5-cm bowl, 50 balls and 280 rpm (Δ) and 200 rpm (○) speeds

affect the temperature inside the milling vial was beyond the scope of this study. However, it has been shown by Magini [13] that any powder present in the vial makes the collisions of the balls less elastic and thereby slows the motion of the balls and reduces the dissipated energy.

Conclusions

Direct calorimetric measurements on the milling balls of shaker and planetary mills indicate that the temperature inside the milling vial does not parallel the milling efficiency as measured by the rate of mechanical dose. Consequently, whenever the temperature may be high enough to influence a mechanochemical reaction or alloying process, it has to be considered as an independent parameter.

Much of the heating results from friction during oblique collisions. As a result, vibratory and shaker mills, such as the SPEX 8000 Mixer Mill, run at relatively low temperature (<100 °C) while the temperature in planetary mills (and most likely attritors) is much higher.

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