

Fabrication of Nickel Coatings on Zirconia Balls by Mechanical Ball Milling and the Process Analysis

Lijia CHEN, Haibin CHEN*, Rongyong LI, Weifeng HE, Yuhui YANG

School of Mechanical Engineering, Dongguan University of Technology, No.1 Daxue Rd, Songshan Lake, Dongguan, 523800, China

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The Ni coatings were prepared from nickel metal powder and zirconia ceramic balls by mechanical coating technique. The relationship between rotation speed and coatings thickness was studied, the thickness of the coatings was characterized by the weight increase of the zirconia balls after mechanical coating. The composition and microstructure of the coatings with maximum thickness were analyzed by scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), and X-ray Diffraction (XRD). The results revealed that the formation of coatings consists of thickening from the cold welding between Ni particles and the thinning from the exfoliation of coatings work hardening. The thickness of the coatings reaches the maximum when the above two process progress are close to each other. The results proved that higher speed was favor of the coatings formation, the maximum thickness was obtained at the 15 h for rotation speed of 240 rpm, and microscopic images show that the average thickness was about 20 μm .

Keywords: mechanical coating technique, rotation speed, cold welding, Ni coatings.

1. INTRODUCTION

In the process of preparing materials by mechanical ball milling, due to the continuous impact and friction of the milling balls, the powder gradually adheres to the surface of the milling balls and causes unavoidable contamination at the end of the process. Recently, ball milling, which is well known for mechanical alloying, has found a new application for mechanical coating using the basic principle of the above contamination phenomenon. If the appropriate process parameters were adopted, several functional coatings have been successfully prepared on the surface of the ball milling medium including ceramic or metal substrates [1–5]. On the one hand, the prepared surface coatings can improve the surface properties of the base material, such as hardness. On the other hand, the milling balls can be used as a coating substrate to prepare available functional coatings materials. Among them, the latter has been favored by many researchers in recent years [6–11], and a coating method called Mechanical Coating Technology (MCT) has been developed. Yoshida et al. [6] used this technique to apply a Ti metal coatings on the surface of the Al_2O_3 milling balls, and then obtained a TiO_2 coatings by subsequent oxidation. The coatings have an excellent photocatalytic performance and non-toxic. However, the wide application of TiO_2 was restricted by its wide band gap, and fast electron-hole recombination. In order to further improve the photocatalytic performance of TiO_2 coatings and promote its wide applications, they used a two-step mechanical coating technology (2-Step MCT) to prepare a double-layer composite coatings. Firstly, metal coatings were formed on milling balls using metal powder and ceramic balls were separately as the coating materials and substrate by MCT, and then the TiO_2 powder was used as the coating material,

and the second layer of the oxide coatings, such as TiO_2 , was prepared on the ceramic balls to which the metal coatings has been attached, a double layered coatings has been formed, such as TiO_2/Ti or TiO_2/Cu , etc. [6–8]. In recent years, attention has been paid to improving the photocatalytic performance of TiO_2 by doping Ni, and reports on whether Ni and TiO_2 can be double-layered like TiO_2/Cu are scarce [13–15].

In this work, metal Ni powder was used as raw material to prepare Ni coatings on the surface of ZrO_2 ceramic balls by MCT. In order to explore the coating process of metal Ni forming coatings on ceramic milling balls, the present study focused on the influence of the processing parameters including rotation speed and milling time on the formation of the Ni coatings. Furthermore, the bonding performance between the coatings and the substrate was also studied, and a exfoliation phenomenon after sintering was proposed to describe the bonding strength.

2. EXPERIMENTAL

2.1. Fabrication of Ni coatings

Powder of nickel (Ni) metal was used to prepare coatings on the surface of ZrO_2 ceramic balls by mechanical coating method, the metal powder as the coating material and ceramic balls as the substrate during this experiment. The ball-to-powder ratio (weight ratio of ZrO_2 milling balls to metal Ni powder) was set at 3:5, a total of 80 g was put into a bowl made of alumina with a volume of 250 mL, and then the mechanical coating was performed by a planetary ball mill. The coating schematically is shown in Fig. 1. In the process, the bowls rotates around their own axes and the rotating support disk rotate simultaneously to accelerate the

* Corresponding author. Tel.: +86-769-22861622.
E-mail address: hbchen@dgut.edu.cn (H. Chen)

grinding balls and powder in the bowls. To study the influence of the milling time and the rotation speed on the formation of Ni coatings, the rotation speeds were fixed at 210, 240 and 270 rpm (revolutions per minute), and the milling operation was carried out for 5, 10, 15 and 20 h from the start of the milling operation. During the fabrication, milling operation was performed 10 min followed by 2 min intermittence to avoid the overheating of the bowl and ensure a stable temperature. The raw materials used to prepare the coatings relevant parameters are listed in Table 1. To evaluate the bonding strength between Ni coatings and ZrO₂ balls substrate, the balls coated with Ni metal powder were sintered, and the larger the exfoliation amount of Ni powder is, the weaker the adhesion is.

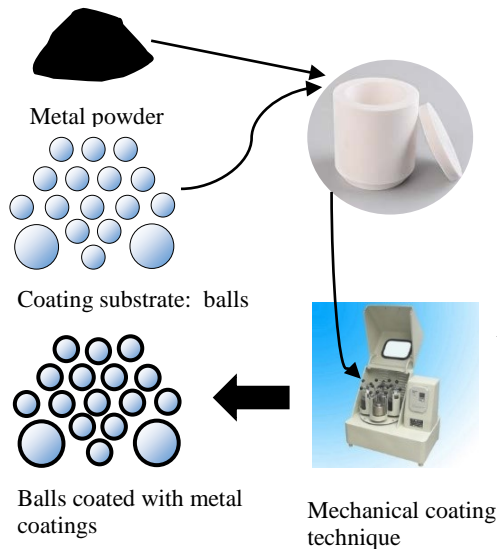


Fig. 1. Schematic diagram of mechanical coating technique

Table 1. Source materials for experimental

Materials	Size	Density	Purity, pct
ZrO ₂ balls	Ø 1mm	6.0 g·cm ⁻³	> 95.0 %
Ni powders	75 μm	8.9 g·cm ⁻³	> 99.5 %

2.2. Characterization

The weight of the milling balls was gradually increased due to the attachment of metal powder on its surface. In the experiment, the average weight increase of the ZrO₂ balls was used to indicate the amount of Ni powder deposited on the surface of the ZrO₂ balls, that is, the larger the weight of the ZrO₂ balls after mechanical coating, the greater the thickness of the coatings. However, when the ZrO₂ balls filling into the bowl, there is no guarantee that the ZrO₂ balls makes the weight exactly 30 g. In order to minimize the error in calculation result, the number of the balls was fixed for each experiment, and the average weight increase rate of all milling balls was used to replace the weight increase. Finally, scanning electron microscope (SEM) (S-3400N, made by Hitachi Company of Japan) was used to observe the surface morphologies and the microstructure of the cross-sections of the Ni-coated ZrO₂ balls with the largest average weight increase rate, the composition in the surface layer of Ni coatings on ZrO₂ balls was measured in SEM by EDS, and X-ray diffraction (XRD) (DX-2500, made by

Haoyuan Company of China) with Cu-Kα radiation at 25 kV and 15 mA was used to characterize the coatings phase component before and after sintering treatment.

3. RESULTS AND DISCUSSION

3.1. Influence of the milling time and the rotation speed

The weight increase of the ZrO₂ balls due to more Ni powder particles coat the balls' surface during mechanical coating at different revolution speeds for different time, was recorded as illustrated in Fig. 2. The weight increase means Ni coatings formation and thickening. We found that the weight increases of the ZrO₂ balls after milling at different speeds for 5 h was: 270 rpm > 240 rpm > 210 rpm. It can be seen that the weight of the milling balls increased with the increase in rotation speed, it is beneficial to the thickening of the coatings. As the coating process continues, the weight increase rate of the milling balls after 5 h shows a trend that the rotation speed of 240 rpm increases faster than that of 210 rpm. However, when the rotation speed was increased to 270 rpm, the weight of the milling balls began to decrease with the increase of coating time.

On the one hand, increasing the rotation speed increases the milling balls' movement and the kinetic energy of the single milling ball. Therefore, local temperature of powder particles was increased quickly because more energy was transferred during the impact of milling balls, which creates the powder particles were deformed and cold welded, and the coating rate was accelerated. On the other hand, due to the increased impacts frequency of the milling balls in the same time period, more powder particles began to move and adhere to the surface of the milling balls under the impact of the milling balls, accelerating the formation of the coatings. This suggests that a higher revolution speed can accelerate the coating of Ni powder particles on the surface of milling balls, which is consistent with the results of other studies [16, 17].

It has been reported that the cold welding and fracturing of the metal powder particles are two opposite processes that occur simultaneously during the mechanical ball milling coating. In other words, the coating was exfoliated from the substrate, there is still powder that continues to be cold welded to the surface of the milling balls, that is, formation and exfoliation of coating occur at the same time. In the theory, the coating thickness will remain unchanged if the rate of the above two processing reaches equilibrium. However, the Ni coatings cannot form a diffusive bond with the surface of the balls due to the ZrO₂ ceramics with a high hardness, resulting in the Ni coatings and the milling balls are only mechanically bonded, the bonding was weak. Furthermore, the coatings got work-hardened and become brittle under the repeating sever plastic deformation during long time of ball milling operation. Subsequently, most parts of the metal coatings peeled off from the surface of the ceramic balls and its thickness decreases [11]. It can be seen that the thickness of the coatings has gone through two stages of thickening and thinning [12], which is consistent with other reports [11]. In the present experiment, when the rotation speed was 270 rpm, the exfoliation stage started earlier. When the rotation speed was 240 rpm, the

exfoliation started after 15 h of ball milling, and when the rotation speed was 210 rpm, it was still in the formation and thickening stage of coatings even after 20 h of ball milling.

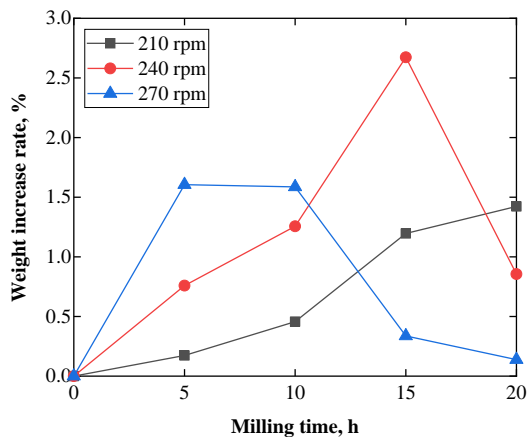


Fig. 2. Weight increase rate of milling balls versus duration of mechanical coating at different revolution speeds

Fig. 3 shows the surface morphology and microstructure of the cross sections of the ZrO₂ balls with the maximum weight increase after coating different milling time at different rotation speeds, that is, Fig. 3 a, d, Fig. 3 b, e and Fig. 3 c, f are the corresponding morphology and cross section views after 210 rpm ball milling for 20 h, 240 rpm ball milling for 15 h and 270 rpm ball milling for 5 h, respectively. The dark gray areas of the balls correspond to zirconia, while bright white regions were confirmed to be nickel. It can be seen from Fig. 3 a that the surface of the ZrO₂ balls is not completely covered with white Ni particles, but only some Ni metal particles are

dispersed on its surface, indicating that dense Ni coatings could not be obtained. The corresponding cross section in Fig. 3 d shows that a small amount of Ni particles is locally dispersed on the outside of the ZrO₂ balls, and no continuous coatings was formed. Compared with Fig. 3 a, the surface of the ZrO₂ balls in Fig. 3 b was covered by more Ni metal particles. Only a small amount of area was not covered, indicating that a dense coating was basically formed. Fig. 3 e shows that a substantially continuous Ni coatings with an average thickness of about 20 μm but uneven was formed on the outside of the ZrO₂ balls when the milling time came to 15 h at rotation speed of 240 rpm. The percentage of the surface of the balls covered by Ni particles in Fig. 3 c is between Fig. 3 a and Fig. 3 b, but it is clearly different from Fig. 3 a that the particles deposited on the surface of the ball in Fig. 3 a are discretely distributed, and Fig. 3 c has formed a local continuous area. Fig. 3 f shows that although no continuous coatings was formed, more metal particles were deposited, and they start to join locally. Therefore, the rotation speed of 240 rpm is a better choice during in the coating process of ZrO₂ balls having a diameter of 1 mm with the Ni metal particles, when the weight ratio of the balls is 3:5, a total of 80 g was placed into a 250 ml bowl. After 15 h of ball milling, a substantially continuous coatings can be obtained. Compared with other metal coating processes, the resulting Ni coatings peel off earlier and the final thickness is thin, which may be related to the nickel metal with a high electronegativity and poor plasticity [18].

3.2. Discussion of mechanism of rotation speed affecting coating

The fact that continuous coatings can not be obtained during the milling operation at 210 rpm and 270 rpm may be explained by the following phenomenon.

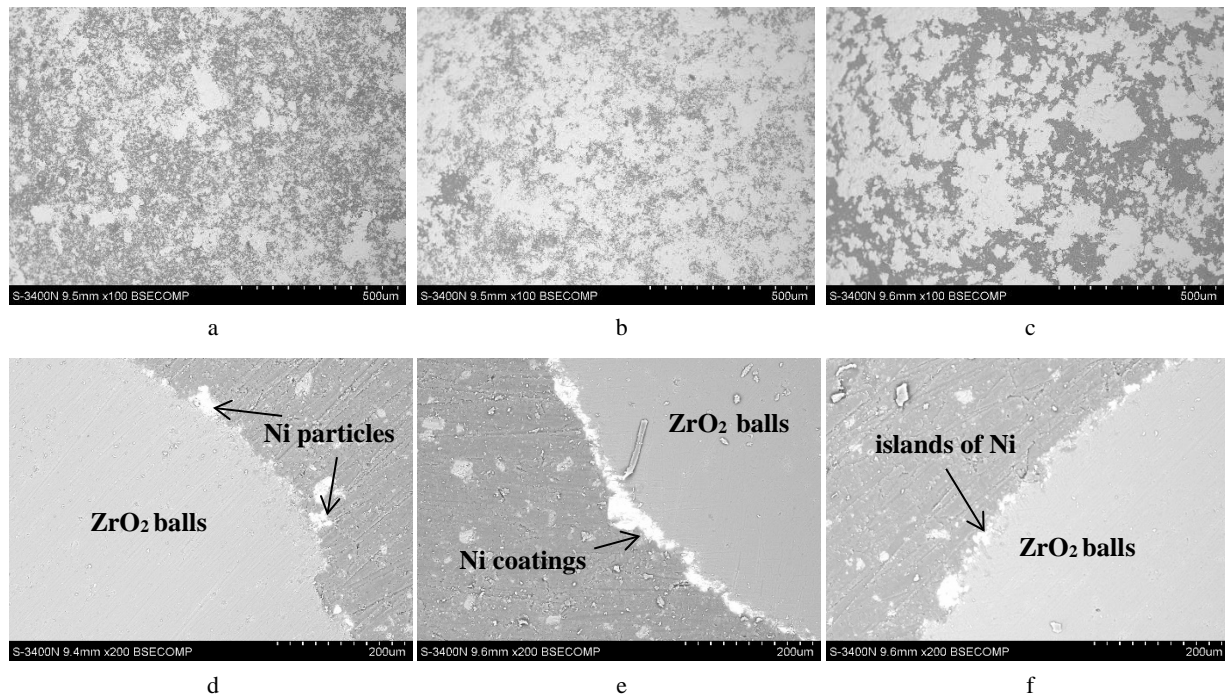


Fig. 3. Surface and cross section morphology of ZrO₂ balls after: a, d – speed of 210 rpm for 20 h; b, e – speed of 240 rpm for 15 h; c, f – speed of 270 rpm for 5 h

During the milling operation at 210 rpm, it was found that the inner wall of the bowl near the top position was clean, indicating that the milling balls and powder began to fall before they reaching the highest position of the top of the bowl. However, with increase in rotation speed up to 270 rpm, the inner wall of the bowl and the lid were all contaminated with the powder, but the bottom center of the bowl was as clean as before. In addition, there were obvious spiral dents on the inner surface of the bowl. This observation suggests that the position of the milling balls motion was higher under this condition at 270 rpm, and the milling balls were pressed on the inner surface of the bowl. The huge friction between the milling balls and the inner surface of the bowl caused the above spiral dents. The motion track of the milling balls at rotation speed of 210 rpm and 270 rpm was described separately in Fig. 4.

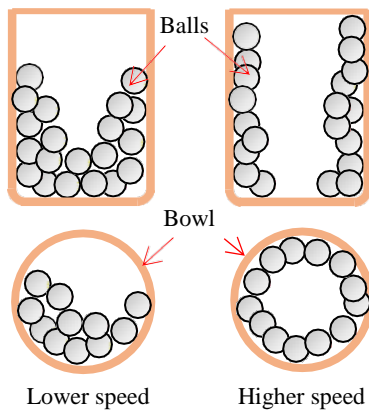


Fig. 4. Simulation of motion of milling balls in different speeds

We might conclude from the above phenomenon that normal force (direct impact force) rather than tangential force (friction force) was dominant during the milling operation at 210 rpm. The Ni metal particles were plastically deformed, cold welded, and deposited on the surface of the milling balls to form a coatings under the action of the impact force from the bowl and the milling balls. On the contrary, with increase in rotation speed up to 270 rpm, the motion speed of the milling balls also increases, and the required centripetal force will also increase. In this case, the milling balls will move closely to the inner wall of the milling bowl and losing the impact force, so the formed coatings peels off under the shear of the friction. Indicating that only friction was not conducive to the thickening of the coatings. In summary, the rotation

speed might affect the formation of the coatings by changing the stress condition among the metal particles, the milling balls, and the inner wall of bowl [11]. Lower speed mainly produces compressive stress, and higher speed mainly produces shear stress. Increasing the rotation speed within the appropriate range, the coexistence of the two kinds of stresses can significantly shorten the ball milling time and significantly improve the process efficiency, but the continues coatings can not be obtained at excessively high rotation speed.

3.3. Ni coatings oxidation sintering

The ZrO₂ balls coated with Ni coatings after 15 h ball milling at 240 rpm were oxidized treatment at 800 °C in air. The macro-morphology of the balls' surface is shown in Fig. 5. It can be seen from the figure that the ZrO₂ balls are no longer white after ball milling, indicating that the surfaces of the balls are almost completely covered with the metal Ni powder. And the surface of the ZrO₂ balls after sintering at 800 °C was light green, which matches the colors of NiO, indicating that Ni powder might have been oxidized to NiO. In addition, parts of the surface of the milling balls was lighter green and has a whitening phenomenon, indicating that the sintered coatings may be peeled off from the surface of balls, causing the white substrate being exposed.

Fig. 6 is the SEM images of the local microscopic morphology of the ZrO₂ balls milled at the optimal rotation speed (240 rpm) for 15 h and after sintering at 800 °C. As shown from Fig. 6 a, the particles on the surface of the ZrO₂ balls showed continuous agglomeration distribution before sintering. Using EDS to perform energy spectrum analysis on these aggregated particles, it was found that these particles were mainly Ni element. In addition, trace amounts of C, O and Al elements may be impurities from corundum bowl and air, and no Zr element was found in EDS results. It might indicate that the milling balls was completely covered by Ni coatings.

However, the particles on the surface of the ZrO₂ balls were no longer continuous after sintering was applied in Fig. 6 b, showing a discrete distribution. EDS results show that the particles on the surface of the balls are mainly O and Ni elements, indicating that these particles are the Ni coatings residue rather than the substrate. The analysis is consistent with the above inference that Ni coating has been peeled off after sintering and causes the substrate to be exposed (Fig. 5).

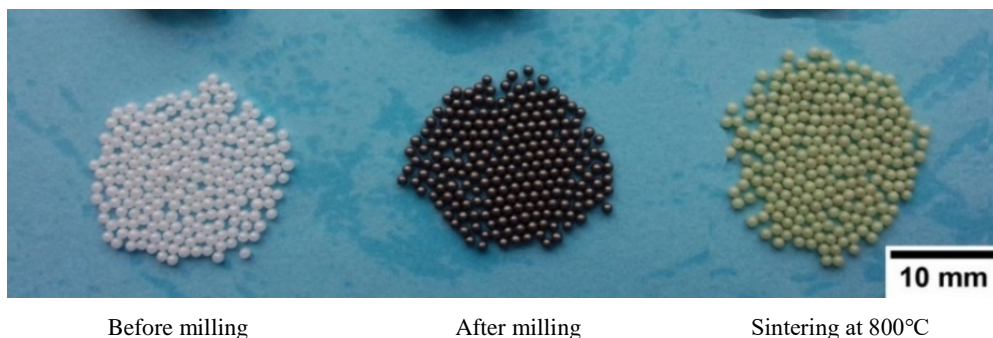


Fig. 5. Surface colors of ZrO₂ balls of: before milling, after ball milling for 15 h at 240 rpm, and after oxidation treatment

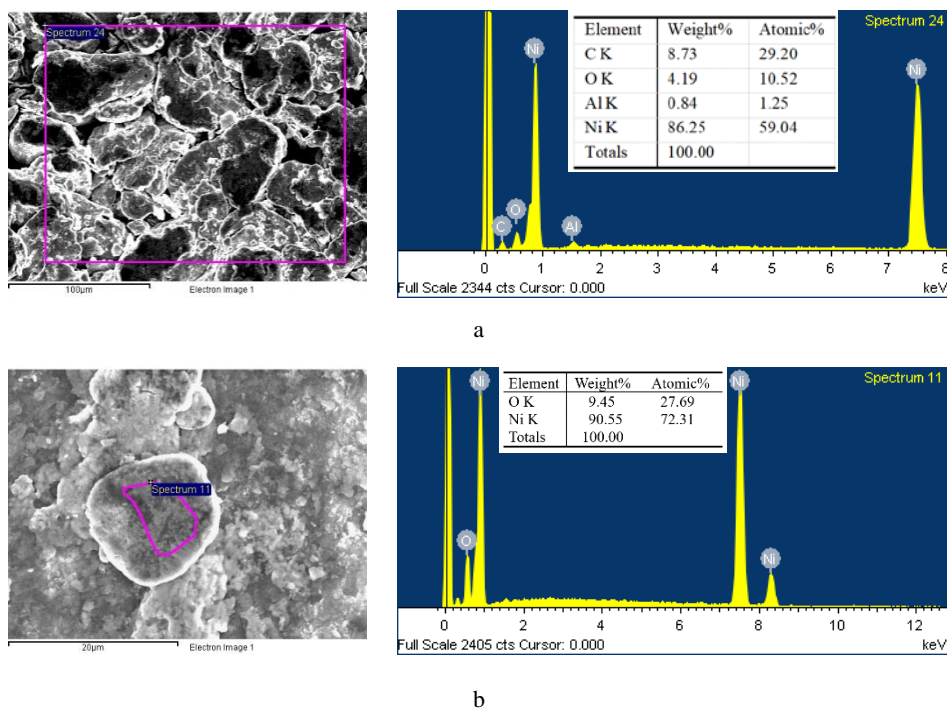


Fig. 6. SEM images and EDS of the surface of ZrO₂ balls: a – after ball milling; b – after sintering at 800 °C

The peeling off coatings from the surface of milling balls after heat treatment can be explained by the two reasons as following. On the one hand, the Ni metal particles will leave a large amount of strain energy after suffered severe deformation under the impact of the milling balls, and eventually adhere to the surface of the milling balls to form a coating and leave residual stress. Therefore, the coatings begin to crack and flake after heat treatment. On the other hand, because the thermal expansion coefficients of the metal Ni and ZrO₂ ceramics differ greatly, which causes large thermal stress during sintering and resulting in the coatings to be peeled off from the surface of the milling balls [19].

Fig. 7 shows the XRD patterns of the surface of the ZrO₂ balls coated with Ni before and after sintering at 800 °C. It can be seen that diffraction peaks of NiO became higher, while those of the Ni became lower after sintering. It suggests that Ni was oxidized to NiO after sintering at 800 °C which correspond to Fig. 5.

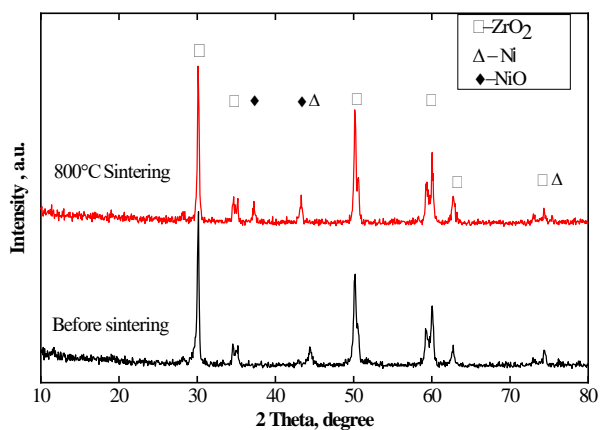


Fig. 7. XRD patterns of the Ni-coated ZrO₂ balls before and after sintering at 800 °C

In addition, it is obvious to find that there are ZrO₂ diffraction peaks before and after sintering, which may be that the coated Ni coatings is not dense enough before sintering, X-rays directly pass through the gap of the Ni coatings and diffract with ZrO₂. On the other hand, the coatings have a lot of exfoliation after sintering, causing the ZrO₂ substrate was bared outside. It is consistent with the phenomena described in Fig. 3 b and Fig. 6 b. However, in the coating process of other metals with better plasticity, the obtained coatings not only have a greater thickness but also have not peeled off significantly after sintering. From the above results, it can be confirmed that the metal Ni is not suitable for coating directly on the ZrO₂ ceramic balls because its bonding strength with the substrate is relatively poor, a composite coatings like TiO₂/Ni might be obtained by first preparing a TiO₂ coatings and then coating Ni coatings on its surface.

4. CONCLUSIONS

In the process of preparing Ni coatings on the surface of ZrO₂ ceramic balls by mechanical ball milling coating method, the formation of the coatings was a dynamic process of the cold welding between metal particles and the exfoliation of coatings were made. The evolution of the coatings' thickness can fall into thickening and thinning stages, higher rotation speed greatly accelerated the evolution of the coatings by increasing the collision strength of the balls. However, when the rotation speed was too high, the maximum thickness of the coatings was smaller, a critical minimum rotation speed was required to form continuous Ni coatings. Continuous Ni coatings with an average thickness of 20 µm was fabricated by mechanical coating technique during rotation at 240 rpm for 15 h, the coatings have a low dense and poor adhesion to the substrate. Therefore, metal Ni is not suitable for mechanical milling directly on ceramic balls to form an ideal coatings,

a composite coatings such as TiO₂/Ni, TiO₂/NiO may be obtained by first preparing a TiO₂ coatings and then coating Ni on its surface.

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