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Arch. Metall. Mater. **65** (2020), 2, 677-684

DOI: 10.24425/amm.2020.132806

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EFFECT OF DIE WALL LUBRICATION ON HIGH VELOCITY COMPACTION BEHAVIOR AND SINTERING PROPERTIES OF Fe-BASED PM ALLOY

Fe-based PM alloy powder of Fe-2.5Ni-0.5Mo-2Cu-0.4C was pressed by high velocity compaction combined with die wall lubrication, and the effect of die wall lubrication on high velocity compaction behavior and sintering properties of the Fe-based PM alloy were studied. The results indicate that the impact force, green density, sintered density of samples increase with the augment of the impact velocity and die wall lubrication. Compared with that without die wall lubrication, the green density and sintered density of the sample with die wall lubrication are about 0.07-0.12 g/cm^3 and 0.08~0.11 g/cm^3 higher at the same impact velocity, respectively, while the ejection force of the die wall lubricated sample is much smaller, and reduced about 26%~36%. The green compact with die wall lubrication has much fewer porosity than that without die wall lubrication, and more mechanical bonding and cold welding regions are observed. The sintered samples mainly consists of gray pearlite and white ferrite, and more pearlite is observed in the sintered sample with die wall lubrication.

Keywords: High velocity compaction; die wall lubrication; green density; impact force; cold welding

1. Introduction

Powder metallurgy (PM) is a technology for effective producing near-net-shape components from metal powders, and widely used in automobiles, machinery, chemicals, electronics and other fields because of their advantages such as fewer chips, economical, high precision, excellent property, and stable quality [1-3]. For Fe-based PM part, its hardness, tensile strength, fatigue strength, toughness, etc. increase with density, especially when the density is over 7.2 g/cm^3, the relevant mechanical properties will increase geometrically [4]. However, the Fe-based PM part cannot be fully densified due to the existence of inherent pores. In addition, higher green density results in less shrinkage and distortion during sintering, which means a better dimensional accuracy. Therefore, improving green density is the key to obtain excellent mechanical properties and perfect dimensional accuracy.

High velocity compaction (HVC) technology is considered as the next breakthrough in the PM industry for cost-effective, high-density parts, which realizes densification within 20 ms by intensive repeated stress waves created by a hydraulically operated hammer traveling at speeds of 2~30 m/s [5]. Compared with that of the conventional compaction, the green density of HVC compact is higher than 0.3 g/cm^3. Studies have shown that die wall lubrication can improve the green density and reduce the amount of lubricant added in conventional compaction and warm compaction [6-8]. HVC combined with die wall lubrication can further improve the density of PM components, however, there have been few reports about the effects of die wall lubrication on HVC behavior and sintering properties of Fe-based PM alloy. The Fe-based alloy powder (Fe-2.5Ni-0.5Mo-2Cu-0.4C) was used as raw material to prepare high density Fe-based PM material by HVC combined with die wall lubrication, and the effects of die wall lubrication on HVC behavior and sintering properties of Fe-based PM alloy were studied in this paper.

2. Experimental

The raw experimental materials powder of reduced Fe powder, Ni powder, Mo powder and electrolytic Cu powder were produced by Nangong Xindun Alloy Welding Material Spraying Co., Ltd, and the particle size of the powders is about 25 μm,

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2.6 μm, 2.6 μm and 25 μm respectively. The graphite powder with size less than 30 μm was produced by Shanghai colloid chemical plant. The stearic acid amide (Chamide S) from Hunan Changsha Hengchang Chemical Co., Ltd was used as lubricant, and polytetrafluoroethylene emulsion (D210C) from Daikin Industries was used as die wall lubricant in the experiments.

The amount of Fe, Cu, Ni, Mo, and graphite powder was weighed to prepare the tested Fe-2.5Ni-0.5Mo-2Cu-0.4C Fe-based alloy powder, 0.5% (mass percentage, the same below) zinc stearate was added to the Fe-based alloy powder. To study the effects of zinc stearate lubricant contents on the HVC green compact, 0, 0.05%, 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% zinc stearate was also added to the Fe-based alloy powder, respectively. The powder was mixed using the QM-3SP2 planetary ball mill for 30 min with the ball-to-powder weight ratio of 5:1 and the rotational speed of 300 rpm.

The Gravitational energy stored type HVC experimental device was adopted in the paper, and its schematic diagram is shown in Fig. 1. The tested device is composed with upper base, lower base, guide pin, impact hammer and HVC die. The height of the guide pin is 3 m, and the maximum stroke of the impact hammer is 2.7 m, and the mass of the impact hammer is 50 kg. The impact velocity of the hammer V can be calculated by the formula: $V = \sqrt{2gh}$, and the kinetic energy of the impact hammer E can be calculated by the formula: $E = mgh$, where m is the mass of impact hammer, g is the acceleration of gravity, h is the height at which the impact hammer falls. The impact velocity and impact energy can be achieved by adjusting the stroke length of the impact hammer (that is the height at which the impact hammer falls), the designed impact velocity and impact energy is listed in Table 1.

TABLE 1

The designed impact velocity and impact energy at different impact hammer heights

Height of hammer (m)	Impact velocity (m/s)	Impact energy (J)
0.82	4	401.8
1.27	5	622.3
1.84	6	901.6
2.50	7	1225

To investigate the effect of die wall lubrication on HVC behavior and sintering properties of Fe-based PM alloy, only half of HVC samples were prepared by brushing the polytetrafluoroethylene emulsion on the inner surface of the die wall before HVC process. 11.3 g of the mixed Fe-based alloy powder was put into the vertical cylindrical steel die with an inner diameter 20 mm for preparing HVC green compact. JHBM-H1 pressure sensor installed on the base of the bottom punch can accurately measure the peak pressure at the moment of pressing, and obtain the impact force in the HVC process. The ejection force is the pressure required for the green compact to be released from the mold, and it was measured by the DL-300 hydraulic stripper. The green compacts were sintered at 1150°C for 1 h in the atmosphere of 10 vol.% H_2 +90 vol.% Ar.

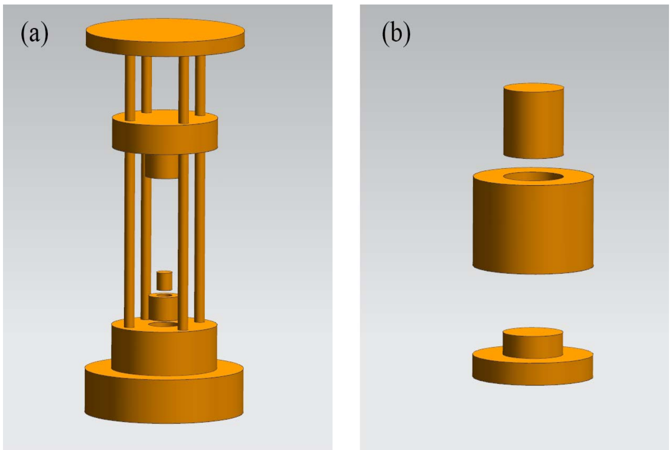

Fig. 1. The schematic diagram of the gravitational energy stored type HVC experimental device; a) Experimental device, (b) HVC die

The microstructure of HVC green compact and sintered samples was observed by optical microscope (Olympus BX41M-LED type), scanning electron microscopy (SEM, Hitachi S-4800). The sintered specimen were prepared by the standard metallographic methods, and etched by 3% nital and the etching times were carefully controlled. The density of the samples was measured according to GB/T 5163-2006 based on the Archimedes principle. An HR-150A type hardness tester was used in measuring the apparent hardness according to GB/T 9097-2016.

3. Results and discussion

3.1. HVC behavior

3.1.1. Effect of die wall lubrication on the impact force

The impact force increases with the increase of the impact velocity as shown in Fig. 2, and the impact force in the case of die wall lubrication is higher than that of without die wall lubrication

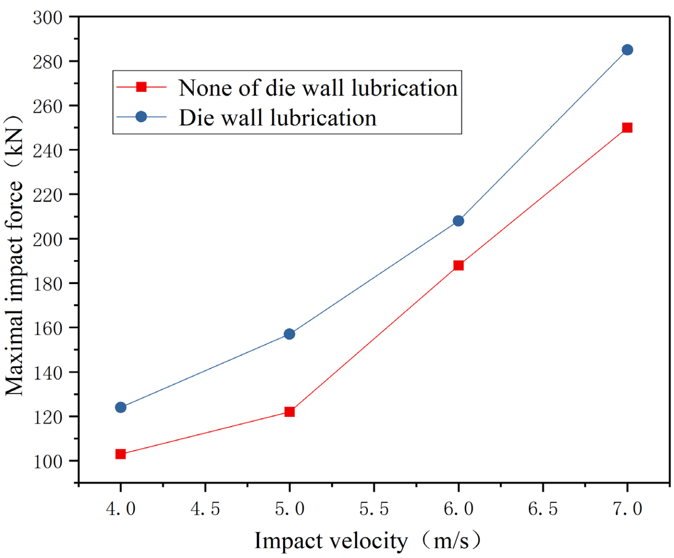

Fig. 2. The effect of die wall lubrication on the impact force

at the same impact velocity. Compared with that without die wall lubrication, the impact force of the samples with die wall lubrication increases about 20~35 kN under the same impact velocity. When impact velocity increases from 4 m/s to 7 m/s, the impact force increases from 103 kN to 250 kN for the samples without die wall lubrication, while increases from 124 kN to 285 kN for the samples with die wall lubrication.

Unlike the conventional compaction process, the transfer of impact energy during the HVC process is in the form of stress wave, which will dissipate rapidly after transmitting several cycles in the compact. The stress wave rises from zero to several tens MPa in tens of microseconds and drops to zero again as it transmits through the Fe-based alloy particles to the bottom punch and then reflects back to upper punch through the compact, and the first peak of stress wave is considered as the main impact force [5,9]. As the impact energy acting on powder body increases with the increase of the impact velocity, so the compacting force also increases. In the case of die wall lubrication, the die-wall frictions during powder pressing is greatly lessened, which is beneficial to the transmission of stress wave, therefore, the impact force in the case of die wall lubrication is higher than that without die wall lubrication at the same impact velocity.

During the HVC process, the impact hammer impacts the powder particles at a great momentum. According to the laws of conservation of energy and momentum, the impact force F is the momentum change rate with time or the kinetic energy change rate with distance. If it is assumed that the kinetic energy is changed uniformly within the whole powder particles moving distance S during HVC process, and the mass of upper punch (83.6 g) is omitted as it is too small compared with that of impact hammer, the impact force F can be calculated with the following equation:

$$F = \frac{d(mV)}{dt} = \frac{dE}{dS} = \frac{mV^2}{2S} \tag{1}$$

Where, V is the impact velocity of the hammer, m/s; m is the mass of impact hammer, kg; S is the whole powder particles moving distance during HVC process, m. The apparent density of the tested Fe-based alloy powder is about 3.05 g/cm^3, the thickness of HVC green compact with the die wall lubrication under the impact velocity of 5 m/s is 4.85 mm, the whole powder particles moving distance S is calculated to be 6.94 mm, the impact force F is 90.06 kN from the equation (1). It is noticed that the calculated impact force is smaller than the measured one of 124 kN, the reason is that the whole powder particles moving distance S is used to calculate the maximum impact force in the equation (1), the maximum impact force is the first peak of stress wave [9], and the actual powder particles moving distance where the maximum impact force reaches is much smaller than the whole powder particles moving distance S in the HVC process, which lead to the greater value of measured impact force. In fact, the above calculated value of the impact force can be accepted as the average impact force during the HVC process. If the measured impact force is used in the equation (1), then, the powder particles moving distance during the first peak of stress wave is calculated to be 5.04 mm, indicating 72.6% of powder particle compression is finished under the first peak of stress wave.

3.1.2. Effect of die wall lubrication on the ejection force

The effect of the impact velocity on the ejection force under conditions of with die wall lubrication and without die wall lubrication is shown in Fig. 3. The ejection force of the die wall lubricated sample is much smaller than that of the sample without die wall lubrication under the same impact velocity, and reduced about 26%~36%. The ejection force increases slightly with the impact velocity (less than 10 kN), especially in the case of die wall lubrication.

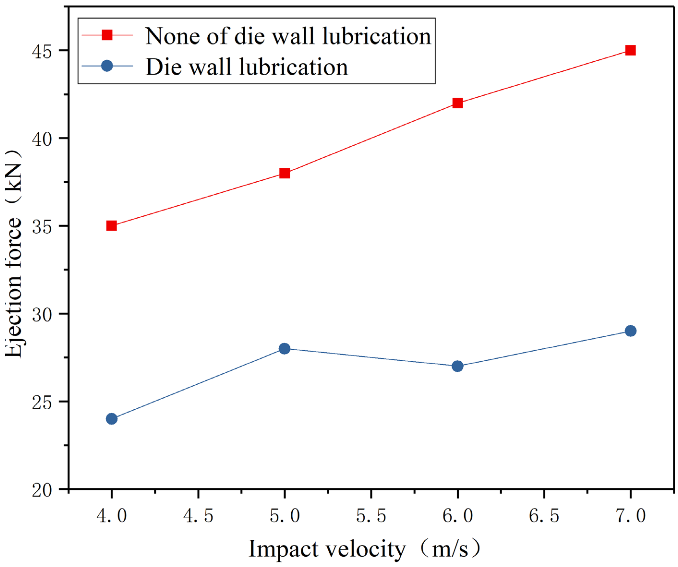

Fig. 3. The effect of die wall lubrication on the ejection force

When the compact is removed from the mold, if the compact has no dimensional change, the ejection force is equal to the loss of friction between the powder and the die wall [10]. Compared with the conventional PM forming methods, the compact formed by HVC has less elastic springback because the high impact force gives better compressibility and good inter-locking of particles [5,11], thus, it can be considered that the ejection force is equal to the maximal static friction force between the die wall and the compact [5]. For the HVC process without die wall lubrication, the compact is easy to adhere tightly to the die wall, the friction between the powder particles and the die wall is increased, which will need much bigger ejection force for the green compact to be draw out from the die, and the compact is easily damaged, obvious scratches can be observed on the surface of the compact. The ejection force of die wall lubricated sample is much smaller than that without die wall lubrication, because the good die wall lubricating effect significantly reduces the friction between the compact and the die wall.

3.2. The microstructure and property of HVC green compacts

3.2.1. Effect of die wall lubrication on the microstructure of HVC green compacts

Fig. 4 depicts the pore distribution of the un-etched HVC green compacts prepared at the impact velocity of 6 m/s, and SEM images of the green compacts prepared at the impact velocity of 4 m/s and 6 m/s are shown in Fig. 5. The HVC green compact with die wall lubrication has much fewer porosity than that without die wall lubrication. The interparticle mechanical bonding and cold welding can be observed in the HVC green compact as indicated by the arrow A and B in Fig. 5, and more mechanical bonding and cold welding region presented in the green sample with die wall lubrication in contrast to that without die wall lubrication, especially under higher impact velocity.

During the HVC process, the densification is achieved by intense shock waves created by the impact hammer, which transfers the impacting kinetic energy through the upper punch

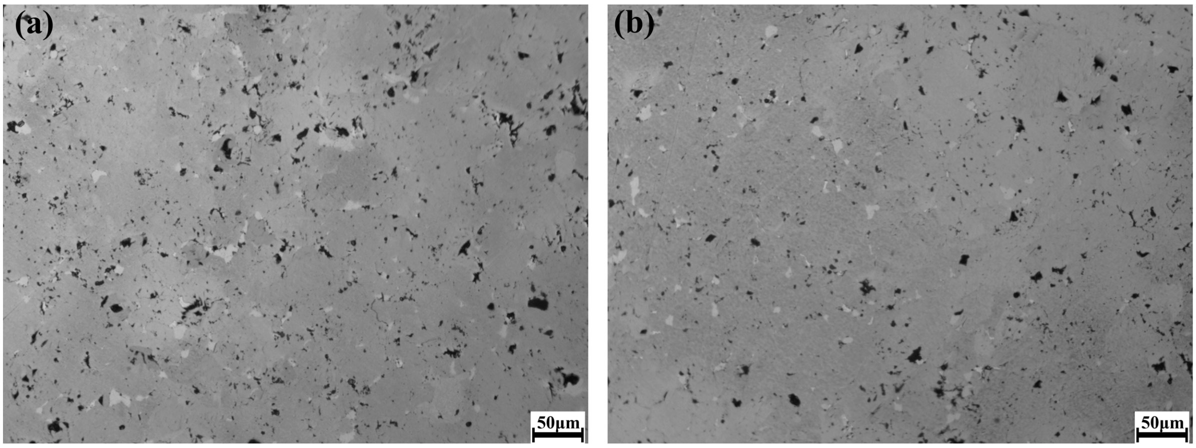

Fig. 4. Pore distribution of green compacts prepared at the impact velocity of 6 m/s; (a) without die wall lubrication (b) die wall lubrication

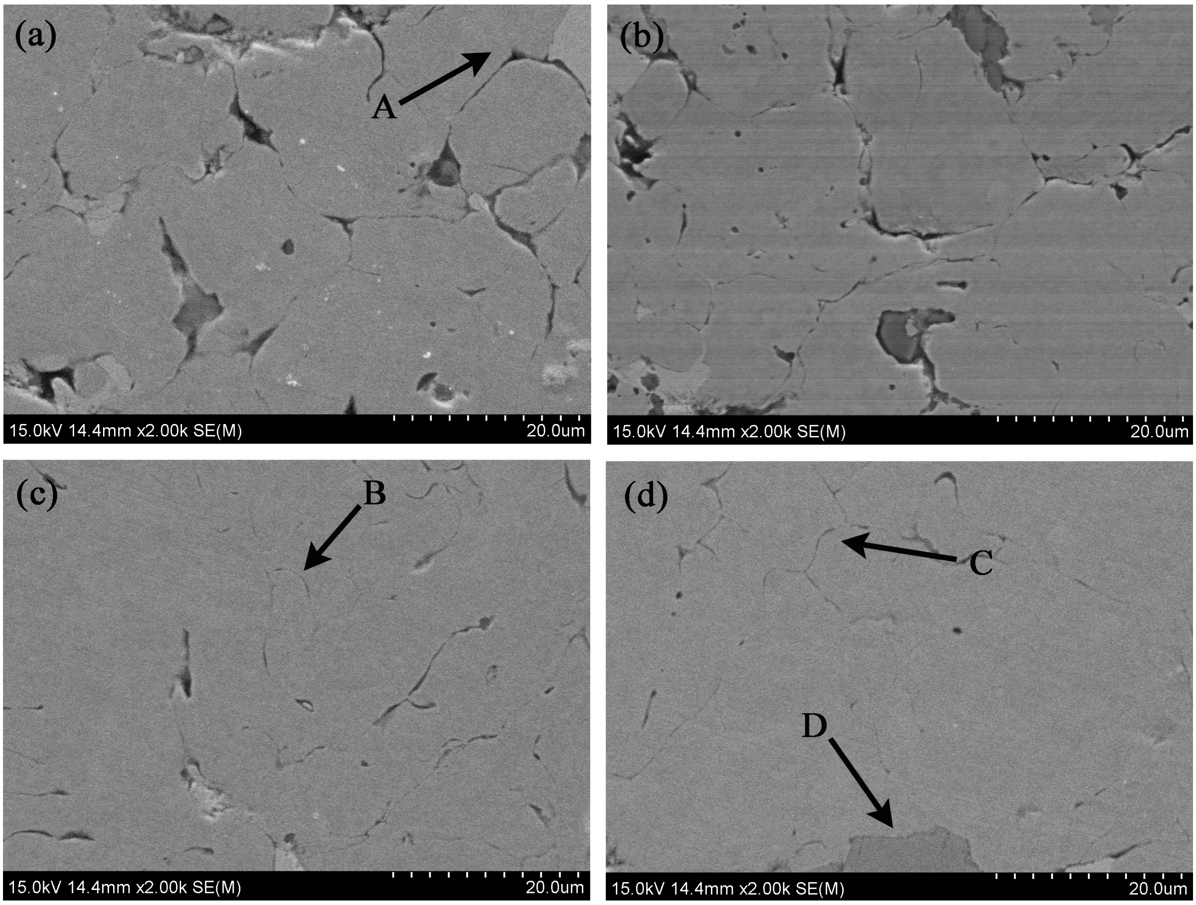

Fig. 5. SEM images of the HVC green compacts; (a) 4 m/s, without die wall lubrication, (b) 6 m/s, without die wall lubrication, (c) 4 m/s, die wall lubrication, (d) 6 m/s, die wall lubrication

to powder body. Particle rotation, displacement and other rearrangement occur instantly under the powerful impact force, and the adiabatic shearing and severe friction among powder particles produce massive heat, resulting in a dramatically rising transient temperature in local powder particle contact areas (the temperature at the particle contact can reach as high as 1000°C), then, the particles surface would be changed into softening or melting layer that enhances the mechanical bonding between particles [5,9,12-14]. The shear force produced by intense friction between particles will make the particle form a new clean surface, the contact between particles changes from point contact to surface contact, thus forming obvious cold welding [15]. In this case, the plastic deformation becomes the major factor for HVC densification of green compact [10]. As shown from the Fig. 2, the impact force increases obviously with the augment of impact velocity and die wall lubrication, so does energy acting on powder particles in unit time. On the one hand, this will lead to displacement of powder particles more easily, fine particles are easily filled into pores among coarse particles, leading to the reduction of porosity. On the other hand, the temperature rise effect of the particles surface due to the intense friction is increased, the deformation of powder particles will happen more easily, as a result, more mechanical interlocking between powder particles and cold welding regions are formed in the green compact.

3.2.2. Effect of lubricant content on the green density of HVC green compacts

Fig. 6 presents the effect of the lubricant (zinc stearate) content on HVC green density at the impact velocity of 6 m/s. The green density of the sample with die wall lubrication is much higher than that of the sample without die wall lubrication. Compared with that without die wall lubrication, when the zinc stearate content is 0.1%, the green density of the sample with die wall lubrication increases 0.26 g/cm^3, and reaches 7.31 g/cm^3. The green density is the lowest for samples without the lubricant addition and die wall lubrication, and it increases with the increasing of lubricant content in the case of without die wall lubrication, but slightly decreases after the addition higher than 0.1% in the case of die wall lubrication.

The addition of zinc stearate reduces the interparticle friction during HVC process, which is advantageous for the particle movement filling, rotation, displacement and other rearrangement, thereby increasing the compact density. Since the lubricant occupies a certain space in the compact, and its density is lower than that of metal powder, the extra lubricant addition will have a negative effect on green density [16]. Moreover, the lubricant generates gas during sintering, which may leave pores in the sintered material and reduce the sintered density, and the generated gas may damage the sintering equipment. Therefore, the amount of lubricant added needs to be effectively controlled, the rational amount of lubricant added in case of die wall lubrication should be no more than 0.1% under tested conditions of this work.

3.2.3. Effect of die wall lubrication on the green density of HVC green compacts

The effect of die wall lubrication on the green density of HVC green compacts is shown in Fig. 7. The green density increases with the increasing of impact velocity for the samples with and without die wall lubricated. The green density of the sample with die wall lubrication is about 0.07-0.12 g/cm^3 higher than that of the sample without die wall lubrication at the same impact velocity. At the impact velocity of 7 m/s, green densities of the samples with and without die wall lubrication is 7.35 g/cm^3 and 7.47 g/cm^3, respectively. The HVC densification is mainly determined by the shock wave's kinetic energy acting on the powder body. Because the kinetic energy absorbed by the powder particles increase with the augment impact velocity or with die wall lubrication, more interparticle mechanical bonding and cold welding region are formed, and the porosity

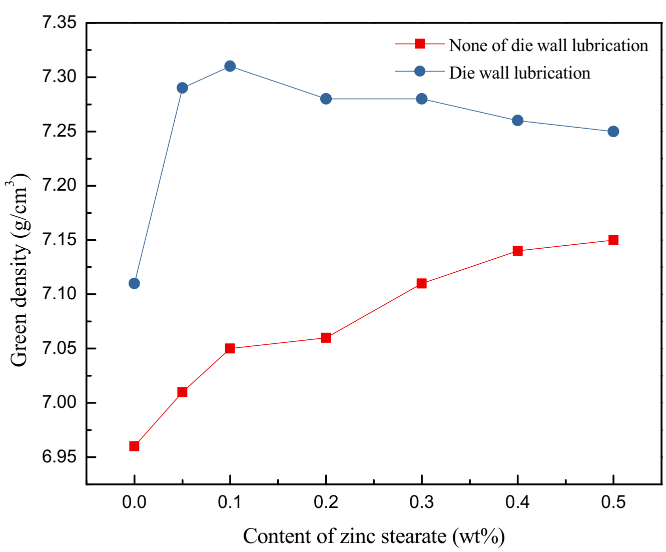

Fig. 6. Effect of the lubricant content on the green density

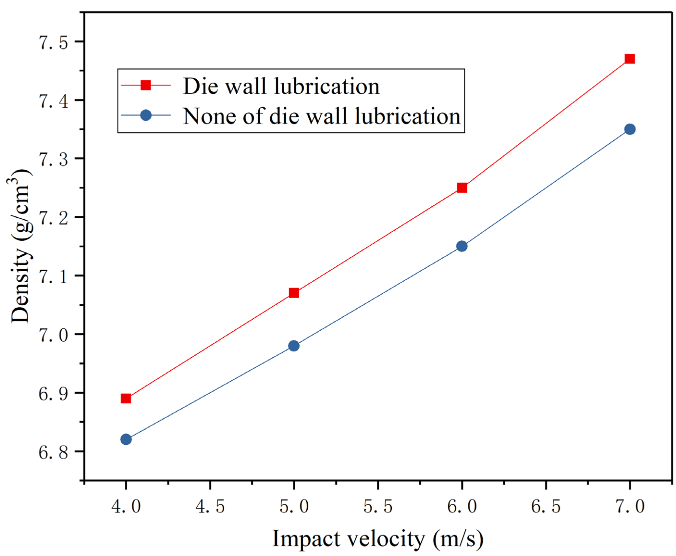

Fig. 7. Effect of die wall lubrication on the green density of HVC green compacts

of the HVC green compact is reduced (Fig. 4-5), leading to the increase of the green density.

3.3. The microstructure and property of HVC sintered samples

3.3.1. Effect of die wall lubrication on the microstructure of HVC sintered samples

Fig. 8 presents optical microstructure of the sintered samples at the impact velocity of 6 m/s. The sintered samples mainly consists of gray pearlite and white ferrite, and more pearlite is observed in the sintered HVC sample with die wall lubrication. Ni and Mo are two of the most commonly used alloying elements for Fe-based powder metallurgy materials, they can give full play to the enhancement effect only in case of diffusion uniformly as their very slow diffusion rate [17]. The cold welding formed between the powder particles in the HVC green compact is a metallurgical bond which can be considered to be a grain boundary or a phase boundary in microstructure [9,15,18], the diffusion activation energy of grain boundary is only half of the volume diffusion, and the diffusion coefficient is about 1000 times larger than that of the volume diffusion. Furthermore, great amount of lattice defects such as vacancies, dislocations presented in HVC process due to the particles deformation also facilitate rapid diffusion of alloying elements during sintering. Ni reduces the critical temperature of the austenite transformation, Mo and Cu are strong pearlite stabilizing elements [19]. As more mechanical interlocking between powder particles and cold welding regions are formed in the green compact with higher impact velocity and die wall lubrication as shown in Fig. 5, the distribution of Ni and Mo elements in the sintered sample will be more evenly distributed, thereby more pearlite will be formed in the sintered HVC sample with die wall lubrication.

3.3.2. Effect of die wall lubrication on the property of HVC sintered samples

As shown in Fig. 9, the effect of die wall lubrication on sintered density is the same as that of the green density of the compacts. The sintered density of the samples with die wall lubrication is 0.08~0.11 g/cm^3 higher than that of samples without die wall lubrication under the same impact velocity. For the sample prepared with higher impact velocity and die wall lubrication, the green compacts have higher green density and much fewer porosity, meanwhile, more metallurgical bonding and cold welding in the green compact can promote rapid diffusion of alloying elements in austenitic matrix during sintering, leading to the improvement of sintered density of the samples.

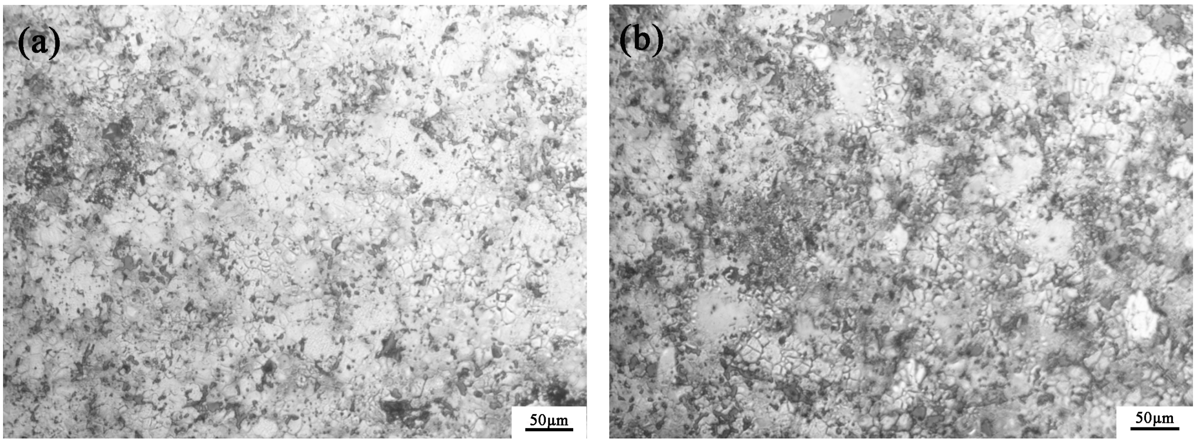

Fig. 8. Optical microstructure of the sintered samples; (a) without die wall lubrication, (b) die wall lubrication

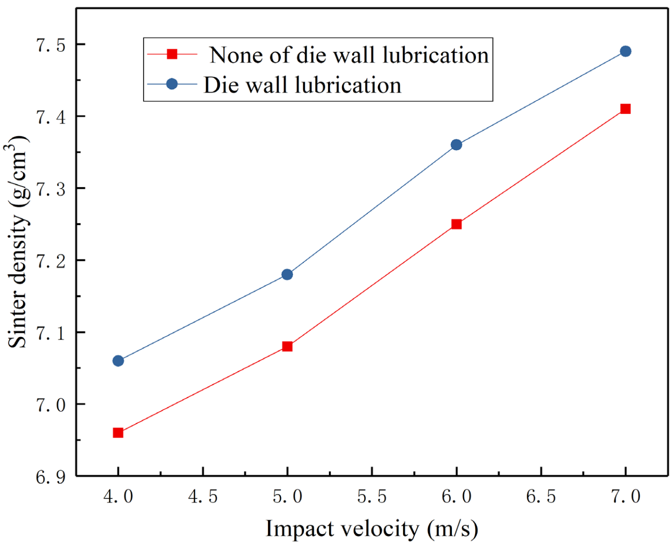

Fig. 9. Effect of die wall lubrication on the sintered density

Fig. 10 gives the hardness at the center and edge of the sintered samples at impact velocity of 6 m/s, and the effect of die wall lubrication on hardness at center of sintered samples is shown in Fig. 11. Compared with that without die wall lubrication, the hardness difference at the center and edge of the sintered samples prepared with die wall lubrication is relatively smaller. The hardness at the center of the sintered sample increases with

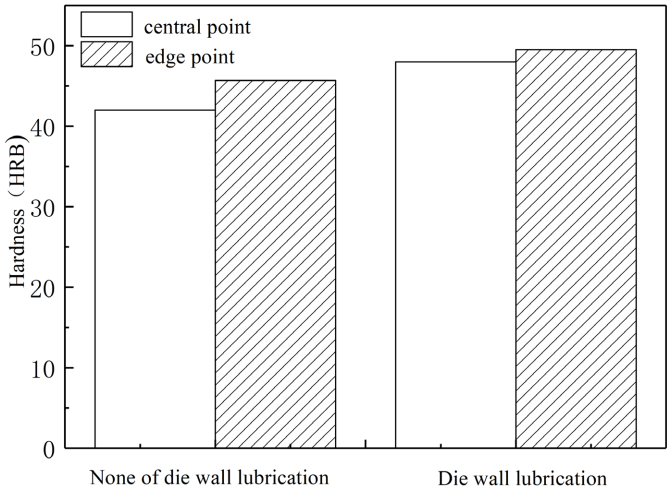

Fig. 10. The hardness at the center and edge of the sintered samples

the increase of the impact velocity, and it is higher for the samples with die wall lubrication at the same impact velocity. As the sample which prepared with higher impact velocity and die wall lubrication has higher sintered density and more pearlite in the microstructure, therefore has higher hardness. Owing to die wall lubrication, the powder particles can be better rearrangement and transfer the kinetic energy, the impact force on the powder compact will be more uniform, which is contributed to the homogenization of green density, resulting in the hardness difference at the center and edge of the sintered samples is relatively smaller.

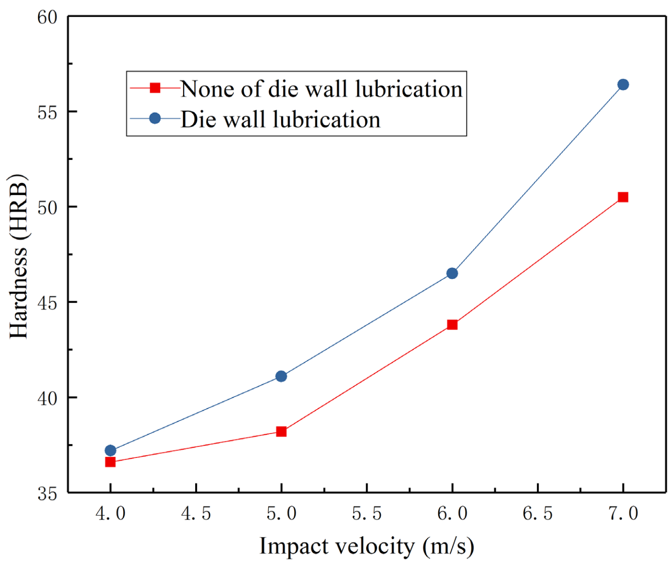

Fig. 11. Effect of die wall lubrication on the hardness at the center of sinter samples

4. Conclusions

The impact force increases with the increase of the impact velocity. Compared with that without die wall lubrication, the impact force of the samples with die wall lubrication increases about 20~35 kN under the same impact velocity. The ejection force of the die wall lubricated sample is much smaller than that of the sample without die wall lubrication under the same impact velocity, and reduced about 26%~36%.

The HVC green compact with die wall lubrication has much fewer porosity than that without die wall lubrication. More mechanical bonding and cold welding region presented the green sample with die wall lubrication in contrast to that without die wall lubrication, especially under higher impact velocity. The sintered samples mainly consists of pearlite and ferrite, and more pearlite is observed in the sintered HVC sample with die wall lubrication.

The green density, sintered density and hardness of the samples increase with the increasing of impact velocity. The green density and sintered density of the sample with die wall lubrication sample is about 0.07-0.12 g/cm^3 and 0.08~0.11 g/cm^3 higher than that of the sample without die wall lubrication at the same impact velocity, respectively. Compared with that without die wall lubrication, the hardness difference at the center and edge of the sintered samples is relatively smaller.

Acknowledgments

This study is financially supported by Enterprises Postdoctoral Program of 2018 Jiangsu Province's Double Creative Plan, China Postdoctoral Science Foundation funded project (Grant No. 2018M630555), Suzhou City Industrial Technology Innovation Project (Grant No. SGC201539), and A Project Funded by the Priority Academic Program Development of Jiangsu Higher Education Institutions.

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