Quantitative investigation of influence of Al₂O₃ non-metallic inclusions on bending strength of powder metallurgy high-speed steel

Qipeng Hu¹,²,³⁎, Yunbo Chen¹,², Miaohui Wang²,³, Xueyuan Ge³, Hengsan Liu³, Lingli Zuo² and Dongyue Zhang³

¹ China Academy of Machinery Science & Technology, Beijing, 100044, China
² Beijing National Innovation Institute of Lightweight Co. Ltd., Beijing, 100083, China
³ China Machinery Institute of Advanced Materials Co. Ltd., Zhengzhou City, Henan Province, 450001, China

⁎Corresponding author’s e-mail: telon_hu@163.com

Abstract. The types, dimensions and quantities of inclusions have been examined to quantitatively investigate their influence on the bending strength and impact toughness of HIPed high-speed steel. MgSiO₃ inclusion was found to be less harmful than Al₂O₃ inclusion in terms of mechanical properties. Additionally, the inclusion of Al₂O₃ of 0-20 μm under 0.02 vol.% showed a slight influence on the bending strength of certain HSS.

1. Introduction

High-speed steel (HSS) is widely used as a tool material due to its high wear resistance, desirable hardness, good toughness, and excellent stability of temperature resistance [1, 2]. Other applications have also been confirmed in wear parts and rolls [3, 4]. The mechanical properties can be influenced by many factors, such as bad heat treatment parameters, organic inclusion (fibre and rubber), and non-metallic inclusion. The appliance of powder metallurgy (PM) technology in manufacturing HSS has refined the size and distribution of carbides which also allowed to produce the higher carbon content high-speed steel.

The consensus that increasing volume fraction of inclusion diminishes the bending strength and impact toughness has been reached due to the fact that the damaged homogeneity of steel matrix results in stress concentration as induced by the sharp shape of inclusion. The volume fraction of inclusion increase is practically in direct proportion to the number of spots of cracks nucleation and spreading, especially to the deterioration of fatigue strength [5-7].

There has been a copious literature on the inclusion influence on the microstructure and fatigue strength. Nevertheless, the correlation between the specifics of inclusion and the resultant mechanical properties, such as impact toughness and bending strength [8] remains to be further explored, particularly, in a quantitative manner. The aim of this study is, from a quantitative perspective, to investigate the inclusion influence on the PMHSS.

2. Experimental procedures

PMHSS nitrogen-atomized spherical powder inclusion was applied, and the morphology of the powder was shown in Figure 1. The chemical and physical properties were shown in Table 1.
Table 1. Chemical content of PMHSS powder

| Element | C  | Mo | W  | Cr | V  | Fe   |
|---------|----|----|----|----|----|------|
| wt%     | 1.30 | 5.1 | 6.4 | 4.2 | 5.12 | Bal. |

Figure 1. The morphology of Al$_2$O$_3$ (a) spherical; (b) irregular.

Table 2. The mixture composition of PMHHS samples

| Type of inclusion | Morphology | Vol.% | Size, μm |
|------------------|------------|-------|----------|
| 1#  Al$_2$O$_3$  | Spherical  | 0.02  | 0~20     |
| 2#  MgSiO$_3$    | Spherical  | 0.025 | 0~20     |
| 3#  Al$_2$O$_3$  | Irregular  | 0.02  | 0~20     |
| 4#  Al$_2$O$_3$  | Spherical  | 0.002 | 0~20     |
| 5#  Al$_2$O$_3$  | Spherical  | 0.006 | 0~20     |
| 6#  Al$_2$O$_3$  | Spherical  | 0.012 | 0~20     |
| 7#  Al$_2$O$_3$  | Spherical  | 0.04  | 0~20     |
| 8#  Al$_2$O$_3$  | Spherical  | 0.02  | 20~80    |
| 9#  Al$_2$O$_3$  | Spherical  | 0.02  | 80~200   |
| 10# Al$_2$O$_3$ | Spherical  | 0.02  | 200~300  |

The HSS powder and additive powder mixture were obtained in a planetary ball mill (QM-3SP2, China), with a rotation speed 400 rpm, a 2h holding time, a ball to powder weight ratio of 3:1, and 5 mm stainless steel balls. The powder mixture composition is shown in Table 2.

The consolidation process by means of hot isostatic pressing (HIP) was employed to prepare the sample of mixture powders. The reason behind our option for HIP instead of other methods, such as vacuum sintering, solar sintering, and sparkle plasma sintering, is that none of those methods can be applied to a steady production of fine component grains with proper chemical homogeneity and high density. In the present study, the powder metallurgy HSS was essentially manufactured following the HIP process, where the mixture powder of 3 kg was put into steel canisters with a dimension of Φ70 × 80mm, under HIPed at 1150 °C and 110 MPa, for 4 hours of consolidation. Subsequently, heat treatment of austenization under a temperature of 1150°C and triple-temper temperature of 560 °C was applied to the as-HIPed canister.

For microstructure characterization, the samples were sectioned, ground, and polished following standard metallographic procedures. The optical micrographs (OM) and scanning electron microscope (SEM) micrographs were captured to analyse the microstructure and three-point bending fracture surface.
The hardness was tested on the Rock-well hardness tester (HRS-150D, China), where the load applied was 1471 N and the dwell time 10 s. To calculate the mean value, the hardness test was repeated five times. The impact toughness was tested by Pendulum Impact Testing Machine (500J, China), where the sample dimension was 10 mm × 10 mm × 55 mm, and the test was repeated a minimum of three times for each set. And the three-point bending strength was tested by testing machine (Zwick/Roell Z020, Germany) using a sample dimension of Φ5 mm × 40 mm.

3. Results and discussion

3.1. The influence of chemical composition and morphology on properties

To pinpoint the influence on the mechanical properties, three types of inclusion were studied at the same levels of addition volume. The morphology of the samples is shown in Figure 2. The OM micrograph shows that the inclusions were distributed evenly in PMHSS matrix, where fewer pores were observable. The impact toughness under the influence of the types of inclusion is shown in Table 3. Among all of the samples, the one involving MgSiO₃ shows the greatest impact toughness, while the sample having irregular Al₂O₃ inclusion exhibited a lower impact toughness than that with spherical Al₂O₃ inclusion.

| Sample                  | Impact toughness (J/cm²) |
|-------------------------|--------------------------|
| 1# Al₂O₃ irregular      | 13.9                     |
| 2# MgSiO₃               | 23.3                     |
| 3# Al₂O₃ spherical      | 17.0                     |

Figure 2. The morphology of different sample containing chemical content inclusion. (a) MgSiO₃; (b) Al₂O₃ spherical.

Figure 3. The relationships between bending strength and (a) inclusion type; (b) dimension of inclusion.
The relationship between the types of inclusion and bending strength under the same level of volume fraction is shown in Figure 3(a). Comparing the shape of Al₂O₃ inclusion revealed that the bending strength of spherical inclusion (2138 MPa) was 10.6% higher than that of irregular Al₂O₃ inclusion (1937 MPa). That finding clearly indicates the influence of inclusion shape on the mechanical properties, particularly on fracture toughness [9]. A growing body of studies on inclusion have shown that the shape has an important role to play in impact toughness as a result of the stress concentration surrounding the sharp shape of inclusion. In addition, the chemical composition of inclusion has also been found to influence the bending strength, which in the sample with MgSiO₃ inclusion (2761 MPa) was found to be greater than the one with spherical Al₂O₃ inclusion (2138 MPa) by 623 MPa. Thermodynamic studies on inclusion have suggested a reduced amount of Al₂O₃ inclusion of MgO refractory [9], affording the possibility of refining the Al₂O₃. In summary, the control of inclusion shape and chemical composition is critically important in protection against the degradation of mechanical properties in PMHSS.

3.2. The influence of inclusion morphology on properties

The results of the influence of particle dimension on bending strength and impact toughness are shown in Figure 3(b) and Table 4. As can be seen, with the increase of dimension of inclusion, the impact toughness showed a downward trend, where an evident drop was observed between 8# sample and 9# sample, suggesting that an inclusion of above 80 μm had a tremendous influence on the impact toughness of PMHSS. In comparison, the difference in the patterns of dimension influence on the bending strength was arguably attributable to the fact that the addition of inclusion was governed by the volume fraction, which means a dimension decrease of inclusion entails an increase of inclusion, where the influence of the inclusion dimension on the bending strength was expectedly discernable.

| Sample          | Impact toughness (J/cm²) |
|-----------------|--------------------------|
| 1# 0~20 μm      | 17.0                     |
| 8# 20~80 μm     | 17.0                     |
| 9# 80~200 μm    | 12.0                     |
| 10# 200~350 μm  | 13.0                     |

3.3. The influence of inclusion volume fraction on properties

The volume fraction increase of inclusion having a negative influence on the mechanical properties has been tested and generally confirmed among researchers. However, research results concerning bending strength are mostly qualitative, although a large body of research has been conducted toward a quantitative analysis of the correlation between fatigue limit and dimensions of inclusion.

In this study, the relationship between the volume fraction of Al₂O₃ inclusion and bending strength was quantitatively investigated, the results of which are shown in Figure 4, where a downward trend can be observed corresponding to an increase of inclusion, and a sharp decline was detected between 0.012 vol.% and 0.02vol.%, a finding suggesting that a threshold volume fraction below 0.012 vol.% is a prerequisite for lessening the damage of Al₂O₃ inclusion.
Figure 4. The relationship between volume fraction of Al\textsubscript{2}O\textsubscript{3} inclusion and bending strength, and the prediction equation curve.

To perform the quantitative investigation of the volume fraction of inclusion in relation to the bending strength of PMHSS, these variables need to be taken into consideration: inclusion-free bending strength, the volume fraction of inclusion, as well as the definitive parameters of the intrinsic properties of inclusion. All of these factors are closely connected with the bending strength, on the assumption that the Al\textsubscript{2}O\textsubscript{3} inclusion is spherical. The powder-law function \( y = a \times (1 + x)^b \) was employed to estimate the bending strength,

\[
R_{bb} = R_{PMHSS} \times (1 + \varphi_{Al\textsubscript{2}O\textsubscript{3}})^{-f}
\]

where \( R_{bb} \) is the estimated three-point bending strength, \( R_{PMHSS} \) (at 3300 MPa in this study) is the bending strength of PMHSS without inclusion, \( \varphi_{Al\textsubscript{2}O\textsubscript{3}} \) is the volume fraction of Al\textsubscript{2}O\textsubscript{3} inclusion, and \( f \) is the parameter resulting from the inclusion type. The bending strength data was applied to fit curve, where the goodness of fit was 0.92, with 95% of the prediction band covering the experimental data, suggesting that the equation can be regarded as a reliable predictor of the \( R_{bb} \).

4. Conclusion
The influence of inclusion on the mechanical properties and microstructure was investigated in this study, with a focus on the different chemical content, morphology, and volume fraction. The conclusions arrived at are as follows:

- Comparing the samples of different inclusion content revealed that MgSiO\textsubscript{3} inclusion was less harmful than Al\textsubscript{2}O\textsubscript{3} inclusion in terms of mechanical properties, where the influence of inclusion was intrinsically associated with the characteristics of inclusion because Al\textsubscript{2}O\textsubscript{3} inclusion was less deformable than MgSiO\textsubscript{3} inclusion during heat processes;
- As far as the morphology of inclusion is concerned, irregular Al\textsubscript{2}O\textsubscript{3} inclusion exhibited a more destructive influence than spherical Al\textsubscript{2}O\textsubscript{3} because of the stress concentration induced by sharp shape of inclusion;
- The consensus that increasing volume fraction of inclusion diminishes the bending strength and impact toughness was retested and consolidated in PMHSS. And the additional Al\textsubscript{2}O\textsubscript{3} inclusion of 0-20 \( \mu \)m under 0.02 vol.% exerted a slight influence on the bending strength of PMHSS;
- The three-point bending strength of Al\textsubscript{2}O\textsubscript{3} addition PMHSS can be estimated using the equation:

\[
R_{bb} = R_{PMHSS} \times (1 + \varphi_{Al\textsubscript{2}O\textsubscript{3}})^{-f}
\]

where \( R_{bb} \) is the estimated three-point bending strength, \( R_{PMHSS} \) is the bending strength of PMHSS without inclusion, \( \varphi_{Al\textsubscript{2}O\textsubscript{3}} \) is the volume fraction of Al\textsubscript{2}O\textsubscript{3} inclusion, and \( f \) is the parameters related to the inclusion type. Possible extension of this work is advised to associate the parameters \( f \) with the type, shape, diameter of the inclusion, and other variables.
References

[1] Meurling, F., Melander, A., Tidesten, M., & Westin, L. (2001) Influence of carbide and inclusion contents on the fatigue properties of high speed steels and tool steels. , 23(3), 215-224.

[2] Chen, N., Luo, R., Xiong, H., & Li, Z. (2020) Dense m2 high speed steel containing core-shell mc carbonitrides using high-energy ball milled m2/vn composite powders. Materials Science and Engineering, 771(Jan.13), 138628.1-138628.9.

[3] Godec, M., Bati, B. E., Mandrino, D., Nagode, A., LeskovEk, V., & Kapin, S. D., et al. (2010) Characterization of the carbides and the martensite phase in powder-metallurgy high-speed steel. Materials Characterization, 61(4), 452-458.

[4] Qu, H., Bo, L., Liu, L., Da, L., Jing, G., & Ren, X., et al. (2012) Precipitation rule of carbides in a new high speed steel for rollers. Calphad-computer Coupling of Phase Diagrams & Thermochemistry, 36(none), 144-150.

[5] Deng, G. Y., Tieu, A. K., Su, L. H., Zhu, H. T., Zhu, Q., & Zamri, W., et al. (2019) Characterizing deformation behaviour of an oxidized high speed steel: effects of nanoindentation depth, friction and oxide scale porosity. International Journal of Mechanical Sciences.

[6] Burnett, M. E., Glaws, P. C., & Gynther, D. K. (2017) The Effect of Nonmetallic Inclusions on Bending Fatigue Performance in High-Strength Steels.

[7] Murakami, Y., Kodama, S., & Konuma, S. (1989) Quantitative evaluation of effects of non-metallic inclusions on fatigue strength of high strength steels. i: basic fatigue mechanism and evaluation of correlation between the fatigue fracture stress and the size and location of non-metallic inclusions. International Journal of Fatigue, 11(5), 291-298.

[8] Lipiski, T., Wach, A., & Karpisz, D . (2020) Influence the non-metallic inclusion on bending fatigue strength of medium-carbon structural steel melted in an electric furnace. METAL 2020.

[9] Pan, F., Ding, P., & Zhou, S. (1997) Effects of silicon additions on the mechanical properties and microstructure of high speed steels. Acta Materialia, 45(11), 4703–4712.

[10] Cha, W. Y., Kim, D. S., Lee, Y. D., & Pak, J. J. (2007) A thermodynamic study on the inclusion formation in ferritic stainless steel melt. Isij International, 44(7), 1134-1139.