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# Dry-sliding wear of the 316L/h-BN composites produced under crack ammonia atmosphere

Ekkarat CHUSONG[1,*], Panya KANSUWAN[1], Naoto OHTAKE[2], Pongsak WILA[3], Nattaya TOSANGTHUM[3], Ruangdaj TONGSRI[3]

[1] *Department of Mechanical engineering, Faculty of Engineering, King Mongkut's Institute of Technology Ladkrabang (KMITL), Bangkok, 10520, Thailand*
[2] *Department of Mechanical engineering, Major in Engineering Sciences and Design, Tokyo Institute of Technology, Tokyo, 152-8550, Japan*
[3] *Particulate Materials Processing Technology (PMPT), Metal and Manufacturing Process Research Group, National Metal and Materials Technology Center, 114 Paholyothin Road, Khlong Nueng, Khlong Luang, Pathum Thani 12120 Thailand*

[*]**Corresponding author e-mail:** 60610029@kmitl.ac.th

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**Abstract**

Wear is one of different problems in mechanical failures of moving components. When a component encounters friction force on its surface, crack initiation tends to occur and wear follows crack propagation. Thus, the moving parts of automobiles should have proper wear resistance for long-time services, in addition to having high strength and hardness for heavy load operation. A self-lubricating material with compromised tribological and mechanical properties is important for some moving components. In this work, self-lubricating composites, metal matrix composites embedded with a solid lubricant, made from 316L stainless steel powder mixed with different hexagonal boron nitride (h-BN) contents of 10%, 15% and 20% by volume. The mixed powders were compacted into green parts (according with MPIF Standard 42) with density of 6.5 $g\cdot cm^{-3}$. Then, the green parts were sintered at 1100, 1150, 1200, 1250 and 1300°C under cracked ammonia (75% $H_2$+25% $N_2$) atmosphere for 60 min. The experimental results revealed that increases of hardness and strength sintered 316L matrix by reduction of pore amount and size were due to the increase of sintering temperature. However, the increase of h-BN content resulted in increase of pore amount and size. Additions of h-BN content up to 20 vol. % reduced friction coefficient of the sintered composites. At sintering temperatures of equal to and higher than 1200°C, h-BN did not react with 316L stainless steel powders to form intergranular boride phase. The sintered composites produced under the maximum experimental sintering temperature of 1300°C showed low specific wear rate.

## 1. Introduction

Wear is one of major problems in moving components. It causes damage by deformation of material at surfaces by contact or scratch with a counterpart material. Lubrication can improve wear resistance by decreasing friction coefficient, which in turn increases tool life and surface quality. Addition of a solid lubricant has benefits of anti-wear properties while it reduces maintenance operation [1]. Solid lubricants, such as molybdenum disulfide ($MoS_2$) [2] and graphite [3] are used in many applications as self-lubricating materials, which are generally produced by sintering. However, high sintering temperatures cause decomposition of such solid lubricants. Hexagonal boron nitride (h-BN) has crystal structure similar to that of graphite so it has lubricating properties [4]. It also has thermal and chemical stability. Due to the lubricating potential of h-BN, it was introduced to sintered stainless steel 316L matrix by 'press and sinter' process [5]. The specific wear rate was greatly reduced when the 316L/h-BN composites were sintered at 1200°C. The lowest friction coefficient and specific wear rate in the composites could be found under the experimental conditions used in that work when using 20 vol% of h-BN at a sintering temperature of 1200°C. The h-BN was also introduced together with $MoS_2$ to sintered stainless steel 316L matrix [6,7]. The results showed that the $MoS_2$ composite had higher wear resistance than the h-BN composite, but the h-BN composite yielded better friction reduction. Furthermore, the addition of h-BN/$MoS_2$ mixture could promote the hardness and wear resistance of the composites.

However, sintering at high temperatures results in interactions between stainless steel 316L matrix and those two solid lubricants with products of boride and sulfide [5-8]. The formation of boride or sulfide means loss of some original solid lubricant amount, which in turn causes reduction of lubricating ability. The compound formation is also anticipated to modify rubbing surface conditions of a solid lubricant-embedded stainless steel 316L composite.

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Recently, it has been found that h-BN became inert under nitrogen-containing sintering atmosphere [9-10]. Decomposition of h-BN in such atmosphere was low. Therefore, it is hypothesized in this work that the interactions between stainless steel 316L matrix and h-BN during producing a self-lubricating composite material by sintering would be minimized and the lubricating performance of the composite is highly maintained. The sintering atmosphere employed in this work is crack ammonia (75% hydrogen and 25% nitrogen). Hydrogen is still required as reducing atmosphere for sintering a stainless steel.

## 2. Experiment

### 2.1 Specimen processing

Powder mixtures made of 316L powder mixed with h-BN powder (0, 10, 15 and 20 vol%) were compacted into green parts (conforming the MPIF Standard 42) with density of 6.5 $g\cdot cm^{-3}$. Green specimens were sintered at 1100, 1150, 1200, 1250 and 1300°C under the crack ammonia atmosphere for 60 min. The sintered specimens were cooled with the rate of 0.1$°C\cdot s^{-1}$ in a sintering furnace.

### 2.2 Characterization and test of mechanical properties

The microstructures of the specimens were observed by optical microscopy (OM) and scanning electron microscopy (SEM) equipped with energy-dispersive spectroscopy (EDS) Micro-hardness of the sintered specimens was measured by a Vickers hardness tester (HV).

### 2.3 Wear test

Dry sliding wear test was performed by using ball-on-disc tribometer according to ASTM G99-05(2010). Disk specimens were employed for dry-sliding testing under a ball made of SKF chromium steel grade G20 with diameter of 7 mm as a counterpart material. The sliding conditions included speed of 0.1 $m\cdot s^{-1}$, load of 15 N and distance of 50 m at room temperature.

## 3. Results and discussion

### 3.1 Density and hardness

Both green and sintered densities decreased with increasing h-BN content. For each composite composition, sintered densities were higher than green densities (Figure 1). Sintered density also increased with increasing sintering temperatures. The higher sintered density indicates pore reduction and shrinkage that are related to pore filling and sintered neck growth, respectively. Both pore filling and sintered neck growth are due to microscopic mass transport during sintering process.

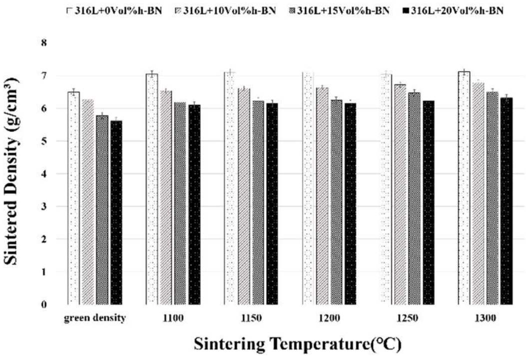

**Figure 1.** Effect of h-BN content and sintering temperature on density of the sintered 316L/h-BN composites.

The hardness values of sintered composites (Figure 2) showed similar trend to that of sintered density (Figure 1). The sintered composite with higher sintered density tend to have higher hardness. Since the hardness of a sintered material is directly proportional to sintered density [11] it can be one of sintering indicators.

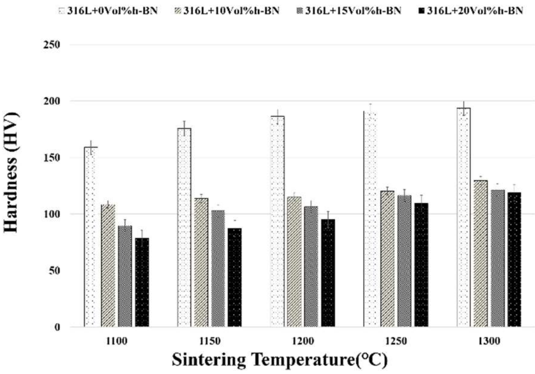

**Figure 2.** Effect of h-BN content and sintering temperature on hardness of the sintered 316L/h-BN composites.

### 3.2 Microstructure

The OM images of polished surfaces of sintered specimens (Figure 3) show pore characters (shape, size and distribution). Porosity increased with added h-BN content and decreased with increasing sintering temperature. The pore characters conform well to sintered density (Figure 1) and hardness (Figure 2). The pore reduction with increasing sintering temperature (Figure 4) suggests sintered neck growth or better bonding between powder particles. However, when the additive h-BN volume exceeds inter-particle pore size (Figure 5), pore reduction is not obtained.

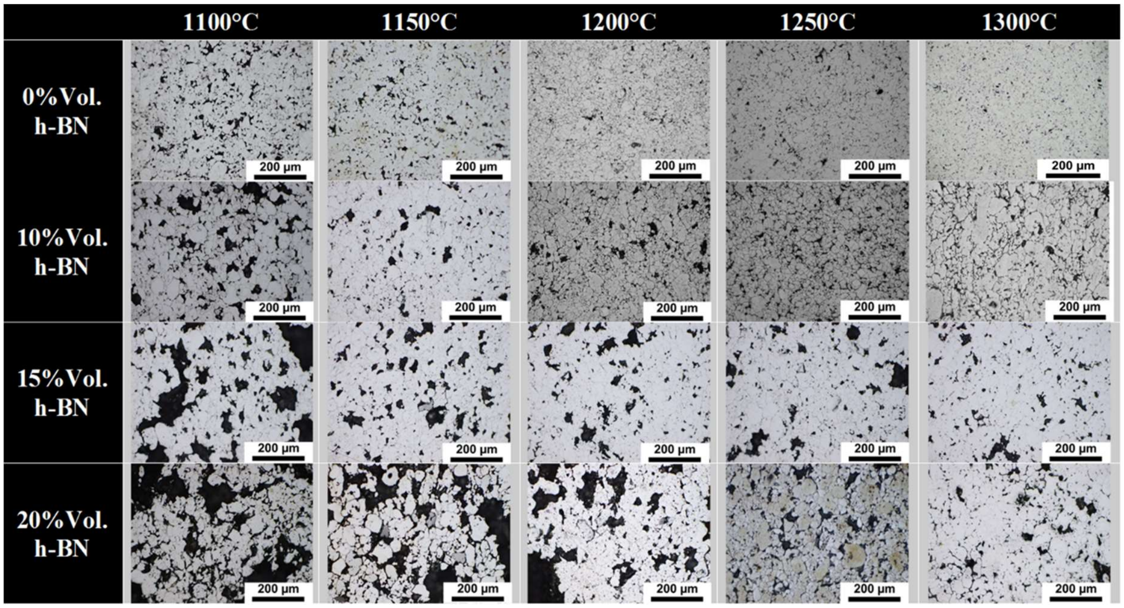

**Figure 3.** Optical micrographs of stainless steel 316L matrix/h-BN composites with h-BN contents of 0, 10, 15 and 20%vol, sintered at different temperatures.

Regarding the interactions between stainless steel 316L matrix and h-BN, it was found that intergranular boride phase was hardly observed (Figures 6 and 7). During sintering, decomposition of h-BN results in free boron atoms that can diffuse into Fe-based matrix. However due to extremely low solubility, boron tends to segregate to grain boundaries, where it reacts with iron and other elements to form boride [12]. Intergranular boride resulted from eutectic reaction between iron-based matrix and boron from h-BN clearly existed in vacuum-sintered materials even low H-BN amount (0.5 wt%) was added [13]. In the stainless steel 316L matrix/h-BN (or h-BN+$MoS_2$) composites sintered under pure hydrogen, evidences of boride were also prominent [5-7]. The absence of intergranular boride phase in the sintered composites (this work) and in other materials sintered under nitrogen-containing atmosphere [9, 10] suggests that nitrogen has strong influence on boride formation. The h-BN decomposition and the reaction to form boride are depicted by the equation given below.

$$2\text{h-BN} \rightarrow 2B + N_2 \quad (1)$$

$$2Fe + B \rightarrow Fe_2B \quad (2)$$

In case of vacuum and hydrogen sintering, nitrogen gas emitted from h-BN decomposition is removed by vacuum and hydrogen. When significant nitrogen concentration presenting in a sintering atmosphere the reverse reaction given in equation (1) would prohibit h-BN decomposition. To validate this hypothesis, chemical analysis was taken on a remaining phase in a pore.

### 3.3 Chemical analysis.

SEM and EDS mapping are shown in Figs. 8-11. The elemental iron was observed only in the stainless steel matrix

whereas elemental boron clearly existed in pores. The chemical analysis of intergranular boride phase, solidified from eutectic liquid formed during heating and sintering, showed co-existence of elemental iron and boron [14]. The chemical analysis results suggest that iron boride is hardly formed in the sintered composites produced under nitrogen containing atmosphere. This conforms to the experimental result conducted in [15], in which sintering in nitrogen containing atmospheres results in rapid deactivation of boron.

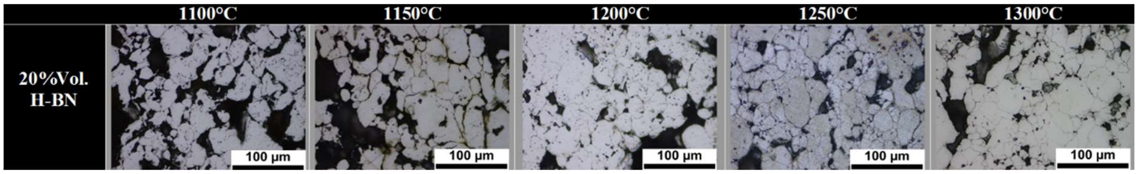

**Figure 4.** Stainless steel 316L matrix/h-BN composite with h-BN contents of 20 vol%, sintered at different temperatures.

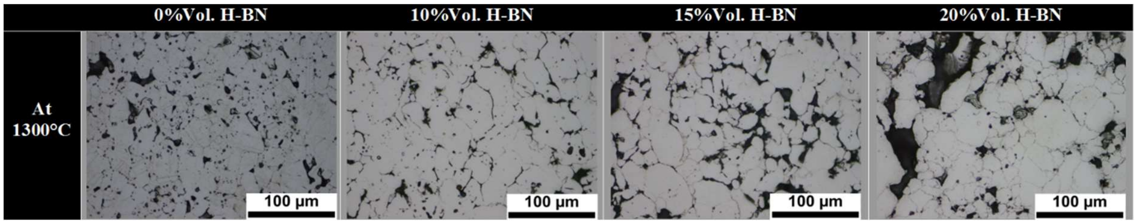

**Figure 5.** Stainless steel 316L matrix/h-BN composites with h-BN contents of 0, 10, 15 and 20 vol%, sintered at 1300°C.

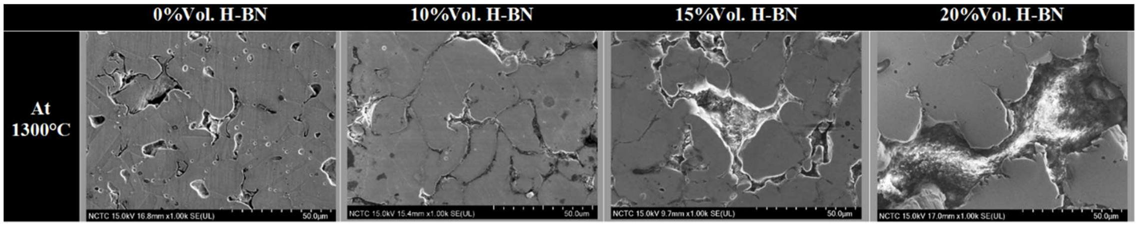

**Figure 6.** Secondary electron (SE) images of stainless steel 316L matrix/h-BN composites with h-BN contents of 0, 10, 15 and 20 vol%, sintered at 1300°C.

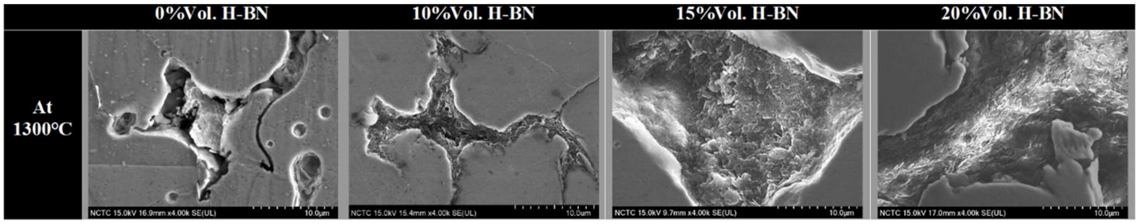

**Figure 7.** Secondary electron (SE) images of stainless steel 316L matrix/h-BN composites with h-BN contents of 0, 10, 15 and 20 vol%, sintered at 1300°C (high magnification).

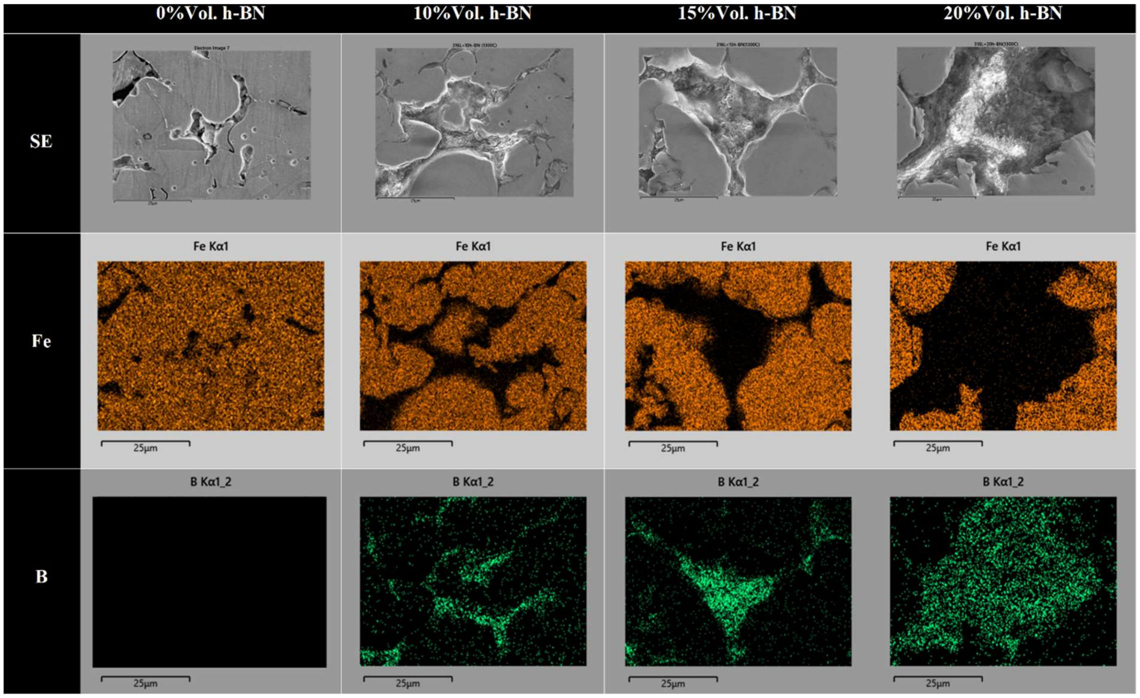

**Figure 8.** EDS mapping analysis of stainless steel 316L matrix/h-BN composites with h-BN contents of 0, 10, 15 and 20%vol, sintered at 1300°C.

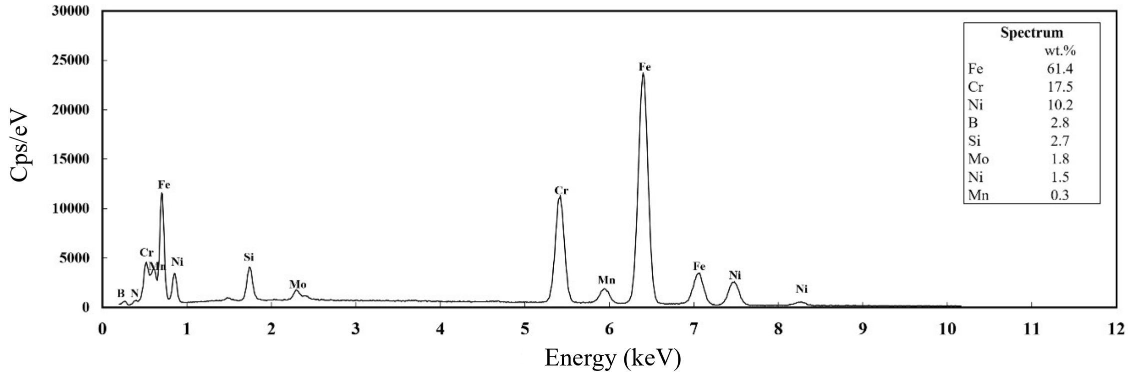

**Figure 9.** EDS Spectrum of stainless steel 316L matrix/h-BN composite with h-BN content of 0 vol% sintered at 1300°C.

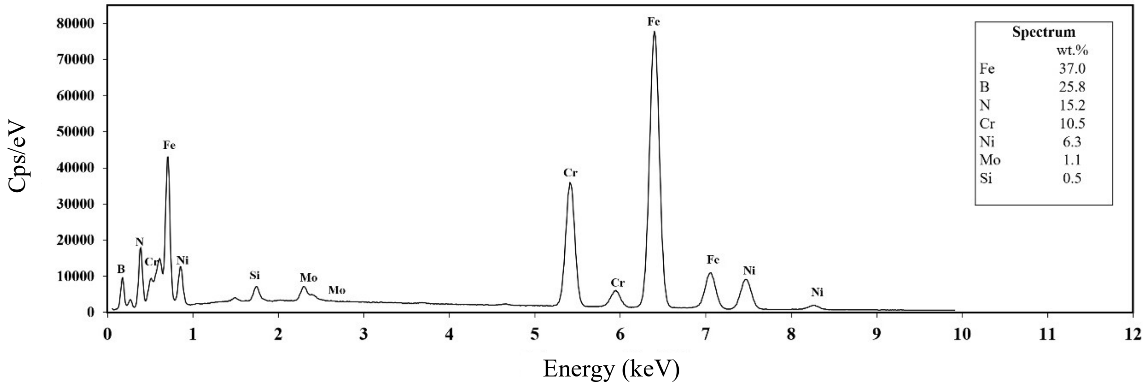

**Figure 10.** EDS Spectrum of stainless steel 316L matrix/h-BN composite with h-BN content of 20 vol% sintered at 1300°C.

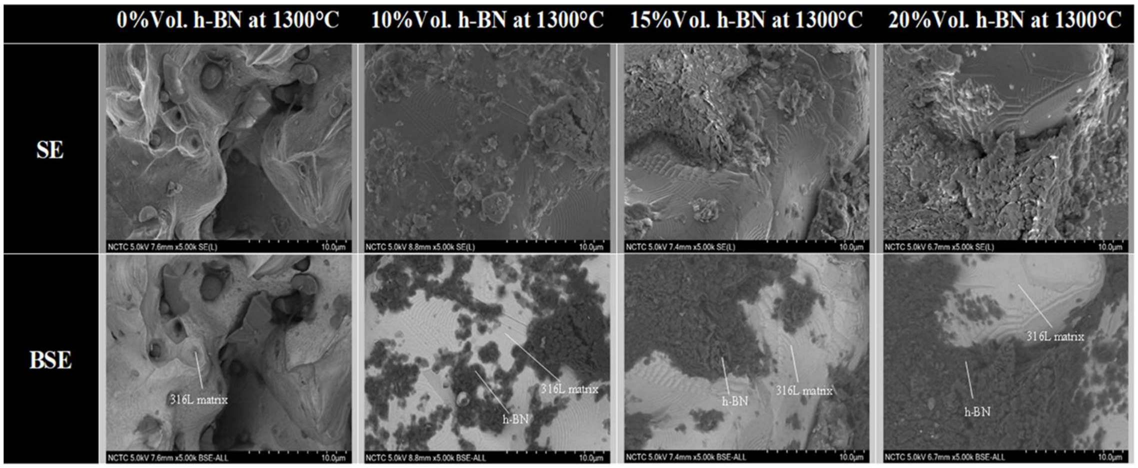

**Figure 11.** Back-scattered electron (BSE) and SE SEM images on the fracture surfaces of stainless steel 316L matrix/h-BN composites with h-BN contents of 0, 10, 15 and 2%vol, sintered at 1300°C.

## 3.4 Dry sliding wear test

Specific wear rates of the experimental sintered composites are shown in Figure 12. The composites with high h-BN content and sintered at low temperatures (1100 and 1150°C) showed high specific wear rate. By increasing sintering temperatures (≥1200°C) low specific wear rates were obtained. According to microstructure and chemical analysis presented above, the sintered composites produced at high temperatures and with high h-BN content have good particle bonding and non-decomposed h-BN still exists in pores. Due to good bonding and high amount of h-BN, the sintered composites have low specific wear rates.

Figure 13 shows non-systematic interrelationship between h-BN content, sintering temperature and coefficient of friction. This may be due to complex rubbing surfaces of the sintered composites. The surface has at least three different components, such as matrix, pore and solid lubricant. All components can give contribution to coefficient of friction.

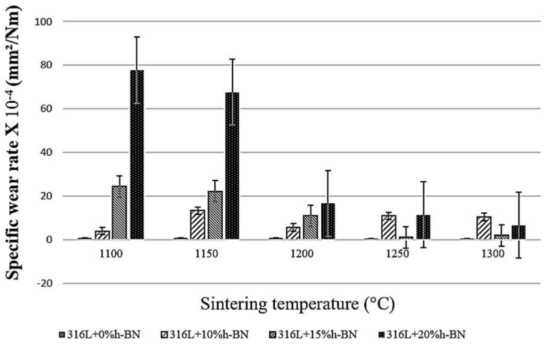

**Figure 12.** Effects of h-BN content and sintering temperature on specific wear rate of the sintered 316L/h-BN composites.

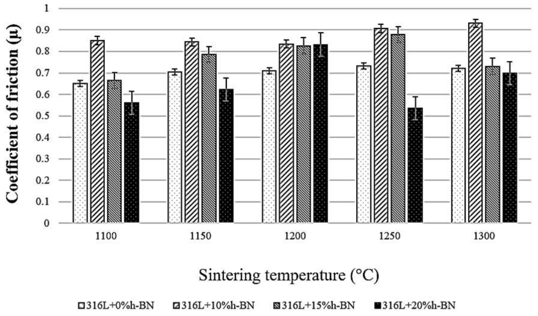

**Figure 13.** Effects of h-BN content and sintering temperature on friction coefficient of the sintered 316L/h-BN composites.

## 4. Conclusions

The experimental results revealed that increases of hardness and strength sintered 316L matrix by reduction of pore amount and size were due to the increase of sintering temperature. However. Too high h-BN content resulted in increase of pore amount and size. Additions of h-BN content up to 20 vol% reduced friction coefficient of the sintered composites. At sintering temperatures of equal to and higher than 1200°C, h-BN did not react with 316L stainless steel powders to form intergranular boride phase. The sintered composites produced under the maximum experimental sintering temperature of 1300°C showed low specific wear rate.

## 5. Acknowledgements

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