O U R N A L O F

Ceramic Processing Research

# Molten state processing of alumina fiber/high-speed steel composite

#### Nobuyuki Kobayashi, Soichiro Sameshima and Yoshihiro Hirata\*

Department of Applied Chemistry and Chemical Engineering, Kagoshima University, 1-21-40 Korimoto, Kagoshima 890-0065, Japan

High-speed steel (HSS) consists of Fe and several kinds of transition metal carbides. This steel is applied to cutting tools and as wear-resistant materials. When used as a cutting tool, HSS experiences relatively high thermal shock because a coolant such as water or oil is flowed over the surface of the heated HSS. The purpose of this research is to increase the hardness, strength, fracture resistance and thermal shock resistance of HSS. A possible strategy is to incorporate a hard ceramic material of high strength into an HSS matrix. This paper describes the molten state processing of such a composite with oriented unidirectional alumina fibers (10  $\mu$ m diameter) - HSS system. The HSS powder layer (5 mm or 1 cm) and the fiber layer (195  $\mu$ m) were alternatively inserted at 2 or 20 vol% fibers in a carbon mold and heated at 1630°C for 5 minutes in an Ar atmosphere to melt the HSS. During the heating, phase separation of the composite occurred to form an upper layer of fibers and a lower layer of the molten HSS. This phenomenon resulted from the density difference and low wettability between the two components. The solidified HSS of 90.5% theoretical density was made of pearlite (a mixture of Fe<sub>3</sub>C and  $\alpha$ -Fe) and transition metal carbides, and provided a high Vickers hardness of 9.14 GPa. On the other hand, our previous study revealed that the hot-pressing of laminated green composites under a pressure of 39 MPa at low temperatures of 900°-1100°C produced homogeneous dense structures without phase separation. The combination of the results derived in the above two processings leads to the conclusion that the key factors to produce dense laminated composites are the control of the heating temperature and applied pressure.

Key words: High-speed steel, Alumina fiber, Pearlite, Transition metal carbide, Vickers hardness, Wettability.

#### Introduction

High-speed steel (HSS) consisting of Fe and several kinds of transition metal carbides has a high fracture toughness (25~28 MPa·m<sup>0.5</sup>) and high hardness (7.5 GPa, Vickers hardness) [1, 2]. This material is applied to cutting tools or as wear-resistant materials. In the case of cutting tools, the use of HSS experiences relatively high thermal shock because a coolant such as water or oil is flowed over the surface of the heated HSS. To overcome the high thermal shock, a high strength is needed for the HSS. The purpose of this research is to increase the hardness, strength, fracture toughness and thermal shock resistance of HSS. A possible strategy is to incorporate a hard ceramic material with a high strength into HSS matrix [3-5]. The strength of the composite depends on the volume fraction and strength of the incorporated ceramic material. The fracture toughness ( $K_{IC} = (2 \text{ E}\gamma)^{0.5}$ ) of HSS increases with the addition of ceramic material with a high Young's modulus (E) and by the introduction of fracture energy adsorption mechanisms providing an increase of fracture energy  $(\gamma)$ .

The critical temperature difference ( $\Delta Tc$ ) to achieve no crack formation in brittle materials is expressed by  $\Delta Tc = \sigma (1-v)/\alpha E$ , where  $\sigma$  is the tensile strength, v the Poisson ratio,  $\alpha$  the thermal expansion coefficient, and E the Young's modulus. It is understood that a high  $\sigma$  and a low E are the effective factors to increase the thermal shock resistance. A higher Young's modulus and a lower thermal expansion coefficient of a ceramic material compared with the properties of HSS, provide the opposite influence on the thermal shock resistance of the composite. When the product of  $\alpha E$  becomes lower in the ceramic/HSS composite, the thermal shock resistance of the HSS may be improved with a similar strength. Based on the above ideas, the incorporation of long alumina fibers into an HSS was planned. This fiber has a high tensile strength of 1.8 GPa in addition to high hardness [6, 7]. This paper describes the processing of a composite by addition of long alumina fibers into a molten HSS. In our previous study [4, 5], long alumina fibers/HSS laminated composites were prepared through the hot-pressing at 900°-1100°C. This processing succeeded in achieving a dense composite with a high degree of dispersion of long fibers in an HSS matrix [5]. In addition, the low processing temperatures were effective in minimizing the degradation of fibers during the processing. However, only a restricted shape of composite is formed by the hot-pressing. The processing using a molten HSS eliminates this type of

<sup>\*</sup>Corresponding author:

Tel:+81-99-285-8325

Fax: +81-99-257-4742

E-mail: hirata@apc.kagoshima-u.ac.jp

shape restriction of forming. A big problem in such a high temperature processing may be the degradation of long fibers due to the grain growth of alumina or sintering between fibers in a filament yarn. This paper reports the challenging work on a molten state processing of the alumina fibers/high-speed steel composite.

## **Experimental Procedure**

A high-speed steel powder (Mitsubishi Steel Mfg. Co., Ltd., Japan) with a cumulative particle size distribution of 4.43 µm/10%, 11.23 µm/50% and 24.93 µm/ 90% and the following chemical composition: 81.91 mass % Fe, 0.85 mass % C, 4.03 mass % Cr, 1.94 mass % V, 4.88 mass % Mo, and 6.01 mass % W was used. The true density measured with a pycnometer using kerosine was 7.931 g/cm<sup>3</sup>. The unidirectional alumina fiber yarn of 195 µm thickness (1000 filament/yarn, Mitsui Mining Material Co., Ltd., Japan) were made of long fibers with an average diameter of 10 µm and a chemical composition of  $Al_2O_3 > 99.5$  mass %. The true density of the fibers was 3.649 g/cm<sup>3</sup>. The tensile strength and tensile modulus are reported by the supplier to be 1.76 GPa and 323 GPa, respectively. Two types of laminated green composites were formed in cylindrical graphite molds. Six 5 mm thick layers of HSS powder and five 195 µm thick layers of alumina varn were laminated alternatively at 2.0 vol% fiber content in a 50 mm diameter mold (sample 1). Another green composite (sample 2) was formed by putting a 1cm thick HSS powder layer on the top of a 1cm thick alumina fiber layer with 20 vol% fiber content in a graphite crucible. These samples were heated to 1630 °C at a rate of 10 K/minute, and held for 5 minutes in an Ar atmosphere (FVH-5 type, Fuji Denpa Kogyo Co., Japan). The microstructures of the heated composites were observed by optical microscopy and scanning electron microscopy (SM-300, Topcon Co., Japan) after chemical etching with a mixed solution of 99.5 vol% ethanol/15.75 M-HNO<sub>3</sub> = 20/1 (volume ratio). Polished surfaces were etched for 15 seconds ~ 30 minutes at room temperature. The Vickers hardness of samples 1 and 2 was measured at a load of 2.94 N on the surface polished with 1 µm diamond paste (Model MVK-F, Akashi Seisakusho Co., Tokyo, Japan).

## **Results and Discussion**

#### Microstructures

Figure 1 shows the appearance of the alumina fiber (2 vol%)-HSS layered composites cooled from 1630°C in an Ar atmosphere. No layered structure was produced in sample 1. Molten HSS spread on the bottom of the graphite crucible. Alumina fibers were observed on the top of the solidified HSS, and the fibers were very brittle. The layered structure of sample 2 (20 vol%)



Fig. 1. Appearance of the alumina fiber/HSS layered composites before and after heating at 1630°C in an Ar atmosphere.

fiber) was completely changed after the heating, resulting in an upper layer of alumina fibers and a lower layer of HSS. The above phenomena observed in samples 1 and 2 are ascribed to the density difference and low wettability between the two components. Figure 2 shows SEM photographs of (a) spherical particles HSS on alumina fibers in sample 1 and (b) transition metal carbides elongated in the HSS of



**Fig. 2.** SEM photographs showing (a) spherical particles HSS on alumina fiber in sample 1, and (b) transition metal carbides elongated in the HSS of sample 2.

sample 2. The alumina fibers in sample 1 were sintered to each other. In addition, a rough surface was produced due to the grain growth of the alumina. No penetration of the molten HSS into the unidirectional alumina yarn was seen, indicating a low wettability of molten HSS on the alumina fibers. No alumina fiber was also seen in the solidified HSS layer in Fig. 2(b). The above information in Figs. 1 and 2 indicates the difficulty of producing a uniform distribution of

alumina fibers in molten HSS under no external pressure. On the other hand, the hot-pressing of laminated green composites at low temperatures of 900°-1100°C was successful in forming the homogeneous dense structure without phase separation [4, 5]. The key factors to produce dense laminated composites are the control of the heating temperature and applied pressure.

Although it was difficult to produce the alumina fibers-HSS laminated composites by a molten state processing, clarifying the microstructural and mechanical difference of the HSS matrix densified by hotpressing and molten state processing is important to improve the processing strategy of the composites. Figure 3 compares the chemically etched microstructures for sample 2 (d) and the monolithic HSS hotpressed at (a) 900°, (b) 1030° and (c) 1100°C under a pressure of 39 MPa in an Ar atmosphere. The features of an alternative array of needle - like white and black grains in Fig. 3(d) represents the eutectoid structure of ferrite ( $\alpha$ -Fe) and cementite (Fe<sub>3</sub>C) produced from austenite (y-Fe) during the cooling process (entectoid temperature: 723°C) of molten HSS from 1630°C [8]. The other transion metal (Cr, V, Mo, W) carbides may coexist with cementite as the black grains. No formation of needle-like grains was observed in the monolithic HSS hot-pressed below 1100°C. (Figs. 3(a)-(c)). The tiny black grains in Figs. 3(a)-(c) are transition metal carbides. According to the phase diagram of the Fe-C system [8],  $\gamma$ -Fe is produced in the carbon content range of 0-1.5 mass % at 900°-1100°C. That is, the matrix of hot-pressed HSS changes to pearlite (a- $Fe+Fe_3C$ ) below 723°C during the cooling process. The formation of  $\alpha$ -Fe and Fe<sub>3</sub>C in the hot-pressed HSS samples was identified from the X-ray diffraction patterns. As seen in Fig. 3, the increased hot-pressing temperature caused (1) densification of the pearlite and (2) grain growth of the transition metal carbides. Apparently, the processing through the molten HSS provided significant grain growth of the pearlite and the transition metal carbides.

### **Densification and Hardness**

Table 1 compares the densification behavior of hotpressed monolithic HSS and molten HSS composite. The HSS powder was densified above 99% of theoretical density by hot-pressing at 1030°-1100°C. The



**Fig. 3.** Chemically etched microstructures for the HSS hot-pressed at (a) 900°, (b) 1030°, and (c) 1100°C, and (d) for the HSS solidified from 1630°C.

Sample	Processing	Temperature (°C)	Applied pressure (MPa)	Relative density (%)	Open pore (%)	Closed pore (%)
HP1	Hot-pressing	900	39	84.97	14.56	0.47
HP2	Hot-pressing	1030	39	99.85	0.05	0.10
HP3	Hot-pressing	1100	39	99.67	0.10	0.23
M1	Molten	1630	0	90.47	7.06	2.47
M2	Molten	1630	0	86.79	4.84	3.36

Table 1. Relative density and porosity of HSS processed by hot-pressing and by solidification of molten HSS



Fig. 4. Vickers hardness of the HSS as a function of processing temperature.

molten HSS at 1630°C was solidified to 87-90% of theoretical density. The formation of open and closed pores in the solidified HSS may be associated with (1) the high viscosity of molten HSS, (2) the coexistence of alumina fibers (obstacles for the migration of molten HSS) and (3) chemical reactions between the molten HSS and graphite crucible (formation of Fe<sub>3</sub>C).

Figure 4 shows the Vickers hardness of the HSS matrix as a function of processing temperature. The hardness increased with an increase of processing temperature, suggesting that the hardness was affected



**Fig. 5.** Shape of Vickers indenter impression at 2.94 N on the eutectoid structure formed during the cooling of molten HSS at 1630°C.

by the density and grain size of HSS. The good linearity of hardness in the temperature range of 1030° to 1630°C indicates a stronger influence of grain size than density (Fig. 3, Table 1). On the other hand, the increased hardness in the temperature range of 900° to 1030°C may be strongly associated with the increased density. Figure 5 shows the shape of the diamond indenter impression at 2.94 N on the chemically etched surface of the HSS processed at 1630°C. No crack was formed in the eutectoid structure, suggesting a high fracture resistance.

## Conclusions

When the layered structure of HSS powder and alumina yarn was heated at 1630°C in an Ar atmosphere, phase separation occurred to form an upper layer of fibers and a lower layer of the molten HSS because of the density difference and low wettability between the two components. The solidified HSS consisted of the eutectoid structure of pearlite (alternative array of needle-like grains of  $\alpha$ -Fe and Fe<sub>3</sub>C) and transition metal carbides. The Vickers hardness of dense HSS processed by hot-pressing or solidified from molten HSS was affected by the grain size of constituent phases, and reached 9.14 GPa in HSS prepared at 1630°C. Our previous study revealed that the hotpressing of laminated composites at 900°-1100°C under a pressure of 39 MPa was effective in producing homogeneous dense structures without phase separation. The combination of the results in the above two processings concludes that the key factors to produce dense laminated composites are the control of the heating temperature and applied pressure.

## References

- A. Hirano, H. Nakayama, J. Funakoshi, H. Okano and A. Kosaka, J. Soc. Mat. Sci., Japan, 46[10] (1997) 1161-1166.
- 2. A. Suzuki, Kinzokubinran (Metal Hand Book), Edited by The Japan Institute of Metals (Maruzen, Tokyo, 1976) p. 790.
- M. Inoue and K. Suganuma, Bull. Ceram. Soc. Japan, 34[6] (1999) 450-454.
- Y. Hirata, Y. Nakashima, Y. Niibo, S. Sameshima, S. Uchida, S. Hamauzu and S. Kurita, Proc. of the 9 th Symp. on Refractories for the Aluminium Industry, Edited by G.

Oprea (The Minerals, Metal and Materials Society, 2000) pp. 95-108.5. Y. Niibo, S. Sameshima and Y. Hirata, J. Ceram. Proc. Res.

- 1[2] (2000) 83-87.
- 6. A.R. Bunsell and M.H. Berger, Key Eng. Mater. 127-131

(1997) 15-26.

- 7. V. Lavaste, M.H. Berger, A.R. Bunsell and J. Besson, J. Mater. Sci. 30 (1995) 4215-4225.
- 8. P. Gordon, Principles of Phase Diagrams in Materials Systems (Robert E. Krieger Pub. Co., Florida 1983) p.171.