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# Influence of Ti<sub>3</sub>SiC<sub>2</sub> content on tribological properties of NiAl matrix

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# ABSTRACT

NiAl matrix self-lubricating composites (NMCs) with various contents of  $Ti_3SiC_2$  were fabricated by in situ technique using spark plasma sintering. The effects of  $Ti_3SiC_2$  content on tribological properties of NMC were investigated. The results showed that NMC were composed of the matrix phase of NiAl alloy, enhanced phase of TiC and lubricating phases of  $Ti_3SiC_2$  and C. NMC with 10 wt.%  $Ti_3SiC_2$  exhibited low friction coefficient of 0.60 and wear rate of  $5.45 \times 10^{-5}$  mm<sup>3</sup> (N m)<sup>-1</sup> at the condition of 10 N-0.234 m/s at room temperature. The optimum content of  $Ti_3SiC_2$  was 10 wt.%. The excellent tribological performance of NMC could be attributed to the balance between strength and lubricity, as well as synergetic effect of enhanced phase and lubricating phases. The wear mechanisms changed with the increasing of the doped content of  $Ti_3SiC_2$ .

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# 1. Introduction

Thanks to high melting point (1638 °C), high thermal conductivity (70 W  $m^{-1}$  K<sup>-1</sup>), low density (5.95 g/cm<sup>3</sup>) and excellent oxidation resistance [1-4], NiAl intermetallic compound is regarded as potential high temperature structural material having promising applications including turbochargers, high temperature dies and molds, furnace fixtures, rollers in steel slab heating furnaces, hydroturbines, cutting tools, pistons, valves and various components within gas turbines [3,5,6]. However, like most intermetallic compounds, NiAl is rather brittle for an engineering material at low temperatures and exhibits a brittle-to-ductile transition temperature (BDTT) at 500-700 °C, depending upon composition, grain size, processing conditions and strain rate. Surprisingly, the creep resistance of NiAl is relatively poor above the BDTT despite the fact that NiAl has an ordered crystal structure with limited slip systems. Composite strengthening is one approach that can improve both toughness and creep resistance. Many studies have been carried out to synthesize reinforcing phase in NiAl matrix. The addition of TiB<sub>2</sub> as a particulate reinforcement to NiAl increases the hardness of the composite with respect to NiAl, and reduces the wear rates (WRs) at all volume fractions on garnet and Al<sub>2</sub>O<sub>3</sub> abrasives. Abrasion on SiC resulted in a minimum of the wear rate for the composite with 20 vol.% TiB<sub>2</sub> for most conditions [7].

Wear resistance of NiAl is the challenging factor which has to be considered since some of the proposed applications, e.g. turbine blade tips, are subject to sliding type wear [6]. Wear testing of three NiAl alloys containing 45 at.%, 48 at.% and 50 at.% aluminum indicated that all of them had low coefficient of friction (COF) and WRs. Wear rate is hardness-dependent for these NiAl alloys, and higher hardness leads to lower wear. Meanwhile, wear process of NiAl is dominated by plastic deformation for all compositions [8]. From the tribological point of view, TiC reinforced NiAl intermetallic matrix composites are expected to process excellently both abrasive and adhesive wear resistance because of high hardness of the TiC reinforcements as well as the strong atomic bonding characteristic and abnormal hardness-temperature relationship of nickel aluminides. As a result, TiC reinforced nickel aluminide matrix composite coatings are potential candidates of wear-resistant coating materials [9].

NiAl intermetallic matrix composites containing solid lubricants have attracted much attention as high-temperature selflubricating materials. NiAl–31BaF<sub>2</sub>–19CaF<sub>2</sub> (mass%) prepared by spark plasma sintering (SPS) exhibited the low friction coefficients and wear rates at 873–1073 K [10]. Zhu et al. [11] investigated the high temperature sliding tribological behavior of NiAl matrix composites with addition of oxides (ZnO/CuO). At 1000 °C, the wear rate of NiAl matrix composites with ZnO was about  $7 \times 10^{-6}$  mm<sup>3</sup> (N m)<sup>-1</sup>, and the superior wear resistance was due to the formation of the ZnO layer on the worn surface. Moreover, the NiAl



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matrix composites with CuO showed self-lubricating performance at 800  $^{\circ}$ C, which was attributed to the presence of the glaze layer containing CuO and MoO<sub>3</sub>.

As a new solid self-lubricating material,  $Ti_3SiC_2$  has attracted much attention recently. Like graphite,  $Ti_3SiC_2$  has a hexagonal structure with a space group of P6<sub>3</sub>/mmc. Besides,  $Ti_3SiC_2$  is readily machined as graphite and possesses excellent thermostability. Thus, it was proposed that  $Ti_3SiC_2$  could be a new solid self-lubricating material with low COF and WRs from low to high temperature ranges [12–15].

However, meager information is available as regards the development of NiAl matrix self-lubricating composites by use of the Ti<sub>3</sub>SiC<sub>2</sub> and C dual lubrication phases and TiC enhanced phase. Recently, in situ technique has been developed to fabricate metal matrix composites. The composites exhibit a clean matrix/ reinforcement interface, which leads to better improvement in mechanical properties and tribological properties of the in situ composites [16,17]. Compared with other traditional sintering methods, the main advantages of SPS are the fast heating and cooling speeds, short sintering time, fine grain size of the prepared materials, controlled organizational structure, energy saving, environmental protection and so on [18–20].

In this study, NiAl matrix self-lubricating composites (NMCs) with different contents of  $Ti_3SiC_2$  were prepared by in situ technique using SPS. The effects of  $Ti_3SiC_2$  content on the dry sliding friction and wear behavior of NiAl matrix self-lubricating composites at the load of 10 N and sliding speed of 0.234 m/s for 20 min at room temperature were investigated through the determination of COF and WRs and the analysis of the compositions of wear debris, worn surfaces of both NMC and  $Si_3N_4$  ceramic ball friction pair.

### 2. Experimental details

### 2.1. Materials

NMC with different contents of Ti<sub>3</sub>SiC<sub>2</sub> were prepared by in situ technique using SPS. The composite powders of NiAl matrix were composed of commercially available Ni, Al, Mo, Nb, Fe and B powders by atomic ratio of 48:50:1:1:0.5:0.02, whose average particle sizes were about 30–50  $\mu$ m. The weight fractions of Ti<sub>3</sub>SiC<sub>2</sub> in the composites were fixed at 0 wt.%, 5 wt.%, 10 wt.%, 15 wt.% and 20 wt.%, respectively. The fabrication process of Ti<sub>3</sub>SiC<sub>2</sub> powder (5 µm in average size, 99.5% in purity) was described in detail elsewhere [21]. NiAl alloy without Ti<sub>3</sub>SiC<sub>2</sub> was denoted as NA. While NB. NC. ND. NE represented NMC with 5 wt.%. 10 wt.%. 15 wt.% and 20 wt.% Ti<sub>3</sub>SiC<sub>2</sub>, respectively. The starting powders were mixed by high energy ball-milling in vacuum. Balls and vials were made of hard alloy. The charge ratio (ball to powder mass ratio) employed was 10:1. The milling time and speed are 10 h and 200 rpm respectively. After being mixed and dried, the mixtures were then sintered by SPS using a D.R. Sinter<sup>®</sup> SPS3.20 (Sumitomo Coal & Mining, now SPS Syntex Inc.) apparatus at 1100 °C under a pressure of 30 MPa for 5 min in pure Ar atmosphere protection. The heating rate was 100 °C/min. The cylindrical graphite molds with an inner diameter of 20 mm were used. The as-prepared specimen surfaces were ground to remove the layer on the surface and polished mechanically with emery papers down to 1200 grad, and then with 0.05  $\mu$ m wet polishing diamond pastes.

# 2.2. Vicker's microhardness and density

The Vicker's microhardness of each as-received specimen was measured, according to the ASTM standard E92-82 [22,23], using an HVS-1000 Vicker's hardness instrument with a load of 1 kg and a dwell time of 8 s. Five tests were conducted and the mean va-

lue was given. The density of as-prepared specimens was determined by Archimedes' principle according to the ASTM Standard B962-08 [24,25].

### 2.3. Tribological test

The tribological test was conducted on a HT-1000 ball-on-disk high temperature tribometer according to the ASTM Standard G99-95 [26,27]. The disks, which were the as-prepared materials, were cleaned with acetone and then dried in hot air before test. The counterpart ball was the commercial  $Si_3N_4$  ceramic ball with a diameter of 5 mm (about HV 15 GPa). The test temperature was room temperature. The sliding speed and applied load were 0.234 m/s and 10 N respectively. The friction radius was 2 mm. The testing time was 20 min. The COF was automatically measured and recorded in real time by the computer system of the friction tester. The wear quantity of NMC was measured by the weighting method, which measured the mass loss of the samples for every friction process. The tests for every given conditions were repeated three times to obtain reliable data. The average value was used as the evaluating data.

#### 2.4. Analysis

The surfaces of the as-prepared specimens were examined by XRD with Cu K $\alpha$  radiation at 30 kV and 40 mA at a scanning speed of 0.01 s<sup>-1</sup> for the identification of the phase constitution. The morphologies and compositions of worn surfaces of NMC and Si<sub>3</sub>N<sub>4</sub> ceramic ball friction pair, wear debris were analyzed by a SIRION 200 field emission scanning electron microscope (FESEM) equipped with energy dispersive spectroscopy (EDS) and a JAX-8230 electron probe microanalyzer equipped with EDS.

# 3. Results and discussion

#### 3.1. Compositions of NMC

Fig. 1 was the XRD patterns of NMC with different contents of  $Ti_3SiC_2$  prepared by in situ technique using SPS. It could be found that the diffraction peaks primarily belonged to the NiAl, Ni<sub>3</sub>Al and  $Ti_3SiC_2$  phases. Additionally, it was clear that there were TiC and C phases existing in the NiAl matrix composites, which were attributed to the decomposition reaction of  $Ti_3SiC_2$  and carburization role of graphite atmosphere caused by the graphite mold.



Fig. 1. XRD patterns of NMC doped with varying weight fractions of Ti<sub>3</sub>SiC<sub>2</sub>.

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