



# Investigation on subsurface layer of Ni<sub>3</sub>Al-alloy and its composites induced by friction

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# Abstract

Friction coefficient and specific wear rate of a Ni<sub>3</sub>Al-based NAC-alloy and its composites, with 6 vol.%  $Cr_3C_2$ - and MnS-particle additions respectively, were studied by pin-on-disk tribological test. Then, the worn surfaces of the tested materials were characterized by using nanoindentation method. It was recognized that the wear properties of the investigated materials are conducted to their strain hardening effect induced by friction. Nanoindentation outlined thickness of the strained subsurface layer and distribution of Nano-hardness in the layers. The added hard  $Cr_3C_2$ -particles reduced the thickness of the subsurface layer, but kept a near-same peak-hardness of the friction surface as the monolithic NAC-alloy. A steep negative slope of hardness in the subsurface layer of NAC-alloy/ $Cr_3C_2$  composite may relate to a lower specific wear rate of the composite. MnS-particles were functioned as a solid lubricant in a NAC-alloy/MnS composite. An ineffective strain hardening effect may lead to a less protected surface layer against wearing, resulted in a less improved specific wear rate of the studied composite.

# 1. Introduction

One of the most attractive engineering properties of Ni<sub>3</sub>Al alloys is their increasing yield strength with increasing temperature up to about 650°C. Therefore, the alloys are considered promising in high-temperature construction applications and have been attracting great interest for many years [1-5]. This type of unique strength behaviour also suggested that the Ni<sub>3</sub>Al-based intermetallic alloys may have good wear properties in the peak-strength temperature range. Consequently, investigations of their sliding friction and wear behaviour have been initiated [6-8]. The works indicated that the greater the proportional improvement in yield strength with temperature is, the greater the improvement in wear becomes. But so far, the studies on the sliding friction and wear for Ni-aluminides were mostly limited on the laboratory investigation. Further work aimed to industrial applications is needed.

The objective of our previous work [9] is to improve the understanding of the service process behaviour of Ni<sub>3</sub>Al-based materials in the engine running system in comparison to commercial vermicular graphite cast iron. The results revealed that a selected single phase Ni<sub>3</sub>Al alloy showed friction coefficient and specific wear rate are similar to that of wear-resistant graphite cast iron under the same loading condition. Unfortunately, the single-phase Ni<sub>3</sub>Al alloy exhibited weaker performance and caused serious wear of grey cast iron counterpart. Added hard Cr<sub>3</sub>C<sub>2</sub> particles in the Ni<sub>3</sub>Al-matrix composite reduced wear on both sides of friction pair obviously. Addition of soft MnS particles in the composite functioned as a solid lubricant, resulted in a low friction coefficient and a low wear rate on its counterpart. Therefore, it was recognized that the monolithic Ni<sub>3</sub>Al-alloy may not be suitable and applied to a certain wearing condition, but may work well as a matrix material to develop high-temperature and high-strength wear resistant composites, tailored with hard or soft particles, according to specified applications.

In general,  $N_{i_3}AI$ -based materials were worn in a manner typical of that of many metals exhibiting severe metallic wear when unlubricated, which related to the structure and thickness of the most heavily deformed and fragmented subsurface layer. Therefore, nanoindentation was applied on the worn specimens in this study to characterise the distribution profiles of nanohardness in the subsurface layer induced by friction. A further understanding of the wear behaviour of the studied Ni<sub>3</sub>AI-based materials and relevant knowledge concerning the alloys development are expected.

# 2. Experimental methods and results

## 2.1 Test materials and preparation

A Fe-alloyed Ni<sub>3</sub>Al (NAC-alloy) was selected in the work. Composition of the alloy is Ni-18.8Al-10.7Fe-0.5Mn-0.5Ti-0.2B (at. %). The monolithic NAC-alloy and its composites, with addition of 6 vol.  $%Cr_3C_2$  and 6 vol. %MnS particles respectively, were produced by hot isostatic pressing (HIP) process, see Table 1. The powders of NAC-alloy for HIP process were prepared by using Plasma Rotating Electrode Process (PREP). The HIP process bulk specimens are in dimension of Ø70 mm x 150mm. The powder sizes of the NAC-alloy,  $Cr_3C_2$  and MnS applied in the process were in a range of 45-120  $\mu$ m. The densities of the NAC-alloy,  $Cr_3C_2$  and MnS used to calculate the compositions of the composites in volume percentage are 7.25 g/cm<sup>3</sup>, 6.68 g/cm<sup>3</sup> and 3.99 g/cm<sup>3</sup>, respectively. The HIP process was applied at a heating temperature of 1130 -1160°C under 140 MPa for three hours.

## 2.2 Pin-on-Disk test

A conventional pin-on-disk tribometer was used to evaluate the friction coefficient of the friction pair and specific wear rate of the test materials. A grey cast iron with a composition of Fe-3.2C-1.1Si-0.8Mn-0.2P-0.1S-0.02B-1.0Cu-0.22V (wt. %) was utilized as the counterpart disk material in testing, which is usually used as a liner material in ship engines. Dimensions of the pin and disk are Ø3 mm x 16 mm and Ø30 mm x 4 mm, respectively. The applied normalising pressures were 2.83 MPa for determining the friction coefficient and





5.66 MPa, for measuring the specific wear rate. The measured wear rates of the tested specimens are reported in terms of Archard's specific wear rate (mm<sup>3</sup>/N•m). In the case, wear volumes of the pin samples were calculated from the weight lost during testing by assuming a density of 7.25 g/cm<sup>3</sup> for the monolithic alloy (pin #1) and 7.23 g/cm<sup>3</sup> and 7.12 g/cm<sup>3</sup> for composite pin #2 and #3, respectively. The collected friction coefficients and specific wear rates are given in Table 2.

#### 2.3 Nanoindentation measurement

The nanohardness of the worn specimens was measured after pin-on-disk testing to investigate the hardness variation of the Ni<sub>3</sub>AI matrix in the subsurface layer due to friction. The indentations were performed on the Ni<sub>3</sub>AI matrix in the studied microstructure. Therefore, longitudinal cross-sections of the tested pins, which are perpendicular to their friction surfaces, were prepared for nanohardness testing.

Nanoindentation test was carried out with a fully calibrated Nanoindenter XP (Agilent) equipped with a standard Berkovich indenter. The applied load is 300 mN, resulted in an indentation depth of less than 1.8  $\mu$ m. The first indentation was located on the Ni<sub>3</sub>Al matrix at a distance of 10  $\mu$ m from the friction surface. Then the specimen stage was removed by moving the X- and Y-coordinates (see labelled directions in Figure. 1) by 30  $\mu$ m and 50  $\mu$ m for the second indent, respectively. Therefore, the surface distance of the next indentation was increased by 30  $\mu$ m. The operation was repeated until the serial sixth indentation was performed at a perpendicular distance of 160  $\mu$ m from the friction surface. In this case, the spacing between two neighbouring indents is greater than 50  $\mu$ m to reduce interference from the nearest indentation. Thus, the interval scale was maintained at 50  $\mu$ m for the seventh to tenth serial indents. Finally, the nano-hardness was measured on the unaffected substrate at a distance of 1.500  $\mu$ m from the worn surface. Four series of nanoindentation measurements were carried out on tested specimen pins #1, #2 and #3, respectively. Therefore, four indents were applied for each specified surface distance to obtain a statistical value. A map of indents on Pin #2 as an example was given in Figure 1. The collected experimental results of nano-hardness are illustrated in Figure 2.

Clearly, the subsurface layers were formed in the worn samples. The nano-hardness of all three tested samples decreased as the surface distance increased, though with different negative slopes. The estimated thickness of the subsurface layer was in the ranges of 400  $\mu$ m, 100  $\mu$ m and 200  $\mu$ m for pin #1, pin #2 and pin #3, respectively. The hardness values at a surface distance of 10  $\mu$ m in the three specimens varied; higher values were observed for pin #1 (5.63 GPa) and pin #2 (5.61 GPa) than for pin #3 (3.98 GPa). It was also recognized that the unaffected substrates of pin #1 and pin #3 have nearly same nano-hardness values of 3.60 GPa and 3.32 GPa, respectively; however a high hardness value of 4.40 GPa was obtained from pin #2. It may relate to the dissolution of Cr<sub>3</sub>C<sub>2</sub> particles in pin #2 by the HIP process, which resulted in an additional content of Cr (1.28 at %) in the matrix [10].

#### 3. Discussion

From our previous investigations, it was recognized that the monolithic NAC-alloy is comparable to the commercial vermicular graphite cast iron on wear properties. The graphite cast iron has an optimised phase constitution and microstructure to against wearing. In the case, the hard cementite phase Fe<sub>3</sub>C in the pearlite microstructure protected mostly against wearing and was assisted by graphite as a solid lubricant. In contrast, the studied monolithic NAC-alloy has a single Ni<sub>3</sub>Al phase in its microstructure. Therefore, it is reasonable to consider that the wear mechanism of Ni<sub>3</sub>Al-based materials differs from that of traditional multi-phase metallic alloys, and the wear of the NAC-alloy was mainly conducted to plastic deformation and intrinsic strain hardenability, which resulted in the formation of a wear-resistant subsurface layer. In fact, the researchers [11-17] have studied the subsurface layer by means of analytical electron microscopy, and indicated that large plastic strains and large rotation angles in the layer are achieved after very short sliding distances. It was determined that the thickness of the subsurface layer under normal conditions of friction is on the order of micrometres.

Intermetallic compounds like Ni<sub>3</sub>Al possess superdislocations due to their long-range ordered crystalline structure represented by a large Burgers vector. And, the deformation mechanism of superdislocation motion in long-range-ordered alloys leads to higher strain hardenability than in disordered alloys. In this work, it can be seen that a strong strain hardening effect induced a high hardness of 5.63 GPa on the surface layer of Ni<sub>3</sub>Al-matrix in pin #1. 50% increased hardness on the friction surface may use to explain the measured specific wear rate of the single phase Ni<sub>3</sub>Al-alloy, which is even comparable to the multi-phase graphite cast iron.

The addition of  $Cr_3C_2$  particles reduced the thickness of the subsurface layer to 100 µm but maintained the same peak hardness of the friction surface as that of the monolithic NAC-alloy, which revealed that the hard  $Cr_3C_2$  asperities effectively protected the friction surface and formed a thin subsurface layer. Thus, a deeper negative slope of hardness at the friction surface compared to the monolithic NAC-alloy was obtained. A steep negative slope of the hardness at the friction surface indicates that the hardening effect vanished rapidly with increasing surface distance in pin #2, resulting in relatively small wear debris particles. Therefore, low specific wear rates of this composite were observed.

As a soft solid lubricant, MnS particles reduced friction coefficient of the friction pair to 0.45. A lower stress induced by the friction force caused ineffective strain hardening on the friction surface of this composite. Therefore, a peak hardness of 3.98 GPa and 200 µm thickness of the subsurface layer of the NAC-alloy/MnS composite were observed, which may relate to a less protected subsurface layer to against wearing.

#### 4. Conclusion

- The wear properties of the Ni<sub>3</sub>Al-alloy are attributed to its strain-hardening effect. High peak hardness on the friction surface of the selected single-phase Ni<sub>3</sub>Al-alloy was identified. Gradual strain-hardening subsurface layer was recognized, and may use to understand the wear mechanism of the studied Ni<sub>3</sub>Al-based alloy.
- 2. The intermetallic matrix of composite reinforced by the Cr3C2-particles also showed high peak hardness on the friction surface, but a less thickness of the subsurface layer. A steep negative slope of the hardness at the friction surface may relate to an improved specific wear rate of the composite.
- 3. The soft MnS particles functioned as a solid lubricant, and decreased both the thickness of the subsurface layer and the peak hardness on the friction surface. An ineffective strain-hardening ability on the friction surface of this composite led to a low friction coefficient, but not a less specific wear rate of the tested composite.

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## Table 1. Designations of the tested material

Designation	Composition	Process	
1#	NAC-alloy	HIP	
2#	NAC-alloy+ 6 vol. % Cr <sub>3</sub> C <sub>2</sub>	HIP	
3#	NAC-alloy+ 6 vol. % MnS	HIP	

Table 2. The tribologically data of the tested materials from pin-on-disk tests

Sample	Friction Coefficient	Specific Wear Rate (x10-5 mm <sup>3</sup> /N•m)
	(20N)	(40N)
1#	$0.55 \pm 0.02$	1.38
2#	$0.68 \pm 0.02$	0.76
3#	$0.45 \pm 0.02$	1.54



Fig.1 A map of the indents in the subsurface layer of pin 2#. An arrow indicated the worn surface.



Fig.2 Nano-hardness versus surface distance in the subsurface layer of pin 1#, pin 2# and pin 3#.





# 5. References

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