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## Structural analysis of  $(Cr_{1-x}Si_x)N$  coatings and tribological property in water environment

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#### Abstract

A series of  $(Cr_{1-x}Si_x)N$  coatings were deposited by a multicathode unbalanced magnetron sputtering system (UBMS) using Cr and Si targets under  $Ar/N<sub>2</sub>$  atmosphere on stainless steel (AISI630). Si content x was varied from 0 to approximately 0.8 by changing the input power to Si target. X-ray diffraction (XRD) analysis revealed that the coating underwent a continuous structural change from crystalline (NaCl structure) to amorphous structure as  $x$  was increased. Average grain size, which was derived from the XRD measurements using the Scherrer's equation, decreased from 17 to 2 nm by increasing the Si amount x from 0 to 0.45. Hardness of  $(Cr_{1-x}Si_x)N$  coating increased slightly from 15 to 17 GPa by increasing the Si content x from 0 to 0.3. Tribological evaluations using a ball-on-disk tribometer of  $(Cr,Si)N$ coated substrates were conducted in distilled water against the (Cr,Si)N coating itself. The friction coefficient decreased drastically nearly down to zero as x was increased more than ca. 0.6. The specific wear rate was also decreased by a factor of 10 from  $4\times10^{-7}$  to  $5\times10^{-8}$  mm<sup>3</sup>  $N^{-1}$  m<sup>-1</sup>. A thin (approximately 10 nm) amorphous Si–Cr oxide layer was formed on the surface of worn section that was estimated to be formed by the tribochemical reaction during the tribological test. It was concluded that this tribochemically formed amorphous oxide layer was responsible for the ultralow friction behavior.

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### 1. Introduction

Recently, there is a certain demand to develop an oil-free (using water as a pressure-transmitting medium) hydraulic system. This water-based hydraulic system is advantageous compared to the conventional oil hydraulic system from the viewpoint of environmental compatibility and energy efficiency due to the low viscosity of the water itself. This kind of hydraulic system is expected to be used, for example, in the food industry and ship industry where pollution is not permitted or not desirable. However, a problem may arise when the pressure-transmitting medium is replaced from conventional mineral oil to water, due to the lack of sufficient tribological properties in water (e.g., friction and wear) of conventional metallic materials that

were used in the conventional oil based hydraulic devices. A conventional solution of this tribological problem is to use Si-containing bulk ceramic such as SiC or  $Si<sub>3</sub>N<sub>4</sub>$  for the components of water-based hydraulic devices. These Sicontaining ceramics are known to show excellent tribological behavior owing to the following tribochemical formation of amorphous silica at the sliding interface, which can reduce the friction coefficient nearly down to zero in water  $[1-3]$ .

 $Si_3N_4 + 6H_2O = 3SiO_2 + 4NH_3$ 

 $SiO_2 + H_2O = Si(OH)_4$ 

However, in general, these Si containing ceramics are expensive, brittle and hard to machine in precision for industrial components. An alternative approach is to take the advantage of metallic materials that is relatively inexpensive and easy to machine. The required tribological properties in water environment can be improved by

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Fig. 1. Relationship between Si content x in  $(Cr_{1-x}Si_x)N$  coating and input power ratio to Si target.

adding a layer which has sufficient tribological characteristics in water environment.

In this work, Si-containing binary coating system of  $(Cr_{1-x}Si_x)N$  was selected as a candidate of tribological coating in the water environment and the change of structural and mechanical properties was investigated as a function of Si content x. Also, tribological behavior of  $(Cr_{1-x}Si_x)N$  coatings with various Si contents x were evaluated in the water environment.

#### 2. Experimental details

 $(Cr_{1-x}Si_x)N$  coatings with various Si contents x were deposited by a unbalanced magnetron sputtering system (UBMS): UBMS202 Kobe Steel) with two round UBM sources (6 in. diameter) installed in a counter-facing arrangement. Metallic Cr and Si targets were sputtered in an  $N_2/Ar$  mixture with 35/65 ratio to produce (Cr,Si)N coatings. These two sources were operated simultaneously at a power of 2.5 kW maximum and by changing the input power to both targets Si content x was controlled. WC–Co cutting inserts and AISI630 stainless steel disks (mirror polished, 55 mm diameter×5 mm thickness) and balls (9.53 mm diameter) were used as substrates for characterization of the coatings and tribological evaluation, respectively. Total pressure during the deposition was 0.6 Pa and substrate temperature was about 400 $\degree$ C. A bias voltage of 50 V was applied. Typical film thickness was about  $3 \mu m$  for WC–Co and from 6 to 7  $\mu$ m for stainless steel substrates. For most of the specimens, a metallic Cr interlayer was used to improve the adhesion to the substrate. Resultant coatings were characterized by standard analysis methods such as EDX (composition) X-ray diffraction (XRD; crystal structure, grain size), SEM (surface morphology), TEM (microstructure) and micro-Vickers hardness tester (hardness). Tribological evaluations in water environment were conducted by a ball-on-disk-type tribometer. This tribometer has a liquid container where the disk can be immersed in water. Normal load of 2 N was applied during the tests and sliding velocity was 0.5 m/s. Sliding length was 1000 m and

all tests were conducted at room temperature. Friction coefficients and specific wear rate was used to evaluate the tribological performance of tested specimens. After the tribological tests, worn sections of some samples were subjected to cross-sectional TEM analysis to examine the possible interface triboreaction.

#### 3. Results and discussions

Fig. 1 shows the change of Si content x in  $(Cr_{1-x}Si_y)N$  at various power input ratio to the Si target, which is defined as input power to Si target divided by a sum of input power to Cr and Si target. As a result, it was found that  $(Cr_{1-x}Si_x)N$  coatings with x from 0 to approximately 0.8 can be deposited in the current deposition method.

Fig. 2 shows the effect of Si content on the microhardness of (Cr,Si)N coatings. Without Si, the hardness of the coating is in between 14 and 15 GPa, with increasing hardness as Si content was increased. However, the hardness became nearly constant around  $x=0.4$  at a value of 17 GPa and no further increase in hardness was observed in this study.

[Fig. 3\(](#page--1-0)a), (b), (c) and (d) shows the surface SEM micrographs of  $(Cr_{1-x}Si_x)N$  coatings with various x values. Without Si, crystal grains were observed on the surface. However, as Si content was increased, the grain became smaller, and at  $x=0.35$ , no surface structure was observed.

[Fig. 4](#page--1-0) shows X-ray diffraction patterns of  $(Cr_{1-x}Si_x)N$ coatings with different Si content x. At  $x=0$ , the coating was crystalline and diffraction lines can be indexed to cubic NaCl structure of CrN. As  $x$  was increased, diffraction line from (111) and (200) from the CrN phase became wider and weaker, suggesting a deterioration of crystalline state. No diffraction line that originates to the coating layer was observed above x=0.68, suggesting that  $(Cr_{1-x}Si_r)N$  coatings with high Si contents were in amorphous state. Average grain size was calculated by the Scherrers's equation [\[4\]](#page--1-0) using the FWHM (full width of half maximum) of (200) diffraction line ([Fig. 5\)](#page--1-0). The average grain size continuously



Fig. 2. Relationship between Si amount x and hardness of  $(Cr_{1-x}Si_x)N$ coating.

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