NEW ESTIMATIONS OF SURFACE PROPERTIES FOR TRIBOLOGICAL STUDIES BY NEUTRON REFLECTOMETRY

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ABSTRACT

Tribological phenomena usually occur at a solid surface or an interface of solids. To carefully examine a tribological phenomenon, information on the state at the surface or the interface must be tried to be gotten from different angles. Estimations of the state, however, are generally considered to be difficult because there are few methods suit for adequately picking up the information on profiles and/or properties of the only nearest region to the surface. Especially for the case that the target is at the interface of metals, the estimation is more difficult because a standard electron beam cannot penetrate into either of the metal. The authors have, accordingly, developed a new estimation method applying a ‘neutron reflectometry’ to such a metal surface or a metal-metal interface. The reflectometry is suitable for analysis of vertical structures of the nearest regions to a surface as less than 1µm. In addition, since a neutron beam can penetrate into many kinds of common metals, the estimation by the neutron reflectometry can be useful even for the metal-metal interface. In this paper, we report some results obtained by some of several basic experiments on neutron reflectometry. Surface profiles and ultra-thin film thickness of PFP E lubricant at a metal surface and/or a metal-metal interface were estimated in the study.

BASIC CHARACTERISTICS OF A NEUTRON BEAM

Prior to outlining our experiments, we will firstly introduce some characteristics of a neutron beam. A neutron beam, as is well known, can either behave as a wave or a particle, like visible light or X-rays. The main characteristics of a neutron favorable for tribological studies are as follows:

- It can go deeply into the inner region of common metals because neutrons physically interact not with electrons but with atomic nuclei.
- It can even observe light atoms such as hydrogen or carbon, in contrast to X-rays, resulting in its suitableness for studies on lubricants.

A serious demerit of using a neutron beam for studies, on the other hand, is the difficulty of carrying out experiments with neutrons. Experiments cannot be performed in laboratories because a high-energy reactor or accelerator is required to generate the neutron beam. Experimental instruments we used, accordingly, are ‘MINE’ established at the JRR-3M reactor of the Japan Atomic Energy Research Institute (JAERI) and ‘ARISA’ at KEK (High Energy Accelerator Research Organization) in Japan. Both instruments are specialized as neutron reflectometers; the former applies monochrome neutron beams to a vertical face of a sample, while the latter does white pulse beams to a horizontal one. In the present study, we observed surface profiles and oil-film thickness at a metal surface and/or a metal-metal interface by those reflectometers.

OUTLINE OF EXPERIMENTAL INSTRUMENTS

A neutron beam naturally reflects from a material surface like a visible ray or an X-ray beam when its incident angle to the surface is below a critical one given as \( \lambda \cdot (Nb/\pi)^2 \), where \( \lambda \) and \( Nb \) are the wavelength of the beam and the neutron scattering length density, respectively. The reflectometry is a way to get information through the reflectivity of a sample surface or the interface between samples. It is generally utilized for analysis of vertical structures, especially for multi-laminar films. A simple schematic diagram of the experimental instrument ‘MINE’ is shown in figure 1, for an example. In the instrument, the neutron beam is generated by a nuclear reactor, and input through two slits to a sample holder (as shown in the figure). The wavelength of the beam is constant at 0.88 nm with an accuracy of 2.7 % in the case of MINE. A reflected beam from a sample is caught by a detector, which is movable over a 2θ range.

On the other hand, the instrument ‘ARISA’ in KEK is basically similar in geometry to MINE. Since white pulse beams are introduced into the ARISA, the TOF (time of flight) method is applied to identify the wavelength of the detected beam at each time.

ESTIMATION OF ROUGHNESS PROFILES OF A METAL SURFACE OR A METAL-METAL INTERFACE

Most surfaces of solids used for tribology are usually rough, at least on a microscopic scale, especially those of engineering materials. Roughness of a surface is one cause friction in any case. Surface profiles at a solid-solid interface, however, cannot be measured by general methods when both bodies are made from metals. However, there are a lot of cases in which it is necessary to grasp the state at the interface for tribological studies, such as in the process of “running in” or to “seize” of two bodies. As explained in the section, we thus tried to get information on roughness profiles of a metal surface or a metal-metal interface by using neutron reflectometry.

To basically confirm a reflectivity profile from a material surface with periodical roughness, a well-defined “grating” sample, as shown in a top of figure 2, was prepared. The grating sample was made by standard etching for silicone-plane. The etching depth was 75nm, the grating width was 10µm, and its pitch was 20µm. The reflectivity profiles of the sample obtained by the ARISA are shown in figure 2; one is for the case where the grating direction is set parallel to the neutron beam, while the other is set perpendicular. The ‘q’ on the horizontal axis in the figure is ‘wavenumber vector’, equal to \( 4\pi \sin \theta / \lambda \). As \( \theta \) is constant in the ARISA, a change of \( q \) corresponds to the distribution of wavelength of the flight beam. Several fringes for the perpendicularly setting case in the figure are formed by the interference between the reflected beams from the top,
interface between metals.

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REFERENCES

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