Nano-indentation Measurement for Heat Resistant Alloys at Elevated Temperatures in Inert Atmosphere

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Introduction. This study aims to develop a nano-indentation measurement approach to evaluate mechanical behaviors of heat resistant alloys at elevated temperatures. Then we have discussed issues of the thermal management and the durability of a diamond indenter in application on a sample of a nickel-based superalloy.

Thermal and Atmosphere Control System in Nano-indentation Measurement. For measurements at elevated temperatures, nano-indentation testing equipment using a high-temperature stage placed in a vacuum chamber on a vibration isolation dampening stage is used. The vacuum chamber is equipped with gas inlets allowing inner environment control and gas introduction, and with an external cooling system. A sample is placed between two independently-controlled heaters and heated simultaneously from the top and the bottom (Fig.1), then the indenter is heated passively. This thermal and atmosphere control system enables us to use a diamond indenter for the following high-temperature nano-indentation measurements.

Application to Nickel-based Superalloy. A sample made of gamma single-phase single-crystal of a nickel-based superalloy was prepared for the measurements. The nano-indentation measurements were carried out for the sample upto 800 °C. The elastic modulus and hardness of the sample were evaluated at elevated temperatures. The results are shown in Fig.2. In both elastic modulus and hardness, a continuous decreasing trend appeared and became obvious above 400 °C.

Creep and surface degradation. In high-temperature measurements, the creep effect and the surface degradation are inevitable. Generally these become dominant beyond 400 °C. For example, the creep effect can be observed as the negative stiffness in the

unloading segment of the load-displacement curve. The effect can be minimized by high loading/unloading rate and short dwell period at the maximum applied load. The surface degradation by oxidation appears even if the measurement is conducted in an inert or vacuum atmosphere. Fig.3 shows the surface roughness of the sample after 3 hours of exposure to the corresponding temperature in a high purity argon atmosphere, measured using a scanning probe microscope. The surface roughness increased above 600 °C and became drastically high at 800 °C. In high-temperature nano-indentation measurements, particular attention should be given to the surface state and an indentation should be performed at an appropriate area.

Conclusion. A new approach of nano-indentation measurement at elevated temperatures up to 800 °C in an inert atmosphere was developed in this study and evaluated the temperature-dependent mechanical behavior of gamma single-phase single-crystal of a nickel-based superalloy (1). By passive heating and calibration of area function at each temperature, reliable measurements could be performed with a diamond indenter. As a further research, a data-assimilation approach using a computational simulation has been developed to estimate temperature-dependent stress-strain relationships from high-temperature nano-indentation measurements (2).

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References.

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Fig.1 High-temperature nano-indentation system.

Fig.2 Nano-indentation results at elevated temperatures.

Fig.3 Surface degradation at elevated temperatures