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Short Note

NOTE ON THE FIELD-EMISSION MICROSCOPIC OBSERVATION OF NICKEL DEPOSITED ON TUNGSTEN

By Katuhiko Azuma

(Received October 31, 1960)

Field-emission patterns of nickel deposited on a tungsten tip were observed. Nickel was deposited on the tip from a ring of nickel wire of 0.25 mm. diameter. The ring was of 1.5 cm. diameter, placed a few mm. apart from the tip, heated at 1200-1300°C by current through the wire in ~10⁻⁴ mm Hg vacuum and anodically polarized against the tip by about 300 volts. The deposition of nickel was followed by the quantity of electricity transmitted. The coverage was about 10¹⁸ atoms cm⁻², as calculated from the quantity assuming that bivalent nickel ions were deposited uniformly on the semisphere on the tip opposite the nickel ring.*""

The tip thus treated was now subjected to heat treatments without electric field and to field-emission microscopic observation at room temperature after each treatment as follows. Nickel deposited on tungsten was in clustered states of fine crystallite at room temperature (Plate 2). These clusters migrated on the tungsten surface as observed from the variation of patterns from Plate 2 to 3 and then to 4 with rise of temperature of the treatments up to 1250°C. As the tip was maintained at 1250°C for further several minutes, the emission patterns were quite transfigured (Plates 5 and 6). A (111)-crystal plane was observed normal to the axis of the tip along with the rise of temperature above 1400°C until 1600°C was attained (Plates 9 and 10), when the pattern vanished altogether after about 30 seconds' treatment.

These phenomena might be attributed to the formation of nickel-rich tungsten alloy of b.c.c. crystal structure by the electrodeposition described. The unstable alloy would begin to change into α-phase of f.c.c. structure around 1250°C and, through the transient states of Plates 5 and 6, settle down to the α-phase with three-fold symmetry axis (Plates 7-10). The alloy would finally melt at a lower temperature of 1600°C than the melting point of pure nickel (1728°C) to blunt the tip, extinguishing the emission pattern.

*) Research Institute for Catalysis, Hokkaido University.
**) The deposition of nickel by evaporation was ignored.
Note on the Field-Emission Microscopic Observation of Nickel Deposited on Tungsten

Plate 1. Field-emission pattern of the tungsten tip before the electrodeposition of nickel, at room temperature. The emission current $i$ was 2.5 $\mu$A and the applied voltage $V$ was 8500 volts.

Plate 2. Nickel was electrodeposited on the tip. $i=2.5$ $\mu$A and $V=7050$ volts.

Plate 3. After heat treatment at about 1250 $^\circ$K for 40 seconds, $i=2.0$ $\mu$A and $V=9800$ volts.

Plate 4. After heat treatment at about 1250 $^\circ$K for 3 minutes, $i=0.8$ $\mu$A and $V=10000$ volts.
Plate 5. After heat treatment at about 1250°K for 16 minutes, $i=2.5 \mu A$ and $V=9820$ volts.

Plate 6. After heat treatment at about 1250°K for 33 minutes, $i=2.5 \mu A$ and $V=9180$ volts.

Plate 7. After heat treatment at about 1400°K for 1 minute, $i=2.5 \mu A$ and $V=6190$ volts.

Plate 8. After heat treatment at about 1400°K for 11 minutes, $i=2.5 \mu A$ and $V=6800$ volts.
After heat treatment at about 1400°K for 21 minutes, $i = 2.5 \mu A$ and $V = 6600$ volts.

After heat treatment at about 1600°K for 15 seconds, $i = 2.5 \mu A$ and $V = 7050$ volts.

The field-emission pattern of a tungsten tip of about 5000 Å radius, treated with ethylene as below, was observed with $\sim 10^4$ magnification, in a liquid oxygen filled cryostat**. Ethylene was introduced into the cell through a capillary pointed at the tip with its end 5 mm. apart from the tip. The pressure inside the cell was increased from $\sim 10^{-9}$ to $\sim 10^{-4}$ mm Hg by this procedure. This treatment with ethylene as well as the heat treatments described below were conducted without electric field.

*) Research Institute for Catalysis, Hokkaido University.

**) Wrinkles shown in Plates 1 to 8 were attributed to water drops condensed on the window of the cryostat, since they disappeared as the patterns were observed without liquid oxygen.