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# Investigation of sample heater material for super high temperature in-situ TEM observation in oxygen existing atmosphere

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In-situ Transmission Electron Microscopy (TEM) observation was method that sample is be able to be heated, cooled, loaded and bombarded by light and gas in TEM. And this method revealed behavior of sample in environment of sample used. An in-situ sample heating TEM observation in oxygen existing atmosphere has been used for especially development of automobile catalysts and fuel cell. In the past, tungsten, which is reasonable and fabricated easily, has been applied for heater of sample and gas. But when tungsten is heated in atmosphere oxygen exist, tungsten is oxidized. Tungsten oxide formed nano-rod on heated and sample, and adhered to sample. It was revealed that observations of material reaction were obstructed by tungsten oxide in the consequent. In this study, heater, which have thermal stability in atmosphere oxygen exist and don't obstructed to TEM observation, is fabricated, and that characteristics as heater for in-situ TEM observation was investigated. Rising temperature, drift speed at any temperature, and existence of heater material evaporation were verified.

Heater materials were platinum, platinum iridium and platinum rhodium that is platinum base metal, which is not oxidized easily. These metal wires were fabricated like coil (Fig. 1), and wire was installed on wire heating TEM holder. After that, TEM holder was inserted in TEM. Electric resistance heating was managed for heating of coil wire. Materials which melting point is known was put on the coil wire and heated. Current when the metal melted and melting point of the metal was inquired, and graph of relationship between current and temperature was prepared. Temperature of heated coil wire was read off from graph of relationship between current and temperature. When the wire is heated, coil wire was drifted by Lorenz force. So movement of heated coil wire was recorded by the video system, and drift speed was calculated from video. Existence of wire evaporation was confirmed by the following method. Silicon powder was located on another wire is different from heating wire, and heating wire was heated up to maximum temperature of heating wire. Since then silicon powders were observed by TEM. If new contrast will be available on silicon powders, it was decided that evaporation of wire material happened.

The Graph of relationship between current and temperature was shown in Fig. 2. each wire was be able to be heated up at around 1800 $^{o}$C. This temperature was similar to melting point that phase diagram indicate. Fig. 2 shows relationships of temperature and drift speed on each wire. Drft speed of platinum wire became high to 3000 nm/sec at about 400 $^{o}$C. In the case of platinum rhodium alloy, drift speed begun to become gradually high from 600 $^{o}$C and approached to 1500 nm/sec at about 1500 $^{o}$C. Drift speed of platinum iridium alloy wire had stabilized low speed until 1000 $^{o}$C, but after temperature of wire was over 1000 $^{o}$C, drift speed became drastically high and

approached to 2500 nm/sec (Fig. 3). The result of evaporation research was indicated. Evaporation weren't confirmed on platinum and platinum rhodium alloy wires, but evaporation was confirmed on platinum iridium alloy wire. These results conduce that platinum rhodium alloy wire befit for heater in oxygen existing atmosphere.

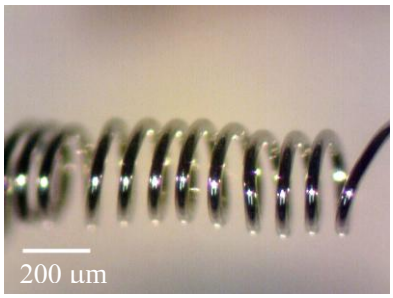


Fig. 1: image of fabricated coil wire

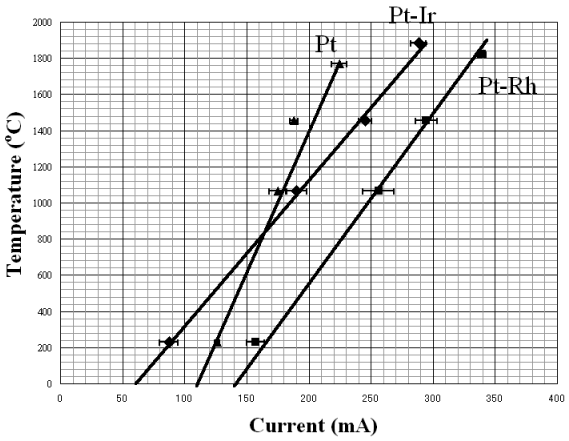


Fig. 2: graph of relationship between current and temperature of each wire coils

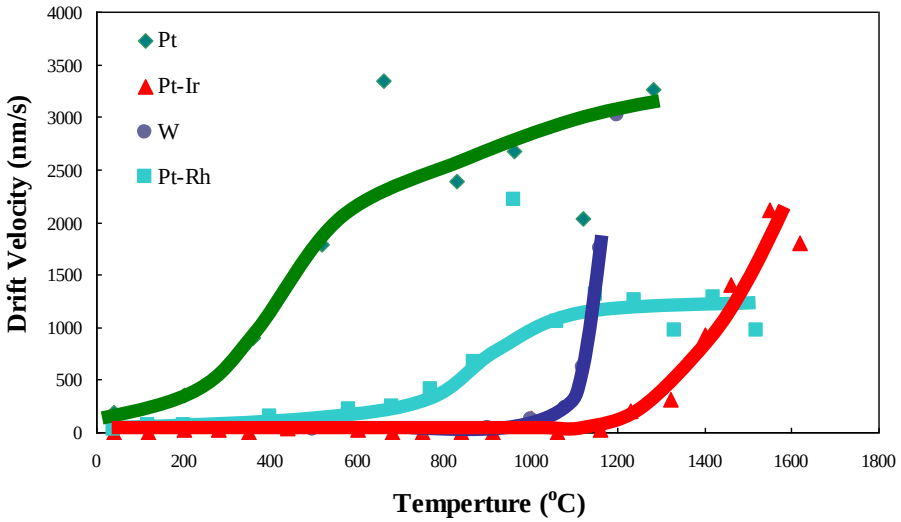


Fig. 3: graph of relationship between temperature and drift velocity of each wire coils