Title
A NEW 1000|C DOUBLE TILT STAGE FOR THE HITACHI 650 kV MICROSCOPE

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A NEW 1000°C DOUBLE TILT STAGE FOR THE HITACHI 650 kV MICROSCOPE

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A new 1000°C specimen holder has been developed for the Hu-650 electron microscope and is interchangeable with other holders for the recently designed orthagonal drive tilting stage(1). The requirements for the high temperature device were as follows: 1. An orthogonal double-tilt stage with all original design criteria maintained(1). 2. Capability of heating standard (3mm. dia.) metallurgical specimens to 1000°C. 3. Geometry and materials should be such that stage and microscope components shall not be damaged by heat from the specimen holder. 4. The electric current to the heating element shall impart a minimum effect upon the microscope electron beam. 5. Heating and cooling rates to be as rapid as possible.

The orthogonal drive double-tilt stage lends itself to modification for high temperature work very readily. The modification to the stage inner cone is shown in Fig. 1. Three beryllium-copper contact fingers are mounted close to the top of the inner cone and are coupled together by a .020" dia. gold wire. An insulated, braided copper wire (which is the power lead-in) is soldered to the gang of three contact fingers and is brought out of the stage area at a convenient location. The stage body (ground) serves as the other power lead. The contact fingers and connecting 0.20 dia. gold wire are insulated from the stage inner cone by alumina which was sprayed on in a molten state using a plasmatron machine and 301E alumina powder (400 mesh). These gave good density and adequate thickness and uniformity of coating. A special specimen holder cartridge assembly was developed (Fig. 2), which is interchangeable with others made for the above stage but with some unique features: 1. Electrical conductors are added to the cartridge to transmit power to the filament winding of the high temperature specimen holder tip. 2. A special high temperature specimen holder tip with wound filament furnace was manufactured. On the cartridge body, three longitudinal grooves and one circumferential groove were machined into which alumina was sprayed. A gold band was fit tightly over the circumferential groove. A set of three .020" dia. wires were spot-welded to beryllium-copper contact fingers, sprayed with alumina, individually spot-welded to the circumferential gold band and carefully laid into the longitudinal grooves. Miniature screws secure the contact fingers to the cartridge body. A final coating of alumina was sprayed over the longitudinal grooves implating the gold wires and upper ends of the contact fingers completing the main body of the cartridge. In designing the specimen holder tips, various materials were experimented with. The first successful assembly has a platinum body with a .003" dia. tungsten wire filament winding (Fig. 2). The platinum band serves to couple the filament winding to the electric current via the three beryllium-copper contact fingers of the cartridge body. The exterior of the high temperature specimen holder tip was threaded (6mm x 0.5mm) allowing adjustment of the elevation of the specimen with respect to the microscope beam focus point and/or the gymbal axes of the tilting stage. As this elevation adjustment is made, the platinum band acts much like a slip-ring, remaining in contact with the beryllium-copper contact fingers through the 1mm of allowable elevation adjustment. The heating characteristics of two "tip" assemblies are illustrated in Fig. 3 and examples of the response curves in Fig. 4. An alternative holder tip assembly was also manufactured, the body being made of molybdenum and the filament winding material 0.003" dia. tantalum. This is the one in current use, adequately satisfying the original design requirements. Current applications include direct studies of phase transitions e.g. order-disorder(2) and the annealing out of defects in irradiated metals and ion-implanted silicon(3).

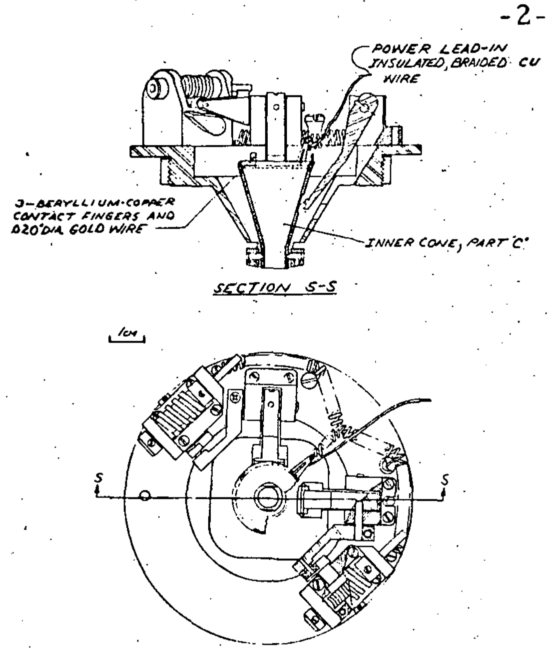

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FIG. 1 Section views of the orthogonal double-tilt stage with high temperature modifications.

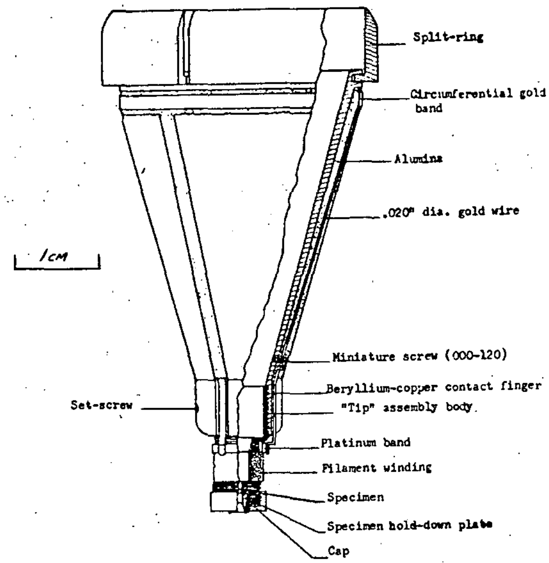

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FIG. 2 Partial section of the high temperature specimen holder and tip assembly.

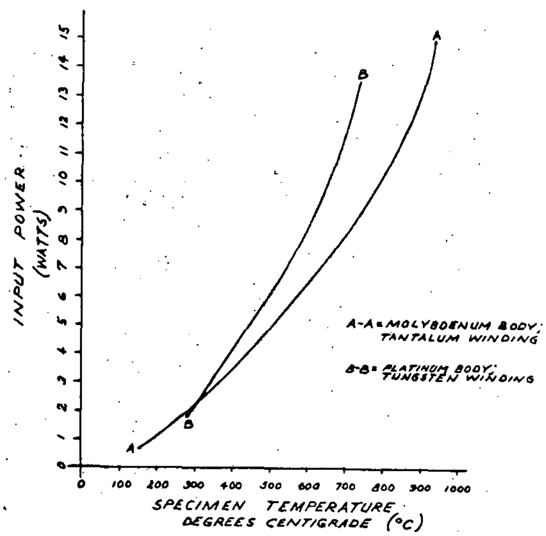

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FIG. 3 Heating calibration curves for both hot stage assemblies.

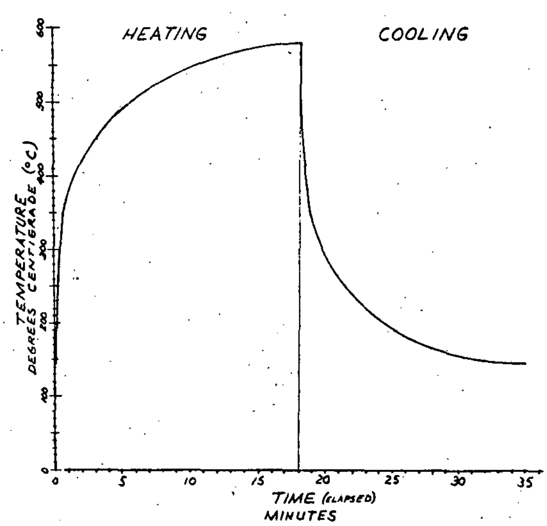

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FIG. 4 Heating and cooling characteristics, tested in simulated conditions in a vacuum bell jar.

1. Hodges, J.C. and G. Thomas: Univ. Calif. Lawrence Berkeley Laboratory Report No. 145 (1971).
2. Sinclair, R. and G. Thomas: (See Figs. 3, 4 of paper by G. Thomas, these proceedings).
3. Kosel, T., K. Seshan, W. Wu and J. Washburn: Univ. of Calif. Lawrence Berkeley Laboratory (research in progress).

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