Operating Experience with a New Reactive Ion Plating Unit for TiN Coating

By A. BRUCE CRAVEN
Research and Development Engr.
Vacuum Furnace Systems Corp.
Souderton, Pa.

During 1986, a physical vapor deposition (PVD) titanium nitride (TiN) reactive ion plating unit (model IP35L), the first of its kind shipped to the U.S., was put into operation. It is designed for electron beam evaporation of titanium, utilizing triode plasma configuration and hot filament ionization assist. This article outlines some start-up and learning problems, and describes present equipment, Vacuum Furnace Systems Corp. (VFS) modifications, and helpful processing tips.

The titanium nitride coating machine, Model IP35L designed and built by Tecvac Ltd. of England, is shown in Figs. 1 and 2. Its front control panel is depicted in Fig. 3. It has five power supplies located in two housings; the 5 kw electron beam gun power supply stands alone (see Fig. 4). The power supplies for sputter, plasma, filament and crucible bias functions are located beneath and behind the main console panel.

Fig. 5 shows the 23 in. (58 cm) diameter by 18 in. (48 cm) high work zone of the chamber with a sampling of parts that are coated. Parts of such varying masses and configurations would not be coated in the same run. Workpieces ranging from a few ounces up to a few hundred pounds can be handled in this machine.

The basic electrical circuits are shown in Fig. 6. Workpieces are negatively biased as a cathode to form the basis of attraction for nitrogen and titanium ions. The water-cooled, copper crucible which holds the titanium slug is positively biased to promote dispersion of titanium ions into the plasma. Plasma is enhanced by means of a hot filament assist which also has a negative electrical bias. Combining all three biases yields a plasma cloud in which the titanium and nitrogen ions circulate and are plated onto the workpiece.

The principle behind this PVD process is the evaporation method of depositing titanium onto the workpiece. By using a 270° bent electron beam, the slug of titanium is melted at which points its vapor of ions and the ionized nitrogen from the nitrogen partial pressure are deposited on the work, by means of the plasma and electrical biasing. This plasma is generated with a partial pressure of grade 5 high purity argon gas from a cylinder and nitrogen from a bulk liquid nitrogen tank. (Both gases are supplied by Air Products.)

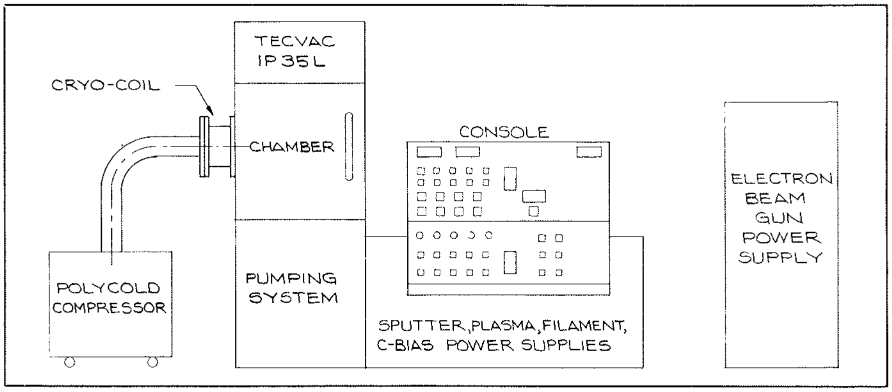

Fig. 2 Titanium nitride coating installation at Vacuum Furnace Systems Corp.

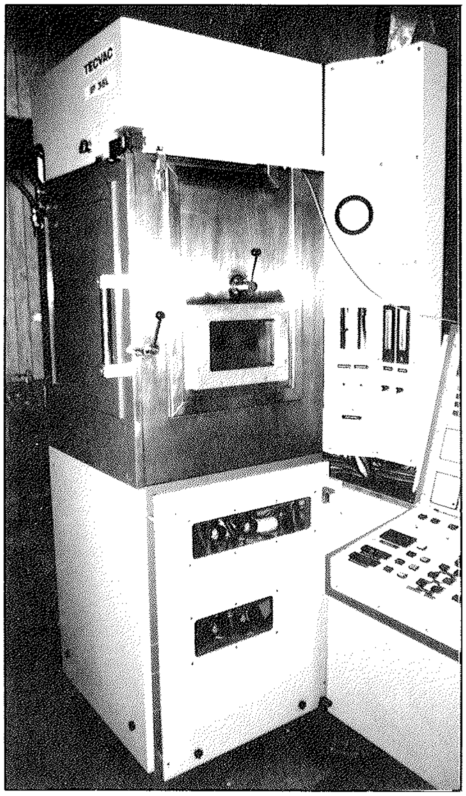

Fig. 1 Coating chamber of ion plating unit.

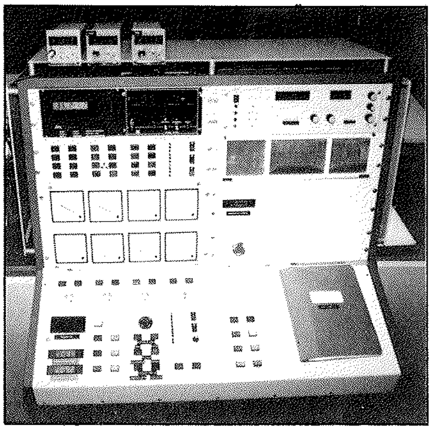

Fig. 3 Front control panel of ion plating unit.

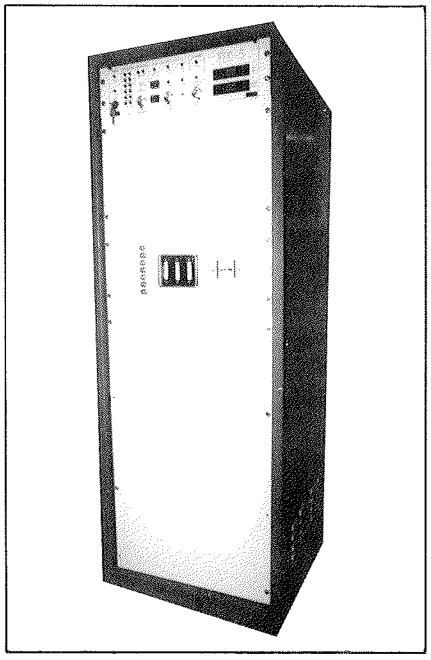

Fig. 4 Electron beam gun power supply.

Contrary to VFS ion nitriding experience which requires a minimum

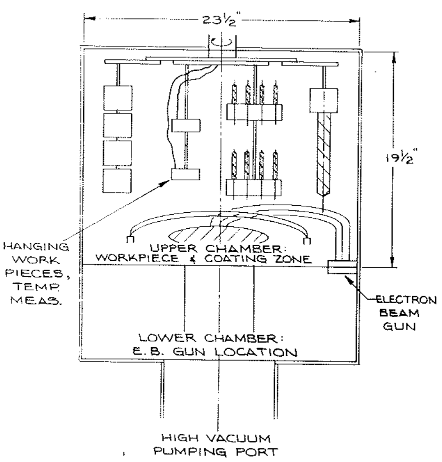

Fig. 5 View of chamber workzone of ion plating unit.

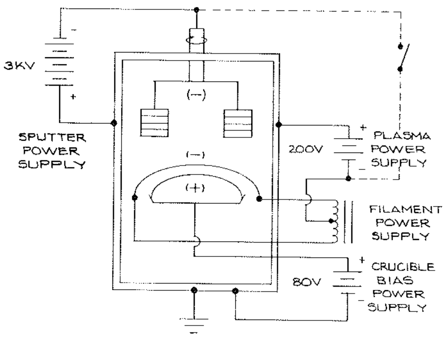

Fig. 6 Electrical line diagram of biasing on ion plating system.

current density of 1 ma/cm^2, TiN coating in this machine has required a much higher density on the order of 6 to 9 ma/cm^2. Anything less does not give good results on edges and flutes of cutting tools nor in some pemetration areas on other types of parts. Too low a current density often causes brown flute lines, believed to be nitrogen rich deposits, and generally poor aesthetic quality on corners and edges. A probable reason for this higher current density requirement is the great difference in the mobilities of titanium and nitrogen (atomic weight 48 and 28 respectively).

Typical Cycle

A typical cycle starts with inspection of the received workpieces for any defects and acceptability. Accepted parts are then thoroughly cleaned, dried and loaded into the vacuum chamber. Typical cleaning procedures include solvent vapor degreasing, ultrasonic washing with alkaline cleaners, distilled water rinsing and drying by means of a freon drier. In cases of low production and testing, final drying of parts with pressurized dry nitrogen from the bulk tank, rather than with the freon drier, has given satisfactory results. The parts are carefully loaded into the work chamber with gloved hands to prevent soil from fingers and then the vacuum chamber door is closed. To assist in gas evolution of any water vapor that has been absorbed on the chamber wall liners when the door was opened to air, a double vacuum pump is operated. Initially, the chamber is pumped to near 75 microns Hg which is the cross-over point from the roughing cycle to the high vacuum cycle. Vacuum valves are then closed and dry nitrogen, which is used very effectively as a cooling and backfill gas, is bled into the chamber up to a partial vacuum of 700 Torr. At this point the vacuum pumping sequence is started again and allowed to follow the vacuum program cycle. Warm water recirculating at about 120°F (49°C) warms the vacuum chamber wall tracings to help remove condensation and assist in the vacuum bake-out during the pump down. At time of high vacuum cross-over point, the cryo-coil unit (Polycold) is switched into operation. This unit helps pump residual water vapor by condensing and freezing, thereby achieving a high vacuum as quickly as possible. An additional aid in raising chamber temperature and degassing water vapor is the energizing of the plasma assist hot filament. The filament heats the chamber, liners and heat shields, and workpieces by radiant heating and thereby releasing water vapor. Once the chamber is at a suitable vacuum level and the outgassing cycle is over, the warm recirculation water is switched to cold to prevent additional water vapor degassing during the coating part of the cycle.

Argon gas then is bled into the chamber through a gas mixer/distributor which assures uniform gas distribution. An electrical plasma is started and the parts are sputter cleaned with operation between one and two thousand volts. Once clean of microscopic soils, usually taking about thirty minutes, the parts are heated by energizing the hot filament ion assist while maintaining an argon plasma. Once the parts have reached coating temperature, normally 600°F for high speed steels, coating begins with a fixed partial pressure plasma of argon. The electron beam gun is energized for vaporizing the titanium and a nitrogen gas flow is added at the appropriate rate to form the TiN compound. The evaporation time required for approximately 2-3 micron coating thickness is 20-30 minutes.

After the coating is complete, the parts are allowed to vacuum cool to approximately 800°F (427°C) or less. The tempering temperature of the workpiece dictates the maximum allowable coating temperature. Some materials, therefore, are not exposed to this high a temperature. Once below the target temperature the chamber is backfilled with dry nitrogen to 700 Torr. Static cooling is maintained at this pressure, with the help of a pressure switch and a solenoid valve, to a temperature of approximately 200°F (93°C) at which point the workload is removed from the chamber.

Details

Temperature measurement is through two Inconel Ni-Cr-alloy sheathed thermocouples that are optically isolated for electrical reasons and inserted directly into "dummy" loads resembling the cross-section and mass of the actual workpieces. The flexibility of this type of temperature monitor is that as part size and geometry changes so can the "dummy" load for conformance, thereby assuring accurate temperature measurements.

Experience indicates that gas flowing through a small orifice or a "cracked open" valve into a high vacuum chamber occurs at supersonic speeds. Furthermore, since this gas flow must be very directional a gas mixer/distributor has been installed to: (1) allow introduction of one to four gases; (2) mix all gases thoroughly; (3) distribute gases to four entry ports at equal flows and velocities.

Mass flow controllers have been installed because of instability of the original rotameter type flow meters in control of gas inputs. The controllers, Fig. 7, can be calibrated for any gas, are very dependable, and have good repeatability with excellent control.

To be sure that water vapor is continuously "pumped out" of the system, the cryo-coil unit was installed on a spare chamber wall port. The cryo-coil is a self-contained refrigeration unit used to pump water vapor by condensation. Prior to switching to this method, liquid nitrogen was dumped into a cold trap with a large surface area. The yearly cost for LN$_2$ had a fixed

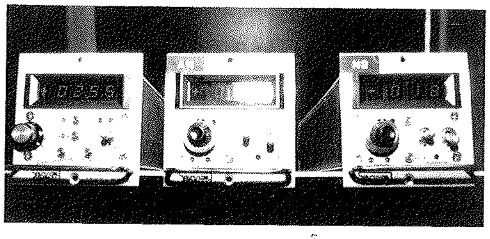

Fig. 7 Pressure gauge and two mass flow controllers for gas inputs.

minimum regardless of the number of runs, since the dewars vent off boiled liquid even if not used.

Other disadvantages of the LN_2 dewars are the difficulty in moving them around and the need for the operator to top off the trap prior to coating. Should the operator fail to top off the tank and the trap runs dry, chamber pressure would rise because of the warming of cold surface area releasing water and causing serious problems with the run. The cryo-coil system is rated at a 7000 liters/sec pumping speed for water vapor. Its size is approximately one half that of a LN_2 dewar and its power consumption is only 1.3 kWh. Installation was relatively easy and efficiency is as good as with LN_2. Figs. 8 and 9 show the cryo-coil unit and method of its installation on the vacuum coating chamber.

Contrary to many published papers stating that there is better stability of the cycle with lower partial pressures in the chamber, the author has found that higher pressures have actually given better equipment control, and no detrimental effects either structurally or in performance of the coating. Numerically, total pressures in the work chamber are 8 to 14 microns of Hg.

The plasma is maintained with an argon partial pressure through a mass flow controller. When ready for coating, the nitrogen mass flow controller is activated and maintains its respective partial pressure keeping both stoichometry and total pressure correct. Because of fixed nitrogen input and ability to monitor excess by use of a residual gas analyzer (RGA), the only adjustment need be the electron beam gun current. This arrangement allows the operator to correct for variations in the heating, cooling, vaporizing, or irregularities in the titanium slug.

Future plans include installation of a turbo pump for the RGA gauge head. This will place the head in a higher vacuum where the head operates at optimum conditions. Such positioning is expected to help prevent the contamination of the head from such things as minute residues of hydrocarbons, etc. Fig. 10 shows this arrangement.

Experiments with various gases for sputter cleaning will continue. Some gases lend themselves better to certain pressures and/or sputter and plasma voltages and currents. Higher currents and lower voltages may yield the best sputtering and heating results, which is somewhat counter to the "higher voltage is better" theory.

Fixturing generally is simple. The fixture can be mild steel or stainless steel. Sharp edges are not desired as arcing problems may occur. Stainless steel fixtures require close attention as they can lend themselves to sputtering problems and hence instability of the run. Small vertical tube holders or larger tubes with holes drilled horizontally can be used for shank tools. Cutters, hobs, etc. can often be hung on a single suspended rod with a plate on the bottom to support them. Stainless steel wire of small diameter is a very good "suspender" for irregularly shaped or difficult parts.

Stainless steel foil can be used very effectively as a mechanical masking material. The method of masking requires care; some techniques are learned only by experience.

Generally, this ion plating equipment is very good. There have been many small problems particularly in the electronics conversion from British to U.S. configuration. Once over these initial start-up problems and the standardizing of the equipment to U.S. practices, the unit has been quite acceptable. Very acceptable coatings have been achieved for the cutting tool and aircraft industries, punch and die work, automotive applications and many others. Examples include cold heading punches for making hand tool sockets, plastic extrusion dies, bending dies for various gauges of wire, level gears for aircraft, and various cutting tools. Anticipated are many new part applications as well as different type coatings in this machine, e.g. other refractory platings.

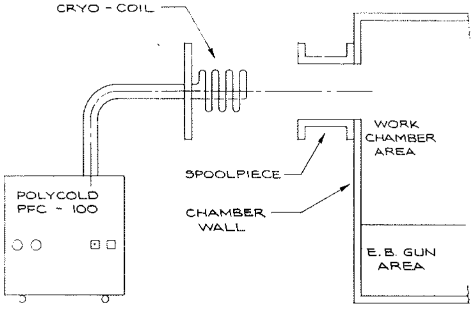

Fig. 8 Orientation of cryo-coil system to vacuum chamber.

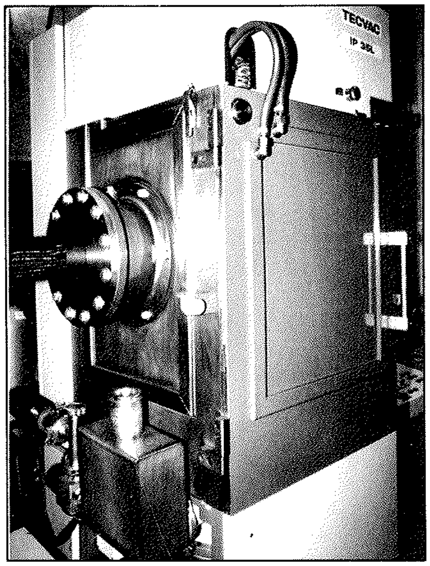

Fig. 9 Spoolpiece — location of cryo-coil.

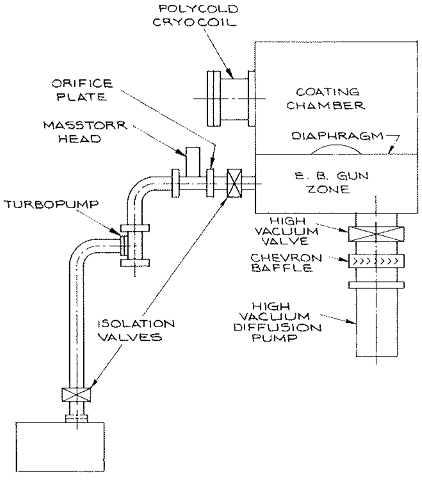

Fig. 10 Turbo molecular pumping arrangement.