ELECTROLYTIC POLISHING OF METALS FOR METALLOGRAPHIC EXAMINATION

JOHN A. BELL

Senior in the Department of Mining and Metallurgical Engineering, University of Illinois, Urbana

To polish a metal by electrolytic means, the piece is first polished on emery papers, and then the article is made the anode in a cell of a suitable electrolyte and cathode. The proper voltage is applied across the cell, and electrolysis is allowed to proceed until the desired polish is obtained. This method was first tried in 1935, and since that time the process has become increasingly popular, especially in the preparation of metals for examination under the microscope.

Electrolytic polishing has many advantages over the standard method of mechanically polishing metallographic specimens. When properly done, the electrolytic method is much faster than the mechanical method. Also, for many metals, electrolytic polishing gives better results, because, when successfully used, it gives a surface that is completely free of scratches, pits, pulled out inclusions, or flowed metal. Another advantage is that one need not be very experienced in order to use electrolysis for polishing, whereas mechanical polishing can only be done successfully and rapidly by one who has had much experience, and who has become very proficient in that type of work. Because of these many appealing characteristics of electrolytic polishing of metals, the process has been of great interest to metallurgists.

The process also involves certain disadvantages that have retarded its wider adoption. One of the largest difficulties involved is the fact that duplication of results are often difficult to obtain unless a setup can be maintained with unvarying conditions, such as constant specimen size, shape, composition and former treatment. Other variables that exert a considerable influence on the results are the amount of stirring of the electrolyte, the composition of the electrolyte, the distance between the specimen and the cathode, the size of the cathode, and the temperature of the bath. When any one of these conditions is varied, the polish that results will probably not be the same as the polish given by the former conditions. This fact has discouraged many metallographers from trying to use electropolishing. Other disadvantages of the method also exist: (1) Sometimes excessive anodic action takes place around inclusions. (2) For some metals, the most popular electrolyte is the dangerous perchloric acid-acetic anhydride solution. (3) The polished surface sometimes shows undulations.

This paper, however, will be limited to the work which was done on trying to find a way to constantly reproduce results, since this is a large barrier to the wider adaptation of electrolytic polishing to metallographic technique. This work was done by the author, under the direction of Mr. B. G. Ricketts of the metallurgical engineering staff at the University of Illinois. The purpose was to see if polishes can be reproduced, under varying conditions, by using the same anodic potential each time a specimen is polished, since anodic potential is the factor that determines the speed and type of corrosion of the metal.

The procedure used was as follows. Two brass specimens of 70 per cent copper and 30 per cent zinc were cut from ⅝-inch round bar. One specimen was ½ inch long, and the other was ⅛ inch long. Two concentrations of electrolyte were made, one being 540 gms. of orthophosphoric acid per liter of solution, and the other being 430 gms. of acid per liter of solution. Two cathodes of copper sheet were formed, one having an area of 22 sq. cm. and the other having an area of 10 sq. cm. A potentiometer was used to measure the combined voltage of a calomel electrode and the specimen anode half cell, and from that measurement, the potential of the anode was determined while polishing was taking place. A rheostat was used to vary the bath voltage from zero to any suitable potential difference, to permit the voltage across the bath to be adjusted to give any desired anode potential. The proper reading was then set on the potentiometer, and the rheostat was adjusted until the galvanometer in the potentiometer circuit showed no deflection. This of course indicated that the known potential of the calomel electrode plus the unknown anode potential was equal to the potentiometer setting. By this means, electropolishing was done consistently at the same anode specimen potential.

The first step in the investigation was to get an electropolish on a specimen under certain set conditions. The appearance of the polished surface was then observed closely at both high magnification (400X) and low magnification (40X). The piece was next sanded and repolished in a bath of different acid concentration, but the same anode potential was used, and other conditions were maintained the same, as much as possible. The resulting polish looked apparently the same as the preceding polish, at both high and low magnifications, so this was an indication that electropolishing might be duplicated by controlling the anodic potential of the specimen.

The polishing was then repeated, using different combinations of large or small specimens, dilute or more concentrated electrolytes, large or small cathodes, with or without agitation of the bath, and varied distances between electrodes. (The electrode spacings varied from 2 cm. to 5 cm.) In each trial, the anode potential was kept the same, and as a result, nearly all of the polishes obtained had the same appearance.

In order to compare the current-bath voltage curves with the current-anode potential curves, these observations were made under the different polishing conditions also. An interesting result was that gassing started on the specimen at the same anode potential, regardless of the variables such as solution cencentration, anode size, cathode distance from the anode, etc.

As a result of the observations made during the investigation, three conclusions are warranted:

(1) The reproduction of polished surfaces is consistently possible by controlling the anode potential during electrolytic polishing of metals.

This statement is further justified by the fact that many investigators of electropolishing have reported that the best surfaces are obtained at bath voltages slightly under that at which oxygen is evolved on the specimen; as stated before, the anode potential is always the same at this point, regardless of the many variables listed.

(2) The best anodic potential for polishing a 70 per cent copper-30 per cent zinc brass in orthophosphoric acid solution is in the range of 1.00-1.25 volts, with the higher voltage preferred.

(3) It is probable that each metal and alloy has an anodic potential range in which a specimen is polished with best results.

It would seem desirable, therefore, to determine the most satisfactory potential for each metal. If this is done, then electrolytic polishing will give consistent results, and a wider application in metallographic technique should be possible.