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Final Analysis

# *Electrolytic Etching for Microstructure Detection in Platinum Alloys*

The most convenient way to reveal the microstructure of platinum alloys used for jewellery or other applications is by electrolytic etching (1–6). The most widely used electrolytic solution is a saturated solution of sodium chloride in concentrated hydrochloric acid (37%). Other solutions can also be used (see Table I).

Electrolytic etching can be considered as 'forced corrosion' where the previously polished specimen surface corrodes inhomogeneously, revealing the different microstructural features which vary from area to area. The surface corrodes selectively as a consequence of different grain orientations, crystal defects such as dislocations and grain boundaries, cold-worked regions and second phases.

Electrolytic etching is usually carried out by means of a DC power supply where the specimen works as the anode. However, for platinum alloys in the saturated NaCl/HCl solution, the best results are obtained with an AC power supply. The degree of electrolytic etching depends on the applied voltage and the etching time. The counterelectrode can be graphite. Better results are obtained if the voltage scale can be varied from 0.1 V to 10 V and the power supply can provide a current of at least 10 A. The specimen dipped in the solution must be kept under a fume hood and must be put in electrical contact with the second electrode.

One of the advantages of electrolytic etching is that the process can be stopped whenever necessary to check the achieved results and started up again to reach the optimal result gradually, a practice that is not always feasible with chemical reagents.

## Other Equipment

In order to carry out metallographic analyses, a laboratory must be equipped with a grinding and polishing machine which allows the use of grinding papers and polishing cloths, and a metallographic optical microscope with a digital image acquisition system. A precision saw with diamond wafering blade can be very useful to prepare small specimens. Some laboratories may have more sophisticated equipment such as automatic systems for specimen preparation and more powerful optical microscopes.

## Metallographic Investigation of Microstructure

Work hardening and thermal treatments cause significant changes to the microstructure of alloys with respect to the as-cast condition. In particular, dendrites are no longer visible. The recrystallisation structure resulting from thermal treatments carried out after work hardening is well revealed in most platinum alloys by electrolytic etching solution 1 (Table I). This information is fundamental when

Table I

Electrolytic and Chemical Etching Solutions for Use with Platinum Alloys

| Etching solution | Composition | Type |
|---|---|---|
| 1 | 100 $cm^3$ HCl (37%) + 10 g NaCl | Electrolytic, 3–6 V AC |
| 2 | 10 $cm^3$ HCl + 90 $cm^3$ $H_2O$ + 1 g $FeCl_3$ | Electrolytic, 3–6 V AC |
| 3 | 100 $cm^3$ HCl (37%) + 3–6 g $CrO_3$ | Chemical[a] |

[a]*Etching solution 3 is chemical, so the specimen can simply be dipped into the solution and no power supply is required. However, it is dangerous for both the operator and the environment as it contains hexavalent chromium. It must be handled with care. This solution is not effective for pure platinum*

setting up working cycles, in order to control the grain size. Furthermore, the metallographic analysis is necessary to characterise possible defects, such as inclusions, porosities, cracks etc.

### Case Study: Microstructure of Two Commercial Platinum Alloys

The microstructures of as-cast 95% platinum-5% copper jewellery alloy and 70% platinum-29.8% iridium high-temperature alloy for industrial use were investigated by electrolytic etching. Etching solution 1 (Table I) was used in both cases. Figures 1 and 2 show the crystal grains of the two as-cast alloys. They are very different in shape and dimension. The grains are in both cases microsegregated and dendritic. When work hardened and heat treated the alloy recrystallises, as shown in Figure 3. Using metallography, the temperature and duration of thermal treatments can be adjusted to obtain a suitable grain size.

### Limitations of the Etching Technique

It is worth remembering that metallographic preparation may not be able to reveal all the microstructural features in each case. Therefore, it is often necessary to use other analytical techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM) or X-ray diffraction (XRD) to get full knowledge of the microstructure.

*Note: this is a revised extract from a paper presented at the 24th Santa Fe Symposium (7).*

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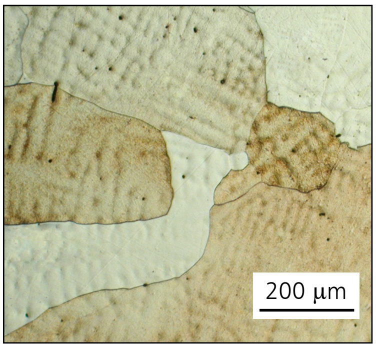


*Fig. 1. Etched sample of as-cast 95 wt% platinum-5 wt% copper alloy. The alloy shows large dendritic grains with copper microsegregation*

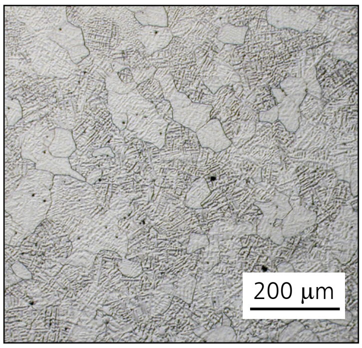


*Fig. 2. Etched sample of as-cast 70 wt% platinum-29.8 wt% iridium alloy. Grains are smaller than in the case of the platinum-copper alloy in Figure 1, and show microsegregation of iridium*

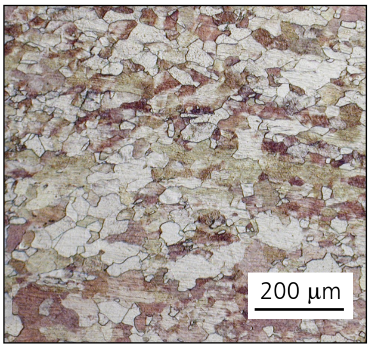


*Fig. 3. Sample of 95 wt% platinum-5 wt% copper alloy after work hardening and annealing, showing the material recrystallisation. Compare with the as-cast microstructure in Figure 1*

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